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BITU2020

Review of the aspects connected to changing supply of bitumen on the Finnish market in the context of performance and quality assurance of asphalt concrete

Michalina Makowska, Mika Köngäs, Leena Korkiala-Tanttu

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Keywords: Asphalt pavements, bituminous mixtures, bitumen

Abstract

Since 2020, the International Maritime Organization (IMO) has had a sulphur limit for ship fuel oils, which will cause changes in the crude oil market and refinery operations. The changes will affect the quality of bitumen used as a binder for asphalt pavements. In Finland, the amount of imported bitumen has increased in recent years so that from 2021 onwards all the bitumen will come from abroad.

The first part of the report describes the background to the imposition of the IMO2020 regulation for setting a sulphur limit, as well as its possible effects, in so far as they may have an impact on the bitumen industry. Changes in the chemical composition of residues from the refining process can affect bitumen products and the properties of asphalt pavements. The second part of the report identifies methods for detecting future differences between various bitumen products, due in particular to their chemical composition. The methods were critically evaluated according to the fact how quickly they could be deployed in the industry. The most promising would seem to be a separation based on infrared spectroscopy combined with machine learning algorithms (MLAs).

The third part of the report examines the causes and possible damage mechanisms leading to the type of damage that has been observed more and more frequently in Finland, suggesting deficiencies in water resistance. They can be caused by raw materials and weakness of the bonds between them, or excessive voids content. These factors affect the cohesion of the asphalt mass under the increasingly varied conditions; winter weather conditions are becoming more fluctuating and demanding as the relative humidity and the freezing-melting cycles increase.

The fourth part of the report surveys and presents laboratory methods for assessing the water resistance of asphalt pavements; test methods previously used in the ASTO study 1989–1991 and currently used in Europe and the USA, as well as possible new test methods. In addition, the effects of the properties of bitumen from various crude oil sources and the use of adhesion-promoting additives on water resistance and studded tyre wear resistance are reviewed, in addition to how they have been evaluated in previous studies. The results of the ASTO study need to be reviewed in the light of current data.

The fifth part of the report reviews the implementation of the questionnaire survey conducted during the project and analyses its results. The survey examined the views and experiences of both those commissioning and producing asphalt pavements regarding damage related to the water resistance of asphalt pavements and their causes, evaluation criteria, test methods and interpretation of their results, as well as the properties of bitumen.

As a result of the study, recommendations shall be made; as bitumen types change and differentiate, it is worth collecting a database of the properties of the bituminous mixtures, combined with the properties of the bitumen used in them (chemical composition and rheological properties of fresh and aged bitumen). A more uniform use of the water resistance tests currently in use should be developed. In addition, it is worth exploring the potential benefits of new methods, such as the Semi-circular Bending (SCB) and Hamburg Wheel methods. Water resistance testing is recommended to verify the dosing of the adhesion agents in the design of the mass. Adhesion agents can also have an impact on studded tire wear and deformation resistance. When improving water resistance, assessments should also include the potential effects on cold resistance, resistance to deformation and fatigue, and the ageing properties of bitumen.

A fast and efficient method for detecting differences in the chemical composition of bitumen would be needed, and this will be investigated in the second phase of the project. The purpose is to collect a database of bitumen compositions determined by infrared spectroscopy and combine them with the rheological properties of bitumen using machine learning methods.

In order to develop the level of know-how, it is noted that awareness of the increase in risks should be raised. It is worth reducing the risks by testing more frequently than the minimums required by the standards, as well as by monitoring variations in the properties of raw materials and masses. It is recommended that the standards, guidelines and quality requirements be more clearly combined.

Michalina Makowska, Mika Köngäs, Leena Korkiala-Tanttu: BITU2020 - Analyysi bitumissa tapahtuvien muutosten vaikutuksista asfalttipäällysteiden ominaisuuksiin ja laadun varmistukseen Suomessa. Väylävirasto Helsinki 2022. Väyläviraston julkaisuja 23/2022. 340 sivua ja 3 liitettä. ISSN 2490-0745, ISBN 978-952-317-960-8.

Tiivistelmä

Vuodesta 2020 alkaen Kansainvälinen merenkulkujärjestö IMO asetti laivojen polttoaineille rikkirajan, joka tulee aiheuttamaan muutoksia raakaöljymarkkinoilla ja jalostamoiden toiminnassa. Muutokset tulevat vaikuttamaan asfalttipäällysteiden sideaineena käytettävien bitumien laatuun. Suomessa tuontibitumin määrä on lisääntynyt viime vuosina niin että vuodesta 2021 alkaen kaikki bitumit tulevat ulkomailta.

Raportin ensimmäisessä osassa kuvataan IMO2020 rikkirajan asettamisen taustaa sekä sen mahdollisia vaikutuksia siltä osin, kun niillä voi olla vaikutuksia bitumiteollisuuteen. Jalostusprosessin jäämien kemiallisen koostumuksen muutokset voivat vaikuttaa bitumituotteisiin ja asfalttipäällysteiden ominaisuuksiin. Raportin toisessa osassa kartoitetaan menetelmiä, joilla voitaisiin tulevaisuudessa havaita erityisesti kemiallisesta koostumuksesta johtuvia eroja eri bitumituotteiden välillä. Menetelmiä arvioitiin kriittisesti sen mukaan, miten nopeasti ne olisivat käyttöönotettavissa teollisuudessa. Lupaavimmalta vaikuttaisi infrapunaspektroskopiaan perustuva erottelu yhdistettynä koneoppimisen algoritmeihin (MLA).

Raportin kolmannessa osassa selvitetään Suomessa entistä useammin havaittuun vedenkestävyyspuutteisiin viittaavaan vauriotyyppiin johtavia syitä ja mahdollisia vaurioitumismekanismeja. Ne voivat johtua raaka-aineista ja niiden välisten sidosten heikkoudesta tai liian suuresta tyhjätilasta. Nämä tekijät vaikuttavat asfalttimassan koossapysyvyyteen entistä vaihtelevammissa oloissa; Talvikelit ovat entistä vaihtelevampia ja vaativampia suhteellisen kosteuden ja jäätymis-sulamissyklien lisääntyessä.

Raportin neljännessä osassa kartoitetaan ja esitellään laboratoriomenetelmiä, joilla asfalttipäällysteen vedenkestävyyttä voidaan arvioida; aiemmin ASTO-tutkimuksessa 1989-1991 käytettyjä, nykyisin Euroopassa ja USA:ssa käytettäviä sekä mahdollisia uusia testimenetelmiä. Lisäksi käydään läpi, millaisia vaikutuksia erilaisista raakaöljylähteistä peräisin olevien bitumien ominaisuuksilla ja tartuntaa parantavien lisäaineiden käytöllä on vedenkestävyyteen sekä nastarengaskulumiskestävyyteen ja miten niitä on aiemmissa tutkimuksissa arvioitu. ASTO-tutkimuksen tuloksia on tarkasteltava uudelleen nykytietojen pohjalta.

Raportin viidennessä osassa käydään läpi projektin aikana tehdyn kyselytutkimuksen toteutus ja analysoidaan sen tulokset. Kyselyllä kartoitettiin sekä asfalttipäällysteitä tilaavien että tuottavien tahojen näkemyksiä ja kokemuksia koskien asfalttipäällysteiden vedenkestävyyteen liittyviä vaurioita ja niiden syitä, arviointikriteereitä, testausmenetelmiä ja niiden tulosten tulkintaa sekä bitumin ominaisuuksia.

Tutkimuksen tuloksena esitetään suosituksia; Bitumien muuttuessa ja erilaistuessa kannattaa kerätä tietokantaa päällystemassojen ominaisuuksista yhdistettynä niihin käytettyjen bitumien ominaisuuksiin (kemiallinen koostumus ja

tuoreen sekä vanhennetun bitumin reologiset ominaisuudet). Nykyisin käytettävien vedenkestävyystestien yhdenmukaisempaa käyttöä tulisi kehittää. Lisäksi kannattaa tutkia uusien menetelmien mahdollisia hyötyjä, esim. Semi-circular Bending (SCB)- ja Hamburg Wheel -menetelmät. Massan suunnittelussa tartukkeiden annostelun varmentamiseen suositellaan vedenkestävyyden testausta. Tartukkeilla voi olla vaikutusta myös nastarengaskulumis- ja deformaatiokestävyyteen. Vedenkestävyyttä parannettaessa on arvioitava mahdolliset vaikutukset myös kylmänkestävyyteen, deformaatio- ja väsymiskestävyyteen sekä bitumin vanhenemisominaisuuksiin.

Bitumien kemiallisten koostumuserojen havainnointiin tarvittaisiin nopea ja tehokas menetelmä, jota tutkitaan projektin toisessa vaiheessa. Tarkoitus on kerätä tietokanta bitumien infrapunaspektroskopialla määritetyistä koostumuksista ja yhdistää ne bitumin reologisiin ominaisuuksiin koneoppimismenetelmillä.

Osaamisen kehittämiseksi todetaan, että tietoisuutta riskien lisääntymisestä tulisi lisätä. Riskejä kannattaa vähentää standardien edellyttämiä minimejä tiheämmällä testauksella sekä raaka-aineiden ja massojen ominaisuuksien vaihteluita seuraamalla. Standardien, ohjeiden ja laatuvaatimusten kokoamista selkeämmin yhteen suositellaan.

Michalina Makowska, Mika Köngäs, Leena Korkiala-Tanttu: BITU2020 - Analys av effekterna av förändringar som inträffar i bitumen, på egenskaperna hos asfaltbeläggningar och kvalitetssäkringen av dessa i Finland. Trafikledsverket. Helsingfors 2022. Trafikledsverkets publikationer 23/2022. 340 sidor och 3 bilagor. ISSN 2490-0745, ISBN 978-952-317-960-8.

Sammanfattning

Från och med år 2020 fastställde Internationella sjöfartsorganisationen IMO en svavelgräns för fartygsbränslen, vilket kommer att orsaka förändringar på råoljemarknaden och verksamheten vid raffinaderierna. Förändringarna kommer att påverka kvaliteten hos bitumener som används som bindemedel för asfaltbeläggningar. I Finland har mängden importerat bitumen ökat under de senaste åren, så att från och med år 2021 kommer allt bitumen från utlandet.

I den första delen av rapporten beskrivs bakgrunden till införandet av IMO2020-gränsvärdet för svavel, samt potentiella effekter av detta på bitumenindustrin. Förändringar i den kemiska sammansättningen orsakade av rester från förädlingsprocessen kan påverka bitumenprodukterna och asfaltbeläggningarnas egenskaper. I den andra delen av rapporten kartläggs metoder för att i framtiden upptäcka skillnader mellan olika bitumenprodukter, i synnerhet sådana som är en följd av deras kemiska sammansättning. Metoderna utvärderades kritiskt utgående från hur snabbt de skulle kunna tas i bruk inom industrin. Det mest lovande verkar vara en sortering baserad på infrarödspektroskopi i kombination med maskininlärningsalgoritmer (MLA).

I den tredje delen av rapporten redovisas orsaker som leder till en i Finland allt oftare upptäckt skadetyp som tyder på brister i vattenbeständigheten, och möjliga skademekanismer. De kan orsakas av råvarorna och en svaghet i förbindningen mellan dem, eller för stort tomrum. Dessa faktorer påverkar asfaltmassans sammanhållningsförmåga under alltmer varierande förhållanden; när den relativa luftfuktigheten och cyklerna av frysning och upptining ökar blir väglagen under vintern mer varierande och krävande än tidigare.

I den fjärde delen av rapporten kartläggs och presenteras laboratoriemetoder med vilka asfaltbeläggningens vattenbeständighet kan bedömas; testmetoder som för närvarande används i Europa och i USA och som har använts tidigare i ASTO-studien 1989–1991, samt potentiella nya testmetoder. Dessutom genomgås vilka effekter egenskaper hos bitumen, härrörande från olika råoljekällor, och användning av vidhäftningsförbättrande tillsatser har på vattenbeständighet och tålighet mot dubbdäckslitage, och hur dessa har utvärderats i tidigare studier. Resultaten från ASTO-studien behöver granskas på nytt mot bakgrund av nuvarande information.

I den femte delen av rapporten genomgås hur en enkät genomfördes under projektet och resultaten av enkäten analyseras. Genom enkäten kartlades synpunkter och erfarenheter både från dem som beställer och dem som producerar asfaltbeläggningar, med avseende på skador med anknytning till asfaltbeläggningarnas vattenbeständighet och orsakerna till dessa, utvärderingskriterier, testmetoder och tolkning av resultaten samt bitumenets egenskaper.

Som ett resultat av studien framställs rekommendationer; när bitumen förändras och differentieras lönar det sig att samla en databas över egenskaperna hos beläggningsmassorna i kombination med egenskaperna hos de bitumener som används i dem (kemisk sammansättning och reologiska egenskaper hos färskt och åldrat bitumen). Det bör utvecklas en mer enhetlig användning av de vattenbeständighetstester som används för närvarande. Dessutom är det värt att undersöka de potentiella fördelarna med nya metoder, t.ex. Semi-circular Bending (SCB)- och Hamburg Wheel-metoderna. För att verifiera doseringen av vidhäftningsmedlen rekommenderas testning av vattenbeständigheten när massan konstrueras. Vidhäftningsmedlen kan också påverka beständigheten mot dubbdäckslitage och deformationsbeständigheten. Vid förbättring av vattenbeständigheten ska även de potentiella effekterna på köldbeständighet, deformations- och utmattningsbeständighet samt bitumenets åldringsegenskaper bedömas.

Det skulle behövas en snabb och effektiv metod för att upptäcka skillnader i bitumenernas kemiska sammansättning, vilket studeras i projektets andra fas. Syftet är att samla in en databas med sammansättningar som bestäms genom infrarödspektroskopi och kombinera dessa med bitumenets reologiska egenskaper med hjälp av maskininlärningsmetoder.

När det gäller att utveckla kompetensen konstateras det att medvetenheten om höjda risker bör ökas. Riskerna bör minskas genom att testa tätare än de miniminivåer som krävs enligt standarderna och genom att observera variationer i egenskaperna hos råvaror och massor. Det rekommenderas att standarder, riktlinjer och kvalitetskrav sammanställs tydligare.

Foreword

The below presented work was conducted as part of the Project *Moisture resistance improvement of asphalt pavements by improving recognition of changes in bitumen. Phase 1* (BITU2020). The project focused on reviewing the changes in refining industry due to the IMO2020 regulations and their impact on the bitumen supply. At the core focus of the project was a gain in understanding of the possible fluctuations on the bitumen market, as well as the potential changes to bitumen composition in the future. Additionally, the investigation evaluates the impact IMO2020 regulations may have on the performance of the asphalt pavements past the transition time of 2020–2022.

Within the project, a special focus was added to the differentiation between bitumen sources and the potential effect it may have on the bituminous mixtures performance. The link between chemical composition of bituminous mixtures and their performance, especially in terms of resistance to moisture damage and low-temperature inflicted damage, was evaluated. For that reason, an effort was paid to investigate the current practice in asphalt concrete industry in Finland related to moisture resistance evaluation and bitumen quality analysis in order to identify the areas for improvement.

The Phase 1 of the project BITU2020 comprises literature and experience review, as well as identification of the parameters for the future experimental evaluation.

The output of the project is a suggestion on the path for the development or improvement of the local specifications in fluctuating market conditions. The goal is to identify the most promising technologies already existing on the market, or on identification of the parameters suggested for evaluation in the quality control methodologies of the future. The output of the first phase of the project is published in form of five sections:

- Part 1 – Bitumen types, composition, modifiers and the effect of the refining process on the composition of the bitumen
- Part 2 – Characterization techniques applied to study bituminous materials
- Part 3 – Moisture damage in asphalt concrete: the mechanism of failure
- Part 4 – National and international experience related to testing methods focused on predicting moisture damage in asphalt pavements
- Part 5 – The survey of practice in Finnish industry related to bitumen characterization and moisture damage prediction.

The Project was co-sponsored by the Finnish Transport Infrastructure Agency (Väylävirasto) and INFRA ry (Infra Contractors Association in Finland) organization.

The document was prepared by Michalina Makowska, D.Sc., with assistance of Mika Köngäs and prof. Leena Korkiala-Tanttu. The work was supervised and commented by the steering group committee. The steering group committee helped to prepare the survey in Finnish language version from the English version file submitted for discussion by Michalina Makowska.

The steering group of the project consisted of:

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Espoo, December 2022

Finnish Transport Infrastructure Agency
Maintenance Management and Development

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1 Project BITU2020

1.1 The goal

The project focuses on reviewing the changes in refining industry due to the IMO2020 regulations and their impact on the bitumen supply. At the core focus of the project is a gain in understanding of the possible fluctuations on the bitumen market, potential changes to bitumen composition. Additionally, investigations evaluate the impact those regulations may have on the performance of the asphalt pavements past the transition time of 2020–2022.

Within the project, a special focus is added to the differentiation between bitumen and bituminous mixtures composition. The link between chemical composition of bituminous mixtures and their performance, especially in terms of resistance to moisture damage and low-temperature inflicted damage, will be evaluated.

The output of the BITU2020 project is a suggestion on the path for the development of the local specifications in fluctuating market conditions. The goal is to identify the most promising technologies already existing on the market, or on identification of the parameters suggested for evaluation in the quality control methodologies of the future during mix design of asphalt concrete.

Project was co-sponsored by the Finish Transport Infrastructure Agency and INFRA (Infra Contractors Association in Finland) organization.

1.2 Scope of Part 1

The hereby report intends to bring to attention some of the aspects related to bitumen due to the change in legislation, as well as the aspects related to the future of its characterization (see Figure 1). Part 1 is reported in chapters 1-5.

This work is prepared for the use inside the European Union, thus the terminology related to bitumen will be used according to the SFS-EN 12597. Additionally, the applied model glossary related to bitumen, its production and its modifiers can be found in the publication by Asphalt Institute and Eurobitume (Asphalt Institute Inc. and European Bitumen Association–Eurobitume 2015).

A large number of available publications, focused on explaining what bitumen is and how it is produced, is available in the literature (Blomberg, Bitumit 1990; Whiteoak 1990; Hunter, Self & Read 2015; Lesueur 2009; Asphalt Institute Inc. and European Bitumen Association–Eurobitume 2015; Blazejowski, Wojcik-Wisniewska & Baranowska 2018). Therefore, this report will not expand on simple definitions covered elsewhere. The short definitions are provided in the glossary section of the report, with the appropriate references to fulfil the needs of more demanding reader.

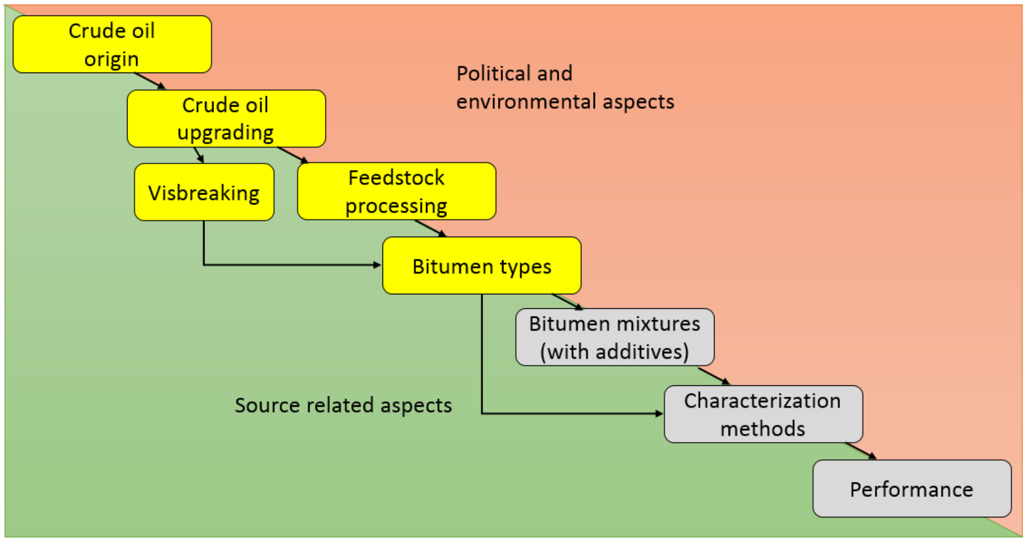


Figure 1. The schematic representation of concepts reviewed within this report (Part 1). The content of the BITU2020 – Part 1 is connected to the boxes marked in yellow, while Part 2 focuses on boxes marked in grey.

The characterization methods of bitumen and asphalt are multiple, and in order to cover every single one of them the publication would need to be much more extensive. Eventually during the literature review, the focus of the project was on techniques and methodologies, which either are the most likely to predict the performance in field or easily adaptable to the industry within short timeframe. The characterization techniques are described in more detail in part 2.

Some of the characterization techniques, such as Gel Permeation Chromatography or Proton Nuclear Magnetic Resonance, are interesting from the point of view of research, but as highly sophisticated are unlikely to be adapted to the asphalt industry within the following 1–5 years due to the laboratory and equipment limitations. Therefore, those methodologies are mentioned or acknowledged, but for practical reason the focus is on methods transferable to the industry.

1.3 Scope of Part 2

In part 2 the different aspects connected to bitumen effect on asphalt, methods of bitumen characterization and some background theories are provided for better understanding of concepts presented in the follow up chapters. Part 2 is reported in chapters 6–7.

1.4 Scope of Part 3

In the part 3 the focus will be on explaining the mechanism through which the damage due to moisture is progressing. Part 3 is reported in chapters 8–12.

1.5 Scope of Part 4

The review of the methods of characterization of resistance of bitumen and asphalt concrete to moisture damage are reviewed critically. The aspect of additives, interaction with inorganic aggregates, composition of bitumen are all considered and reviewed. Part 4 is reported in chapters 13–17.

1.6 Scope of Part 5

In the part 5 of the report, the survey was drawn and presented to the officials and contractors in Finland in respect of procedures of quality control, production control, certification testing and specification understanding. The results of the survey are analyzed and justifications behind the questions are provided. Part 5 is reported in chapters 18–21.

2 Crude oils

The chemical composition of the bitumen is linked to the performance of the bitumen (Redelius & Soenen 2015; National Academies of Sciences, Engineering, and Medicine 2017). The chemical composition of bitumen is inevitably a result of the initial composition of the crude (Schieldrop 2018), as well as the processes applied to its distillation and upgrading (Dente, Bozzano & Bussani 1997; Stratiev, Kirilov et al. 2008; Joshi et al. 2008; Tong, Shen & Fang et al. 2017).

The crude oils are typically differentiated between using their content of sulfur and their density. The chemical composition of the crude oil in terms of content of aromatics, paraffinic and naphtenic species, may also define the final properties of the final product, i.e. the bitumen.

2.1 Sour and sweet crude oils

The crude oils are classified based on their sulfur (S) content, expressed as weight percent of sulfur in oil. The **sweet crude oil** refers to material of approximately less than 0.5% wt. S, while **sour crude oil** to the material of more than 0.5% wt. S. However, Platts separates the sour from sweet at 0.6% wt. S. (S&P Global Platts 2019), while Marafi et al. divide the sour from sweet at 1% of sulfur (Marafi, Albazzaz & Rana 2019). The higher content of sulfur is present in the higher boiling point fractions of crude oil, and the highest sulfur content is measured in asphaltenes (Hunter, Self & Read 2015).

The sour crudes need to be pre-processed to remove the excess sulfur content in order to assure good performance of refinery installation and quality of the products. The trend to reduce sulfur content in the fuels produced by the refineries pushes refineries to

- limit feedstock types or
- to invest in preprocessing units focused on lowering the S content.

Due to the continuously globally increasing content of sulfur in the crude oil sources (J. Speight 2012), the second solution allows for better flexibility on continuously changing market.

2.2 Light and Heavy crudes

The crude oils can be divided by the means of American Petroleum Institute gravity known as API gravity (Blomberg, Bitumit 1990). This value allows for a fast conversion between metric tons and barrels, therefore is widely used in the industry.

Light crude oils are a type of liquid petroleum of low specific gravity, low viscosity and high API gravity (above 20°). Heavy crude oil, on the other hand, is having high specific gravity, high viscosity and low API gravity (below 20°). The heavy crude oils are the ones which contain more asphaltenes, and with lower proportion of light hydrocarbon fractions.

A division into four API grades (Louisiana Department of Natural Resources 1989) is also used and could be defined accordingly:

- Light crude oil – API > 31.1°, density lower than 870 kg/m^3
- Medium crude oil – API between 22.3–31.1°, density between 870–920 kg/m^3
- Heavy crude oil – API < 22.3°, density between 920 to 1000 kg/m^3
- Extra heavy oil – API < 10°, density greater than 1000 kg/m^3

However, varying sources seem to define the crude oil type according to differing API values (Blomberg, Bitumit 1990; S&P Global Platts 2019; Marafi, Albazzaz & Rana 2019; U.S. Geological Survey n.d.). The division to sweet and sour, light and heavy seems to differ between the regions of the world and date of the reference, thus it is not apparent to the author of hereby report what is the internationally accepted cut off threshold criteria.

For the purpose of understanding further sections of hereby report it is advised that the reader remembers that bitumen originating from different sources may be inheriting the characteristics of the original crude oil, and therefore be more sour or sweet, etc..

2.3 Chemical type of crude oils

The crude oils can be also divided based on their main component composition. This is useful for the purpose of evaluation of so-called pressure-volume-temperature (PVT) properties. As a result of the classification by chemical components the crudes can be divided into:

- naphthenic crude – a crude oil containing less than 50% saturated hydrocarbons and over 40% naphthenic hydrocarbons
- paraffinic crude oil – the crude oil containing more than 50% saturated hydrocarbons and over 40% paraffinic hydrocarbons
- aromatic crude oil – the crude oil containing less than 50% saturated hydrocarbons, and over 50% aromatics, resins and asphaltenes (BCN B.V. 2019).

The crude oil chemical composition is often connected to the geographical location (Society of Petroleum Engineers 2015).

The initial chemical composition will influence the yields of the distillates and quality of the bottom of the barrel (Louisiana Department of Natural Resources 1989). The paraffinic crudes were known to produce the waxy bitumen.

However, nowadays the refineries tend to use a blend of various crudes for their operation. It is very rare that a refinery has a specified installation for processing of only one crude oil (Schieldrop 2018).

2.4 The route from crude oil to bitumen

The global average composition of the crude in 2019 and the most abundant sources of crude oil determine the average composition of crude oil on the market, as well as the future composition of average crude oil. The certain trend of increasing sulfur worldwide due to the increased consumption of sweet crudes and their low availability will demand changes for refineries (Schieldrop 2018).

To understand which fractions of distillation can be sold as bitumen is crucial. To understand that the same fractions will be affected by the changed in the legislation after 2020, is a first step to identifying or predicting the composition of the bitumen in the future.

2.5 Crude Oil in the global context

Based on the data provided in the International Energy Statistics database (2018), the main reserves of the crude oil worldwide are presented in Figure 2.

Table 1 presents some chosen data for the most abundant sources of crude oils in the world gathered from the literature. Based on the data available for the crude oil sources, their abundance and composition, the global barrel composition was calculated as API = 32 and sulfur content of 1.3% (Schieldrop 2018). The worldwide trend with time is the global decrease in the API and global increase in the sulfur content as the sweet crude oil is in higher demand (J. Speight 2012).

The monitoring of the sulfur content and API gravity is a mean to follow the quality of the crude oils. The crude oil composition available on the market is important to optimize the operations of refineries and certain monitoring of the quality is ongoing globally (S&P Global Platts 2013; 2019). More up to date information about the sulfur concentration, API gravity, origin of the crude oil and its output in barrels per day can be found in so-called Platts periodic table of oil for 120 crude oils (see Figure 3) (S&P Global Platts 2019).

In principle, the crude oils with highest reserves are the heavy and sour crude oils. In the following decades, with decreasing availability and depletion of the sweet and light crude oils, those feedstocks will be the most abundant on the market.

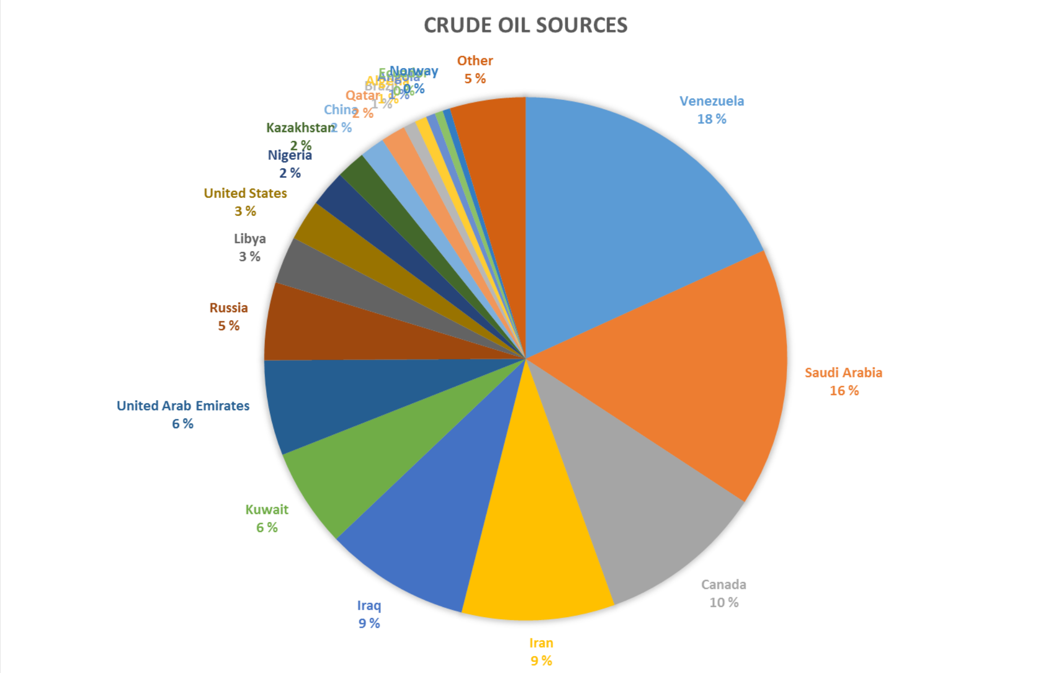


Figure 2. The sources of crude oil worldwide divided by the country of origin. Data source: International Energy Statistics 2018.

Table 1. Example of crude oil compositions by their API and sulfur content gathered from various literature sources.

Country of origin	Name	API [°]	Sulfur con-tent [%wt.]	Ref
Venezuela	Heavy Merey	15	2.7	(Oil &Gas Journal 1997)
	Leona	24	1.5	(Oil &Gas Journal 1997)
	Orinoco	2.6	4.4	(Sawarkar, ym. 2007)
Saudi Arabia	Light Arabian	7.4	4.1	(Sawarkar, ym. 2007)
	Heavy Arabian	4.5	5.25	(Sawarkar, ym. 2007)
Canada	Par Crude	40.0	0.3	(S&P Global Platts 2013)
	Mixed Light Sour	29.3	1.6	(S&P Global Platts 2013)
	Cromer Light Sour	35.1	1.2	(S&P Global Platts 2013)
	Sour at Edmonton	32.5	1	(S&P Global Platts 2013)
	Cromer - Midale	29.3	2%	(S&P Global Platts 2013)
Iran	Iran Heavy	31–32	1.8	(S&P Global Platts 2013)
	Iran Light	33–34	1.4	(S&P Global Platts 2013)
Russia	Siberian Light	35–36	0.6	(S&P Global Platts 2013)
	CPC blend	43.5	0.5–06	(S&P Global Platts 2013)
Libya	Es Sider	36–37	0.4%	(S&P Global Platts 2013)
Global Barrel (calcu-lated)	All	32	1.3	(Schieldrop 2018)

Country of origin	Name	API [°]	Sulfur content [%wt.]	Ref
Optimal barrel for no changes in classical average global installation	e.g. Bonny Light	37	0.14	(Schieldrop 2018)

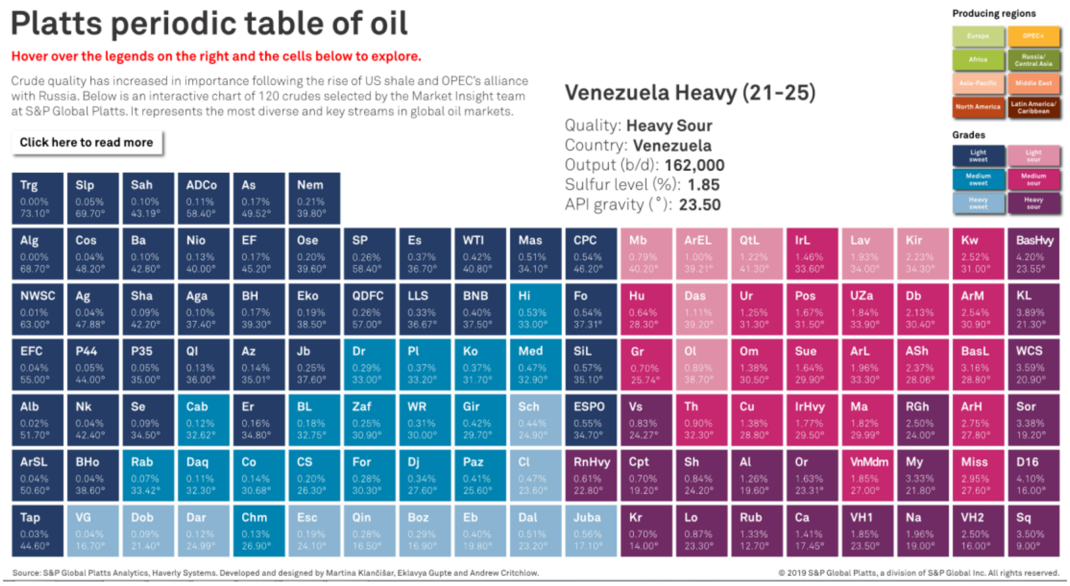


Figure 3. The Platts periodic table of oil in which the current information about the crude oils on the fluctuating market can be easily accessed.

2.5.1 Mainstream refinery production

The classical refinery construction includes the atmospheric residue distillation column, from which the atmospheric residue (AR) is collected. The more upgraded versions of installation include vacuum distillation processing unit, which allows for division of the AR into vacuum gas oil (VGO) and vacuum residue (VR). The processes and installations are discussed for example in (Whiteoak 1990; Blomberg, Bitumit 1990; Hunter, Self & Read 2015).

The heavy fuel oil (HFO) sold on the market can be equal in composition to AR, VR or VGO. The bitumen produced from the crude oil comes on the other hand from processing of the VR (Figure 4). Hence, prior to year 2020 during the low market demand for the bitumen, the bottom of barrel could have been sold as a High Sulfur Fuel Oil (HSFO) of the sulfur content below 3.5 % (HFO < 3.5 %), if the feedstock allowed for the AR, VR and VGO meet the sulfur content of below 3.5%.

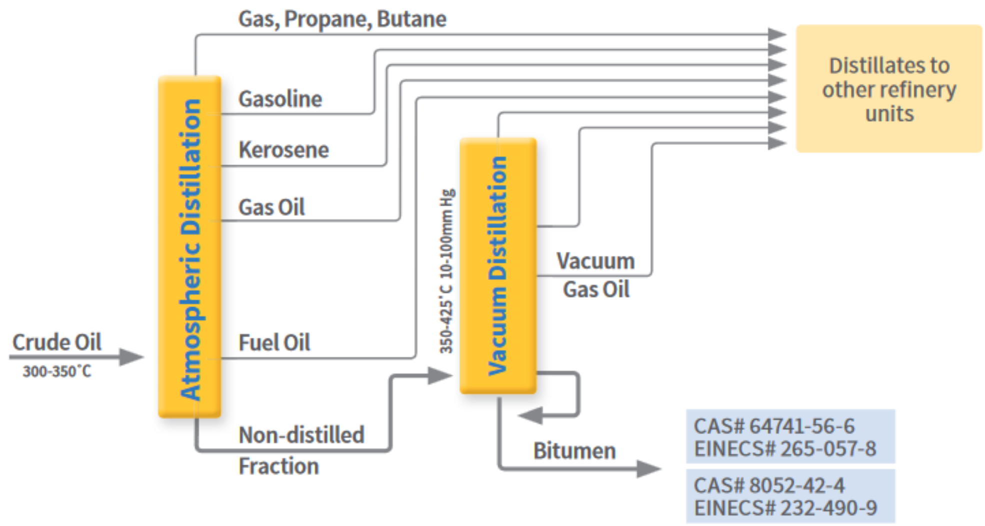


Figure 4. Schematic diagram of the distillation process. Reprinted from Asphalt Institute Inc. and European Bitumen Association–Eurobitume (2015).

This overlap between VR and HFSO meant, that in case of the surplus of bitumen fraction, a second market was available for it – the energy use. The bitumen market is not strong enough to consume all of the VR produced. Especially with the crude supply continuously moving toward more sour global composition, the volume of the VR fraction is expected to increase.

3 Bitumen types and definitions

Definitions used in this work are uniform with the standard SFS-EN 12597:2014 and are provided in the definition box below.

bitumen - virtually not volatile, adhesive and waterproofing material derived from crude petroleum, or present in natural asphalt, which is completely or nearly completely soluble in toluene, and very viscous or nearly solid at ambient temperatures

paving bitumen - bitumen used to coat aggregate and/or reclaimed asphalt, mainly used in the construction and maintenance of paved surfaces and hydraulic works

industrial bitumen - bitumen used for purposes other than the construction or maintenance of paved surfaces

modified bitumen - bituminous binder whose rheological properties have been modified during manufacture by the use of one or more chemical agents

Note 1 to entry: In this context, “chemical agent” includes natural rubber, synthetic polymers, waxes, sulfur and certain organo-metallic compounds, but not oxygen or oxidation “catalysts” such as ferric chloride, phosphoric acid and phosphorus pentoxide. Fibres and inorganic powders (“fillers”) are not considered to be bitumen modifiers. Modified bitumens may be employed “directly” or in the form of cut-backs or emulsions, or blended with (for example) natural asphalt.

multigrade bitumen - special bitumen for road applications which is less temperature susceptible than paving grade bitumen and having a penetration index *I*p positive

The standards corresponding to the bituminous binder types, which define them in more detail are listed in Figure 5.

Even though a perception exists that polymer modified bitumen is a product used very widely in United States of America, according to the National Academy of Science report, it comprises only 12% of the market (National Academies of Sciences, Engineering, and Medicine 2017). The most popular bituminous product is the non-modified bitumen. In Europe the popularity of the type (or grade) of product depends on the country and its specifications. In Finland, the paving bitumen is mostly non-modified, while industrial bitumen is often used for special applications such as moisture insulation, roof tiles and waste immobilization and is often modified.

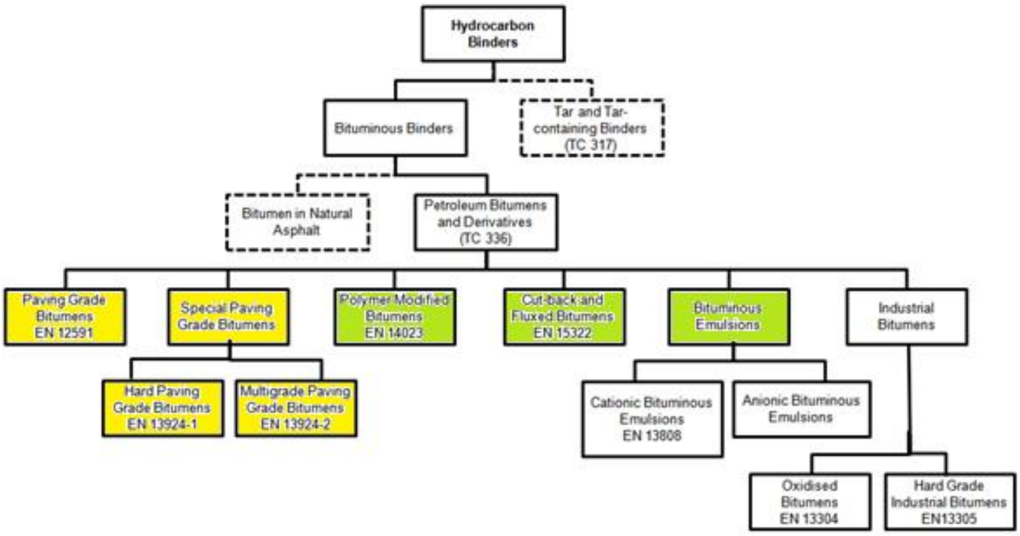


Figure 5. The classification of hydrocarbon binders according to the SFS-EN 12597, where yellow boxes represent issues discussed within BITU2020 – Part 1 and green boxes represent materials discussed in BITU2020 – Part 2.

3.1 Soft bitumen production related terminology

Soft bitumen is a special subcategory of the paving grade bitumens, with higher importance for Nordic countries. Soft bitumen is used for the preparation of the Soft Asphalt Concrete mixtures (Fin: PAB), applied on the low trafficked roads in the cold climate regions. Bitumens of viscosity grade V1500, V3000, as well as Penetration grade 650/900 (SFS-EN 12591), typically used in PAB mixtures, will be understood as the soft bitumens in this work.

Soft paving bitumen can be obtained as a result of distillation of crude oil. However, technically, according to the terminology the soft paving bitumen can be a harder paving grade bitumen fluxed with petroleum flux (e.g. gas oil) to reduce the viscosity (Singh & Kumar 1997), but in such case currently it should be referred to as petroleum fluxed bitumen. If the flux oil is of non-petroleum origin, the name should reflect if at least stating that it is a flux bitumen.

The glossary related to soft bitumen production in line with SFS-EN 12597 is provided below in the definition box.

soft paving bitumen – paving bitumen mainly used in the manufacture of soft asphalt

flux / cut-back solvent – fluid added to another to reduce its viscosity

flux or flux oil – relatively involatile fluid (oil) used in the manufacture of fluxed bitumen

fluxed bitumen – bitumen whose viscosity has been reduced by the addition of flux oil(s) (2.7.1)

petroleum fluxed bitumen – bitumen whose viscosity has been reduced by the addition of a flux oil derived from petroleum

4 International Maritime Organization (IMO) – Sulfur cap of 2020

In order to adhere to the goal of reducing the impact of transport on the climate change, the IMO decided to inflict the sulfur cap regulation on all the ships (International Maritime Organization 2019). The reduction of the emissions of sulphur oxides (SOx) into the atmosphere, the product of combustion of sulphur contained in organic fuel, is envisioned to

- reduce the impact on the human health (Marine Environment Protection Committee estimated based on a report submitted by Finland that a prevention of 570 000 deaths is achieved worldwide, on the condition that the legislation is enforced on 2020 as opposed to 2025 (Corbett et al. 2016))
- decrease severity of acid rains, (protecting crops, forests and aquatic species; prevention of acidification of the oceans).

Currently, the allowed sulphur content in the fuel used by the vessels is on the level of below 3.5%wt. (High Sulphur Fuel Oil or Heavy Fuel Oil, fin: *raskas polttoöljy*, HFO < 3.5% or HSFO) (Schieldrop 2018). The suggested limit for the ships starting from 2020 is defined as a desired emission of 0.5% wt. of sulfur per mass of consumed fuel. Effectively it is estimated that the global average content of sulphur in the HFO is on the level close to 1.3% wt. (Schieldrop 2018).

4.1 Methods of reducing the sulfur emissions

The suggested method of achieving such reduction in SOx pollution is to use the fuel designed for marine application with the Sulphur content below 0.5 % wt (marine fuel oil, MFO < 0.5 %).

The emerging alternative solution, which is not currently infringing on the proposed change in limits on emission gases, is in the installation of so-called “scrubbers”. Scrubbers are essentially an installation which post-processes exhaust gases, causing the absorption of SO_x into the absorbing fluid or solid used in the scrubber. There are multiple technological designs of scrubbers. The open-loop wet system simply sprinkles water, often seawater, in order to absorb the SO_x and produce acidic liquid. However, the liquid after such post-processing is released into the seawaters, causing local change of acidity, temperature and turbidity (Lange, Markus & Helfst 2015). The discussion about the environmental impact is ongoing between the maritime industry and IMO, but is not the core focus of hereby report.

Other solutions such as wet scrubbers with close loop, in which post-processing liquid is released in ports or at disposal stations, as well as membrane and dry scrubbers, which reduce the need to dispose of liquid exist. Some of the solutions in fact produce by-products such as gypsum or potassium sulphate (fertilizer), which can be sold to create net zero operational cost for the installation (Latarche 2018).

The reason behind investment into the scrubbers in order to plan for the transition period is the predicted lower price of HFO < 3.5% in the future, due to decreased market demand and continuous surplus of production. However, the reason widely vocalized behind this approach is due to the safety issues (Lowry 16). There is a concern that the use of novel and highly fluctuating in composition fuel may cause the issues for engines during combustion, and it is feared that on long distance shipments the safety of crew and cargo may be compromised (Lowry 16). Especially the ships which will have an easy access to HFO < 3.5%wt., such as those transporting between refineries and ports, may benefit from the use of fuel at a reduced price, but at the same time are at a higher risk in case of engine damage (Lowry 16). The scrubbers are mostly fitted on cruise ships, Ro-Ro-ships, gas tankers, crude oil tankers, long distance cargo ships (Schieldrop 2018).

However, for the bitumen and asphalt industry the most important is the effect to the refinery processes inflicted by the emerging of the new type of fuel, namely MFO < 0.5%.

Fuel Oil is a product of distillation of crude oil, it can be obtained either as a distillate or a residue. The overlap with the bitumen definition is underlined hereby. The residue from the vacuum distillation unit (VDU) – the second stage of distillation after AR – can be marketed as fuel oil, post-processed in upgrading units or used as a substrate in the production of bitumen. Over the course of time the production of this product from the crude oil barrel, expressed by a percent of the barrel, has been continuously decreasing due to the emergence of new upgrading proceses (J. Speight 2012). The fuel oil may be a direct straight run from the bottom of the distillation column under atmospheric conditions (AR), or Vacuum Gasoil (VGO) or Vacuum Residue (VR).

Figure 6. A sample of residual fuel oil. Source: Wikipedia, author: Glasbruch2007 (wikipedia.org n.d.)

As mentioned previously, the average global crude barrel composition is estimated to have API 32 and 1.3% wt (Schieldrop 2018). The distillation of products low in sulfur (namely removal of fractions such as for example kerosene) leads to the accumulation of sulfur into the bottom of the barrel (AR, VGO and VR). Using the global average Sulphur content in crude oil, it is estimated that the concentration in AR, VGO and VR would be too high to meet the requirements of MFO < 0.5% wt. Sulphur content. It is estimated, that in order not to conduct changes in the refinery installations, the African Sweet crude oils should be preferred to meet the requirements of MFO < 0.5%.

It was calculated that the Sulphur content of 0.14% would allow for all the barrel cuts to meet the requirements inflicted by the IMO. Therefore the Bonny Light crude oil (which has the sulfur content of 0.14%) is used as a reference crude oil for the IMO < 0.5%. (Schieldrop 2018). This is not necessarily practical for the whole globe to rely on the small number of crude oil sources, but indicates one of the possible trends of demand shifting towards certain type of crudes.

The typical split of S content between various cuts of the barrel is demonstrated in Table 2. The heavier and more sour crude oils are lowering the yield from feedstock in terms of fractions that are marketable after 2020. In the current global situation, the atmospheric distillation unit (ADU) would be sufficient to produce the cut (AR) with the sulfur level below the HFO < 3.5% mark. If the sufficiently sweet crude is used as a feedstock, this is still possible in the future without investments into the installation (Schieldrop 2018).

It is important to mention, that the Shieldrops's report is simplifying refineries operations by analyzing the product marketability by just sulfur content for global economical analysis purposes (Schieldrop 2018). Refineries may use different parameters for optimization of their production, depending on local legislation, markets, fuel prices and demand.

Table 2. The estimated split of sulfur between the volume cuts of the barrel for the global average feedstock and the example of the sweet crude Bonnie Light (Schieldrop 2018).

Name of the cut	Feedstock: Bonnie Light, **0.14% S** and **37°**		Feedstock: Global average, **1.3%** S and **32°**	
	Volume Cut [%]	Sulfur content [%wt.]	Volume Cut [%]	Sulfur content [%wt.]
Butane lighter	2	-	2	-
Light Naphta	6	-	6	-
Heavy Naphta	19	-	16	-
Kerosene	17	0.0	14	0.1
Diesel	19	0.1	18	0.7
AR	**36**	0.3	**45**	2.2
Total	99		101	
When AR is used as feedstock for Vacuum Distillation Unit it splits into two cuts:				
VGO	**26**	0.2	**28**	1.7
VR	**10**	0.5	**17**	3.2

Not all refineries contain the VDU, thus some of them are able to produce only AR. Without investment into the installation the refineries willing to market the AR and VR as IMO2020 compatible MFO, require the use of light and sweet crude oils. Those crudes are expected to become thus consistently more expensive and in higher demand.

The second path to achieve this upgrade is the investment in the installation and processing units. Investment in the VDU is not sufficient enough to lower the sulfur content below the required for the MFO < 0.5% as is visible in Table 2.

Currently the HFO < 3.5% is sold at a lower price than the price of the barrel. The current lower cost than that of raw material assumes that in order to process this cut of the barrel a substantial investment would need to occur. In the situation in which the HSFO in pre-2020 form does not meet the requirements of the IMO2020, the market for the high sulfur content cuts shrinks, causing potential losses for the refinery due to lack of market. The refineries are thus more likely to invest in upgrading technologies.

This is the reason behind the necessity of using crude oil upgrading processes. They are targeted at an increase of the yields of low sulfur containing cuts. Some of the upgrading units may unfortunately provide sufficient amount of premium for the alternative products, which leads to the complete halt on the production of VR and bitumen as we knew it (ABB Lummus Global B.V. 1996).

4.2 Residue Upgrading Processes

The residue upgrading processes aim at one thing, namely increase of the yield of the lower boiling point cuts of the barrel. Doing so increases the sulfur content in the various residues from those upgrading processes but lowers the sulfur content in the lighter fractions.

With the continuously increasing global sulfur content and lowering API gravity, the drive to invest in the upgrading units may be due to multiple reasons:

- Protection of installation and personnel– sulfur in feedstock changes into H_2S gas during heating operations, this gas is corrosive for the installation and dangerous for the workers (Joshi et al. 2008)
- The necessity for maintaining transport through pipelines – the feedstock from heavy and sour crudes is rich in asphaltenes which foul inside the pipes; deasphalting of the crude prior to transport lowers the pipeline installation maintenance (Zachariah & de Klerk 2017)
- Prevention of coking on the installation – the asphaltenes and coke formed during heating operations on the sour and heavy crudes, leads to precipitation of those compounds on the walls of heat exchangers and reactors, causing the increased consumption of fuel for the heating operations of the refinery; the lower coking is reducing the down time of the installation, allowing continuous operation of refinery (Joshi et al. 2008; Sawarkar et al. 2007)
- Prevention of the catalyst deactivation – sulfur is known to deactivate many catalysts, thus refineries focused on production of gasoline and equipped in the catalytic crackers may benefit from the lower poisoning of the catalyst (Marafi, Albazzaz & Rana 2019)
- Financial aspect – higher premium on the low boiling point cuts (Schieldrop 2018)
- Legislative – the sulfur restrictions on diesel in the past decades, and – coming in 2020 – on fuel oil, lead to the situation of a potential loss of market to sell a third of the barrel (International Maritime Organization 2019).

It is the type of crude oil and the profile of refinery, which determine the upgrading unit chosen by the refinery. There is a variety of existing and emerging processes (Elshout et al. n.d.). The common upgrading processes include:

1. Visbreaking
2. Coking
3. Deasphalting
4. Hydroprocessing (steam-cracking)
5. Fluid catalytic crackers (FCC)

The hydroprocessing is requiring substantial amounts of energy. For this reason, the visbreaking units became more popular. The fluid catalytic crackers replace the steam crackers in refineries focused on the production of gasoline. The FCC unit produces short chain hydrocarbons and coke, which is burnt in the process and used as the fuel for the process (Sawarkar et al. 2007). As such, there is no by product for reuse in the bitumen industry from this process and for that reason it will not be discussed further. The visbreaking and deasphalting are both capable of producing a fraction, which meets the definition of the paving bitumen according to SFS-EN 12597 and for this reason the two processes are looked at in more detail.

Due to the active marketing of Shell Visbreaking technology (ABB Lummus Global B.V. 1996), the European market is heavily equipped with visbreakers, in comparison to U.S.A., where perhaps coking was the preferred upgrading methodology up to 1998 (Sawarkar et al. 2007). The deasphalting process was not preferred, until the discovery of the potential use of supercritical CO_2, which lowered the thermal cost of the process.

The residue from the deasphalting and/or visbreaking can still be marketed as very hard bitumen under the definition provided in SFS-EN 12597 (Asphalt Institute Inc. and European Bitumen Association–Eurobitume 2015; Tong, Shen & Fang et al. 2017; Tong, Shen et al. Solvent deasphalting of Saudi residue to produce 30# hard asphalt 2016; Preparation and evaluation of 30# hard grade asphalt 2016).

4.2.1 Visbreaking

The processes used to reduce viscosity (break the viscosity) of the feedstock are known as visbreaking. The visbreaking processes are a subset of mild thermal cracking processes. The process is used to convert 5–10% of VR (bitumen component) into naphtenic products to achieve a tenfold reduction in viscosity. (Joshi et al. 2008; J. Speight 2012).

The parameters which affect the yields of the desired fractions and composition of the products are

- Feedstock type
- Temperature
- Pressure
- Residence time.

The later three parameters are affected largely by the design of the visbreaker unit (Kulkarni et al. 2010: Joshi et al. 2008).

The issues which affect to the industrial process include

- Characteristic of the feed
- The induction of instability
- The coke formation and fouling on the installation.
- The type of visbreaker (commercially available are: soaker visbreaker and coil visbreaker).

The visbreaking feedstock is typically the vacuum residue, which otherwise would be used for the production of bitumen.

To sum up, the type of visbreaker, time of residence and temperature within the unit will determine the composition of the visbroken residue (VBR). Underlined is the possibility of marketing the visbroken residue as the bitumen according to the common definition (Figure 7).

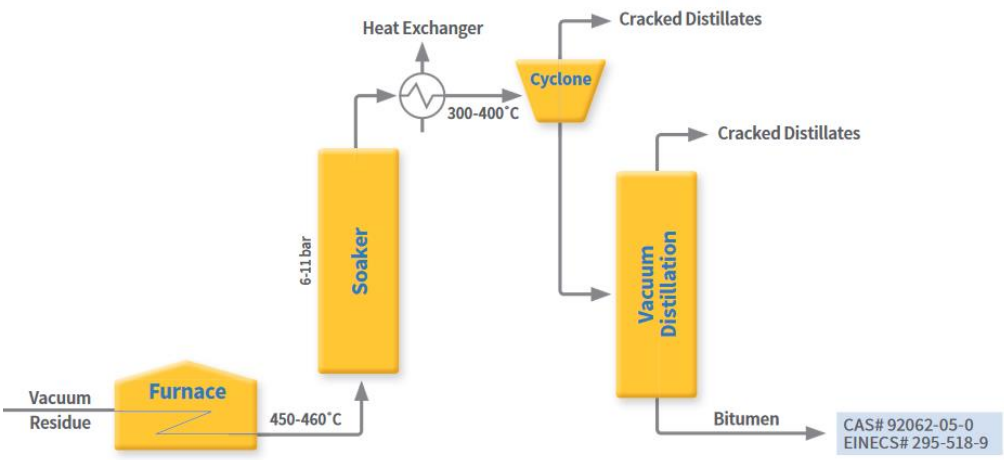


Figure 7. The schematic representation of the Visbreaking installation into which the vacuum residue is used as a feedstock. Reprinted from Asphalt Institute Inc. and European Bitumen Association–Eurobitume (2015).

The conversion of the feedstock in the visbreaker causes the increase of the saturates and asphaltene fraction content, while reducing the aromatic fraction content (Stratiev, Kirilov et al. 2008). The resin content seemingly remains on a constant level. The colloidal stability of the bitumen depends on the ratio between the saturates, aromatics, resins and asphaltenes. The sudden shift of the composition towards low molecular compounds and increased concentration of asphaltenes causes the potential loss of stability. The loss of stability leads to the precipitation of the asphaltenes and separation of the oily phase (Lesueur 2009).

From the point of view of refinery operation this limits the possible yields and conversion of VR into VBR. Certain yields are chosen in order not to cause the precipitation of asphaltenes in the installation.

Because of the different colloidal stability of visbroken residue to that of vacuum residue, the colloidal stability of the bitumen containing the visbroken residue becomes of importance and concern (Dasek et al. 2017).

The content of the sulfur in the visbroken residue after vacuum distillation is higher than in the feed of the visbreaker (Kulkarni et al. 2010). Sulfur content could be one of the parameters to follow for qualitative differentiation between bituminous products.

Stability of the feed can be also evaluated by the content of metals or by conductivity of the feed (Joshi et al. 2008), as the crude oil's minerals also concentrate in the products after visbreaking. The sodium content in the visbroken residue may be different than in the vacuum residue from the same crude oil.

The content of sodium is known to influence the emulsification properties of the bitumens (Hunter, Self & Read 2015). This parameter may prove very important in terms of preparation of emulsions but also in protection against moisture damage. Sodium content is postulated as one of the variables to follow up on after the uptake of the new bitumen into the asphalt concrete production.

4.3 Coking

Coking is a batch process focused on conversion of the asphaltenes into the solid particles and their precipitation. The coke itself cannot be considered as a bitumen production substrate. However, its production from the VR is of significance. The coke can be a premium product as a fuel and substrate for syngas and synthetic fuel generation. The coke can be stored as solid in non-heated tanks (as opposed to liquid bitumen, which requires hot storage) and further sold as fuel for the brick and metal industries. The coke can be also a substrate for the production of anodes in batteries (Elshout et al. n.d.; Sawarkar et al. 2007; J. Speight 2012).

4.4 Deasphalting

The stability of crude oil is understood as the homogenous mixture of asphaltenes in the lighter fractions. However, in certain conditions, related to temperature, pressure and concentration the onset of instability in crude oils is observed. With increasing distance between the asphaltene molecules, those molecules connect creating aggregates and precipitate from the solution. The definition of asphaltenes is of the material insoluble in certain amount of n-heptane, i.e. in excess of alkane solvent (C. Hansen 2007). This phenomenon will be addressed in the further parts of the report (see 7.7).

In order to remove the asphaltenes from the bitumen, an excess of the alkane solvent is required to force precipitation. The shortest liquid chain alkane with low boiling point, i.e. pentane, is used in the industrial process. The obtained asphaltenes contain some maltenes, thus the product can be also considered a bitumen, hard paving bitumen to be precise. However, the very hard paving bitumens have very little application in cold climate countries such as Finland.

The non-asphaltene component known as deasphalted oil is extracted from the residue (VR, VBR or bitumen) with the alkane solvent, leaving the asphaltenes as solid product or very hard asphalt, depending on the installation and solvent used.

Originally, the requirement to separate the expensive alkane solvent by distillation required large amounts of energy (200 °C). One of the advances for the process development include using the CO_2 as a third component in the blend, which allows for separation of the asphaltenes at lower temperatures, around at 40 °C. The blending of CO_2 and alkane solvent creates a mixture insoluble with deasphalted oil, leading to separation of two liquids and providing the opportunity for decanting of alkane solvent and its reuse (Henning and Glatzmeier 2014; KBR (Kellogg Brown & Root) n.d.)

The deasphalting level depends on the need and can be controlled with the ratio of dilutant to asphaltenes. In refinery the full deasphalting is plausible. In remote areas, such as drilling sites in Canada (J. Speight 2013), partial upgrading by partial deasphalting and visbreaking is used, or suggested for use, in order to achieve a good stability of the crude and lower viscosity, to allow for piping across large distances (Zachariah & de Klerk 2017).

Due to the fact, that asphaltenes are the fraction of crude oil which contains the most Sulfur per mass, deasphalted oil has lower content of Sulfur than the feedstock used for deasphalting. Deasphalted oil can then be used as feedstock into the following installation which benefit from lower sulfur content in the feed, for example hydrocracker or catalytic cracker, visbreaker.

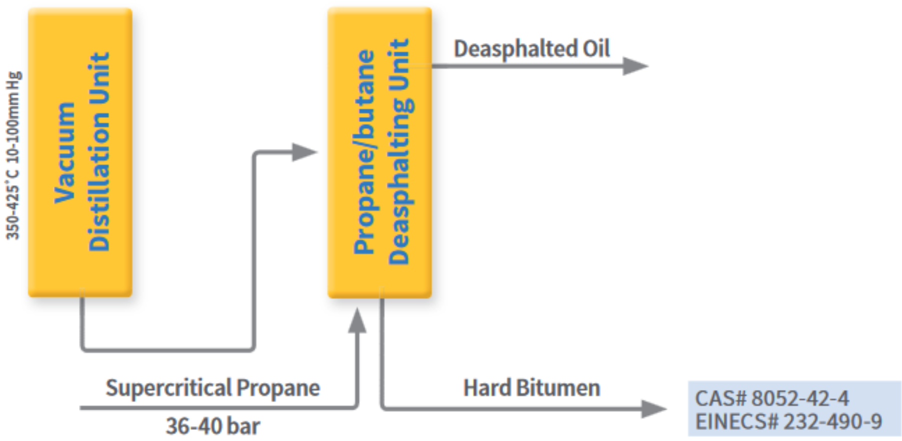


Figure 8. Schematic diagram of the propane deasphalting process (Asphalt Institute Inc. and European Bitumen Association–Eurobitume 2015).

4.5 Future of refineries

It is envisioned that more and more investment will be applied to the full utilization of crude oil into the low boiling point fractions. The coking process allows the creation of the so-called syngas, which can be used as a feed for the Fischer-Tropsch process of production of synthetic fuels. Unless a full shift towards electrical vehicles occurs, the necessity to use combustible fuels may be driving the need for the synthetic fuels production.

The use of the nanosilica particles during the deasphalting process was demonstrated to decrease the Sulphur content in the resulting deasphalted oil

(Guzman, Franco & Cortes 2017). How the nanoparticles would influence the bitumen produced using deasphalting residue is not discussed. In some cases, deasphalting plant may produce asphalt of a hard grade (around Pen = 30 dmm) and bright stock (Tong, Shen & Fang et al. 2017).

Inorganic scavengers, e.g. calcium oxide, are envisioned for use as upgrading particles to reduce the sulfur content (J. Speight 2013). If the calcium oxide is capable of certain reactions with sulfur rich feedstock, perhaps the caution should be applied in using sulfur rich bitumens with aggregates containing this type of inorganic chemical types, e.g. slags.

The developments in desulfurization are predicted to develop in three areas the fastest:

- Development of catalysts for hydrotreating
- Development of reactive adsorbents and redesign of the processes
- Oxidative desulfurization by catalyst and upgrading process

In summary, the decrease of the bitumen production is envisioned as new upgrading systems are invested into, and the influx of the new, bitumen-like products, on the market is predicted.

5 Implication for the bitumen industry

5.1 Availability of paving bitumen

During the surplus of the HSFO on the market in the first months after switch to IMO2020 regulations, the bitumen situation may be in a very good shape. The surplus of HFO would provide the availability of feedstock for the bitumen producing plants at low cost. However, more and more refineries are restructuring their refineries to completely halt the bitumen production and focus on production of lighter cuts, energy and synthetic fuels. (EDIT FROM END OF 2020: This situation in 2020 was affected by the COVID-19 inflicted pandemic. The decreased consumption of aeroplane fuel and personal passenger car fuel lead to changes in demand and supply of the fuels affecting the market prices. Additionally, the OPEC agreement to monitor the prices of fuel on the market was broken and a price war between Russia and Saudi Arabia started in March 2020, which influenced the market. However, due to the possibility to perform more paving operations due to the limited traffic inflicted by COVID-19 and lowered bitumen price, the tonnage demand of bitumen grew.)

Some bitumen producing unit closures in recent years:

- Kuwait Petroleum at Europoort, Rotterdam
- Total's La Mede in southern France
- Colas' Dunkirk plant in northern France
- ExxonMobil and Total bitumen units in Antwerp
- Neste in Porvoo in Finland

BP's decision to end its European bitumen sales operation and close its only remaining bitumen-producing unit, Gelsenkirchen refinery in northwest Germany.

As the COVID-19 pandemic progresses, further permanent refinery closures are observed around the world with the amounts of 1.7 mld barrels per day production capacity (Ahmad Ghaddar 2020).

5.2 Financial

What will be the long term effect to the availability of bitumen and its price remains to be seen. Currently the price of bitumen is expressed as a premium or loss towards the heavy fuel oil. However, the price of HSFO is expected to drop due to oversupply in initial months. The bitumen price will probably be linked to the crude oil, rather than like currently, to the HFSO. However, the preferences towards certain low sulfur crude oils may influence the cost and availability of bitumen.

5.3 Composition

The choice of crude oil type will influence the composition of the final barrel cut, i.e. VR and thus bitumen. Switch to lower sulfur containing crudes may affect the performance of bitumen (Dasek et al. 2017; National Academies of Sciences, Engineering, and Medicine 2017). The use of visbreakers, causes the production of visbroken residue (VBR). VBR was reportedly blended into the paving grade bitumens at various percent as an extender (Dasek, et al. 2017). Technically, the definition of bitumen according to SFS-EN 12597, is not discriminating against the VBR or HSFO as components of bitumen. Both can be non-volatile adhesive and waterproofing materials derived from crude petroleum. However, their performance as paving grade bitumen may differ from the performance of the bitumen obtained from the classical processing of the vacuum residue (Dasek et al. 2017).

5.4 European import-export market

The biggest exporters of bitumen in Europe in 2016 are presented in Table 3.

Table 3. The situation on the bitumen market in numbers in the year 2016 in terms of bitumen production, consumption for non-energy uses, export and import. The entries for the geographically European countries are bolded. Source: UN data (UN data 9).

	Bitumen production		Bitumen consumption for non-energy uses		Bitumen trade – import		Bitumen trade – export	
No.	Country	1000 metric tons		1000 metric tons		1000 metric tons		1000 metric tons
1	China	21716	China	25958	China	4946	Korea, South	3560
2	United States	19721	United States	21254	United States	2234	Singapore	3331
3	**Russia**	**7633**	India	5867	Indonesia	1316	Iran	3169
4	India	5185	**Russia**	**5660**	**France**	**1219**	**Russia**	**2009**
5	Korea, South	5064	Canada	3534	India	955	Canada	1982
6	Iran	4837	**Turkey**	3370	Saudi Arabia	587	**Germany**	**1819**
7	Canada	4763	Saudi Arabia	3365	Australia	571	**Spain**	**1694**
8	**Germany**	**4065**	**France**	**2525**	Canada	531	United States	1226
9	**Turkey**	**3436**	**Germany**	**2273**	**United Kingdom**	**530**	**Italy**	**1199**
10	Singapore	3432	Brazil	1876	**Romania**	**523**	**Sweden**	**663**
11	Japan	3249	**Italy**	**1478**	Morocco	427	**Greece**	**499**
12	Saudi Arabia	2778	Korea, South	1433	**Ukraine**	**413**	**Netherlands**	**492**
13	**Italy**	**2557**	**United Kingdom**	**1425**	**Norway**	**411**	**Belgium**	**480**
14	**Spain**	**2471**	Indonesia	1318	**Netherlands**	**405**	**Poland**	**479**
15	Brazil	1955	**Poland**	**1170**	**Czech Republic**	**329**	**Belarus**	**426**
16	**France**	**1706**	Japan	1170	**Austria**	**278**	**Hungary**	**415**
17	**Poland**	**1427**	Thailand	1127	**Switzerland**	**277**	Bahrain	402
18	Thailand	1364	Mexico	955	**Ireland**	**256**	Thailand	297
19	**Belgium**	**1271**	**Belgium**	**889**	Algeria	255	**Czech Republic**	**223**
20	Mexico	982	Iran	745	**Sweden**	**228**	Malaysia	220
21	**United Kingdom**	**968**	**Spain**	**740**	**Poland**	**227**	**Serbia**	**219**
22	**Sweden**	**961**	Egypt	605	Burma	224	China	205
23	**Greece**	**748**	**Romania**	**601**	Tunisia	213	Côte d'Ivoire	195
24	**Belarus**	**677**	Kazakhstan	574	**Denmark**	**200**	**France**	**181**

	Bitumen production		Bitumen consumption for non-energy uses		Bitumen trade – import		Bitumen trade – export	
No.	Country	1000 metric tons		1000 metric tons		1000 metric tons		1000 metric tons
25	Egypt	605	Venezuela	473	Philippines	151	**Austria**	**144**

The bitumen export from European countries is the highest from Russia. However, the data provided above does not specify in to which direction the export is happening. It is highly likely that China is the importer of the read product from Russia.

However, despite the production of bitumen being relatively sufficient to supply the European Union region, it is worth underlining that the source of the crude oil used for the production in the European refineries can still be of different origin. Political disturbances are likely to affect the price and availability of the bitumen as well.

5.5 Nordic context

The refinery which provided the product to the small market of Finland, closed the bitumen production line (Neste Corporation 2016). The same company still operates the refinery in Naantali as of 2019 (edit: closed in 2021). However, the upkeep of the installation may be costly, especially if the other side products from this refinery such as HFO have no demand from other industries other than those requiring bitumen for operation. It is envisioned that this refinery will not be able to compete economically with other providers of bitumen. One may expect a significant premium price for the bitumen produced locally, or expect sudden increase of importance of the import of the bitumen from other refineries.

This opens rather hermetic bitumen market of Finland to influx of imported bitumens. Expected is the influx from refineries located in the geographical proximity to Finland.

Considering the geographical proximity to Finland, the likely imported good would come from Russia, Germany, Sweden, Netherlands, Belgium, Lithuania and Poland. Considering the situation of own production and consumption as well as import situation into the country, we could assume that Russia, Germany and Belgium are exporting mostly bitumen of own production, while Sweden and Poland to some extend may export partially imported bitumen. The situation of Netherlands indicates the problem associated with harbor and transit country. Netherlands exports over twice as much as it produces, and about 3.5 times more than it consumes. Majority of the bitumen coming from Netherlands is expected to originate elsewhere, which may be associated with highest risk or variability of product and increased problems with its tracing.

6 Bitumen Characterization Techniques - Part 2

6.1 Objective

The objective of this review was the evaluation of the bitumen characterization techniques available on the market but not yet used routinely in Finland by all asphalt producers, and identification of the ones most easily adjustable to become used in field. Therefore, the measurements such as for example Softening Point, Penetration and Fraass are not a subject of this report, as they already are used as characterization techniques in Finland, are well known to the practitioners and are described at lengths in other publications in both Finnish (Aromaa 2016; Blomberg, Bitumit 1990) and English (Blazejowski, Wojcik-Wisniewska & Baranowska 2018; Hunter, Self & Read 2015). However, few words were included about the standardized tests suggested in Finnish Asphalt Specifications for Polymer Modified Bitumen, as the knowledge related to those techniques is not homogenous within the local industry.

The objective was evaluated based on the:

- Potential to answer to the interesting question (performance and differentiation of the products at hand)
- Price of the equipment
- Stability in field conditions (the level of training of operators and management of laboratory conditions)
- Availability of portable solutions,
- Measurement time and price.

However, the text of hereby report is targeted towards practitioners of asphalt industry, who may not have a strong theoretical background in physics and chemistry. The main point and aim was to bring understanding of the methodology rather than to focus on very detailed description of academical theories connected with discussed phenomena. For this reason, the report can be used as a training material for students starting in the asphalt industry. The explanation of concepts, nomenclature connected with each methodology and reported use of the technique in applied sciences was covered within each section, with examples if possible.

The reader is advised to search further information in appropriate books and publications in which more detailed information and explanation of theories can be found and to extend the search using the keywords connected to each subject. The author attempted to provide the references to wide range of sources, but acknowledges that most likely more research exists on each of the touched subjects. The effort was placed on referring to the appropriate sources where more detailed information is provided, and when possible, review papers, round robin and consensus reports. The omission of sources (if noticed) is not done on purpose, but rather due to the time and resource restrictions within the project.

7 Characterization techniques

To start, the composition of bitumen can be described as a mixture of organic molecules, which are susceptible to oxidation. Due to that fact, the exposure to oxygen, heat and ultraviolet light is inflicting chemical changes in the mixture (J. C. Petersen 2001). Those changes influence the response of the material to physical stimulus (such as traffic or temperature) over time (J. C. Petersen 2001). Therefore, to predict the behavior of bitumen in field, one should investigate its behavior as a fresh material as well as after aging. Aging is defined and simulated as an exposure to oxidation at elevated temperatures.

The below review focuses on chemical changes in the material, rather than physical hardening, which can also be considered a contributing factor to aging, if it was defined as an increase of the viscosity with time. More information on that aspect can be found in (Lesueur 2009; J. C. Petersen 2001; Hunter, Self & Read 2015).

7.1 Chemical aging susceptibility

Over the course of multiple decades, the development of methodologies, which would artificially age the bitumen to the state similar to that observed in field, were developed and include:

- RTFOT (Whiteoak 1990)
- TFOT
- PAV
- 2XRTFOT
- UV resistance testing
- Oxidation on the FTIR prism (Makowska, Hartikainen & Pellinen 2017)
- Lisbone ageing machine (Crucho et al. 2020)
- Vienna aging machine (Maschauer et al. 2019).

7.1.1 Rolling Thin Film Oven Test (RTFOT)

The Rolling Thin Film Oven Test (RTFOT) is standardized in Europe under SFS-EN 12607-1 "Determination of the resistance to hardening under influence of heat and air. Part 1. RTFOT method". The test is part of the binder evaluation protocol according to the Finnish Asphalt Specifications (Finnish Pavement Technology Advisory Council 2017) and is assumed to simulate the short-term aging of bitumen due to exposure to air at high temperature during production, loading, transport and paving. The bitumen after the RTFOT processing is similar (as characterized by Penetration and Softening Point) to the binders extracted and recovered from asphalt in field of approximately 3 month age (Whiteoak 1990).

The principle of the test is in heating a certain portion of bitumen to 163 °C (temperature assumed during spraying of binder in asphalt production facilities) and rolling the bottles to assure thin film on walls of the bottles is spread. A stream of air is applied into each bottle with every covered cycle of rolling for a period of 75 minutes.

7.1.2 Thin Film Oven Test (TFOT)

Thin Film Oven Test is an older version of the RTFOT, which was used to predict resistance to short-term aging in Finland, especially still during the ASTO research program 1989–1993 (Kurki, Halttunen, et al. 1993). The method is standardized under *SFS-EN 12607-2:en Determination of the resistance to hardening under influence of heat and air – Part 2: TFOT method.*

The principle of the test is again in keeping the binder in the form of thin films at elevated temperatures, when the flow of air is supplied to the oven. However, the samples are spread on the plates rather than inside of the rolling bottles and thus the binder is more static during the oxidation itself.

7.1.3 Pressure Aging Vessel (PAV)

Pressure Aging Vessel (PAV) was designed as the aging procedure to simulate the conditions the binder would be exposed to during the long-term performance, namely pressure and air (Whiteoak 1990). The standard for the PAV aging procedure can be found from the *AASHTO R28 Accelerated Aging of Asphalt Binder Using a Pressurized Aging Vessel (PAV).* The aging temperature and pressure depend on the grade of the binder tested, usually the test is performed for 20 h. The test is expected to inflict changes in binder that correspond to approximately 3–4 years in field. But more often, 40 h aging is found to represent the materials behavior in field for up to 5 years (Asphalt Institute Technical Advisory Committee 2019).

7.1.4 Triple RTFOT (3xRTFOT)

Because the investment in PAV equipment may be a financial strain on some laboratories which routinely are not performing PAV measurements, or in developing countries, an attempt was made to evaluate if multiple aging by RTFOT would result in similar binder characteristics as those obtained using PAV aging procedure. It was suggested that the time of RTFO test should be expanded by about three times in order to achieve the binder of a penetration and softening point values similar to those obtained using PAV (Siddiqui & Ali 1999). The chemical composition of such materials may differ but using the rheological traditional binder characterization techniques (Penetration and softening point) the binders are comparable. Some researchers using this approach refer to the test as “extended thermo-oxidative aging” and abbreviate it as 3xRTFOT (Dasek et al. 2017; Pellinen & Makowska 2018).

7.1.5 Performance grading (PG)

The Performance grading (PG) system known as PG-grading is focused on assuring performance of the binder at different temperatures and during the one life cycle (Dore & Zubeck 2009; Little, Allen & Bhasin 2018).

The PG-grading is evaluating the performance of the binder taking account its influence on rutting, fatigue and resistance to low temperature cracking after long term aging (Anderson et al. 1994). The assumption is that fresh binder is not likely to crack due to thermal loading or fatigue. The effect is visible after the pavement was in field for multiple years and hence the onset of cracking should be linked to the aging resistance[1]. Therefore, the application of PAV tests before testing the parameters of the binder such as stiffness, complex modulus and elasticity are suggested. Likewise, to evaluate the resistance to rutting, a binder which is corresponding in properties to that laid down in field, i.e. short-term aged, is to be tested after RTFO for resistance to rutting.

In comparison, the currently applied binder gradation in Finland evaluates resistance to cracking of the binder based on a fresh binder and determines the resistance of binder to only short-term aging (Figure 9).

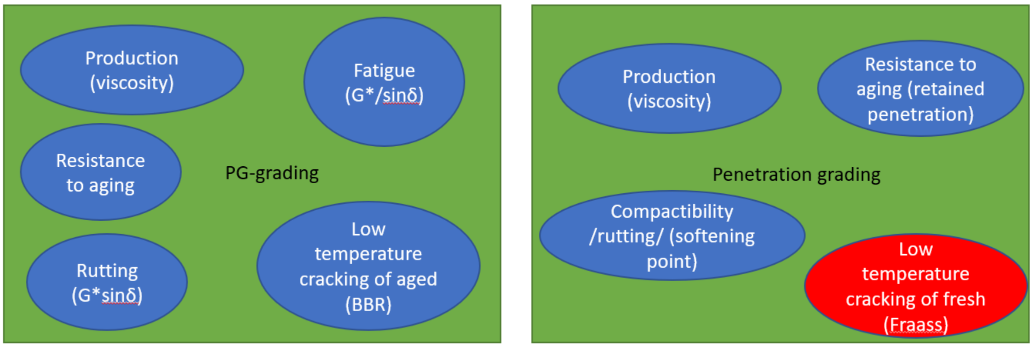


Figure 9. Comparison of the PG and Penetration grading systems in terms of their relation to performance parameters.

The PG-grading system was developed during the SHRP in 1993 and since then was adapted by different Transportation Agencies in U.S.A., but also worldwide. At the time of the PG-grading system introduction, the use of polymer modified binders was not common. The system was therefore expanded by additional set of tests in the following years that focus on properties that can be improved by the incorporation of polymers into the asphalt concrete composition.

The importance of this gradation system is not only in testing of the performance parameters, but in testing them at temperatures during which the pavement is expected to be performing. An average of the high temperatures recorded in the region within 7 years is recalculated into the highest probable temperature to be observed at the depth of 2 cm from the surface (XX in PG-XX-YY). Likewise, the lowest recorded temperature within the region is considered the critical temperature (YY in PGXX-YY). (Little, Allen & Bhasin 2018; Pszczola, Rys & Jaskula 2017).

Once it is established that a binder, for example used in the pavement in Helsinki region, would require to meet the requirements of the PG-58-22. A series of tests is then conducted on the fresh and aged binders to assure performance at 58 and -22 °C.

1 Finnish Asphalt Specifications and Penetration grading system require the Fraass cracking temperature to be measured strangely from fresh binder.

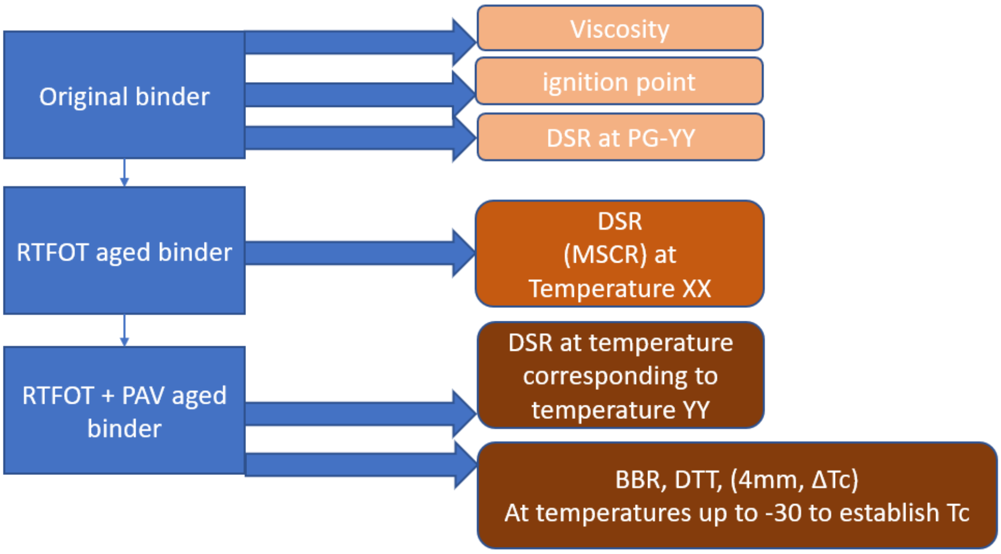


Figure 10. Schematic of the PG-system characterization for the binder.

The main difference between the Penetration or viscosity grading systems and PG system, is that PG indicates in the grade in which conditions the material should be used and its properties at specific temperatures will be tested (see Figure 11). The designer gets a clear picture if the chosen binder is suitable for the conditions in which the road will be operating.

In comparison: assigning the grade to bitumen as 70/100 means just that the penetration is on that level. The grade itself in such case does not limit or suggest in which types of pavements the material should be used, and it is the designers experience which dictates the use of certain grade. The lack of specification of the rules of use of binders in the mix design in Finnish asphalt Specifications is assuming the correct level of training of the workers in the industry, but therefore leaves a possibility for an error.

7.1.5.1 PG Temperature range determination – design grade determination

To determine what type of binder to choose for the layer in the bound structures depends on the lowest and highest recorded temperatures in the region in which the road is to be constructed according to the PG-system.

Grade	**PG46**			**PG52**							**PG 58**					**PG 64**					
Average 7-day Max pavement design Temperature [°C]	<46			<52							<58					<64					
Min Pavement Design Temperature [°C]	-34	-40	-46	-10	-16	-22	-28	-34	-40	-46	-16	-22	-28	-34	-40	-10	-16	-22	-28	-34	-40
ORIGINAL BINDER (unaged)																					
Flash point temperature, Min [°C]	230																				
Viscosity, ASTM D4402:, Test temperature 135[°C]	Max 3Pas																				
Rutting parameter, G*/sinδ> 1kPa, at 10rad/s (1.59 Hz), [°C]	>46			>52							>58					>64					
Rolling Thin Film Oven Test + analysis of residue																					
Mass loss, mass %	<1																				
Rutting parameter, G*/sinδ> 2.2kPa, at 10rad/s (1.59 Hz), [°C]	>46			>52							>58					>64					
Pressure Aging Vessel residue																					
Aging Temperature during the test [°C]	90			90							100					100					
Physical Hardening	Report																				

Grade	PG46			PG52							PG 58					PG 64					
Fatigue parameter, G*sinδ<5 000 kPa at 10 rad/s(1.59 Hz), [˚C]	10	7	4	25	22	19	16	13	10	7	25	22	19	16	13	31	28	25	22	19	16
Creep Stiffness Test (AASHTO T 313 + AASHTO PP 42), [˚C]	-24	-30	-36	0	-6	-12	-18	-24	-30	-36	-6	-12	-18	-24	-30	0	-6	-12	-18	-24	-30
Direct Tension Test result (AASHTO PP 42) [˚C]	-24	-30	-36	0	-6	-12	-18	-24	-30	-36	-6	-12	-18	-24	-30	0	-6	-12	-18	-24	-30

Figure 11. The basic performance grading classification system for softer grades of bitumen after (Dore & Zubeck 2009).

The maximum pavement temperature (Pszczola, Rys and Jaskula 2017) is calculated from the equation:

$$T_{max}^{d} = 54.32 + 0.78 * T_{air} - 0.0025 * \phi^2 - 15.12 * log_{10}(d + 25) +$$

$$+ z(9 + 0.61 * \sigma_{air}^2)^{0.5} \quad (1)$$

where

T_{max}^{d} = the maximum pavement temperature at depth d in the pavement, the upper PG value [°C]
T_{air} = the mean value calculated from the maximum 7-day averaged daily the highest air temperatures for the particular years
ϕ = the latitude of the metrological station [°]
d = the design depth of the layer temperature, see Figure 12 b
σ_{air} = the standard deviation calculated from the maximum 7-day averaged daily the highest air temperatures for the particular years [°C]
z = a statistical quantity resulting from the normal distribution of the temperature values, such as the “z testisuure” in the Finnish Asphalt Specifications, e.g. z = 0 for probability of 50% results falling within the average +/- standard deviation, z = 0,84 means that 80% of the averages fall within the range, and z = 2,05 means that with 98% probability the averages fall within the range.

The minimum pavement temperature is calculated from equation:

$$T_{min}^{d} = -1.56 + 0.72 * T_{air} - 0.004 * \phi^2 + 6.26 * log_{10}(d + 25) +$$

$$- z(4.4 + 0.52 * \sigma_{air}^2)^{0.5} \quad (2)$$

where

T_{min}^{d} = the minimum pavement temperature at depth d in the pavement, the lower PG value [°C]
T_{air} = the mean value calculated from the minimum annual air temperatures for the particular years
ϕ = the latitude of the metrological station [°]
d = the design depth of the layer temperature, Figure 12 a
σ_{air} = the standard deviation calculated from the minimum annual air temperatures for the considered years [°C]
z = a statistical quantity resulting from the normal distribution of the temperature values, such as the "z testisuure" in the Finnish Asphalt Specifications, e.g. z = 0 for probability of 50% results falling within the average +/- standard deviation, z = 0,84 means that 80% of the averages fall within the range, and z = 2,05 means that with 98% probability the averages fall within the range (Pszczola, Rys & Jaskula 2017).

After the high temperature is determined, the number will be rounded to the value in the sequence of numbers starting from 46 by adding 6, i.e. 46, 52, 58 and so on. This number will be referred hereby as XX, and will correspond to the place in schematic PG grade expressed as the PGXX-YY. After the low temperature is determined, the number will be rounded to the value in sequence of numbers starting from -10 by withdrawing 6, i.e. -10, -16, -22 and so on. This number will be marked as YY in the PGXX-YY.

7.1.5.2 Effect of loading on the PG

Due to the more pronounced traffic loading on the structure, the binder should have better resistance to permanent deformation. This was typically achieved by upgrading the higher PGXX by one or two levels, depending on the traffic levels (Pszczola, Rys & Jaskula 2017) and nature of traffic, e.g. bus stop, street of slower traffic or highway. The higher PGXX assures that the rutting criteria is stricter, because it needs to be fulfilled at higher temperature (a binder is typically softer at higher than at lower temperatures). Specific example discussing how to adjust PG temperatures is described in the following section.

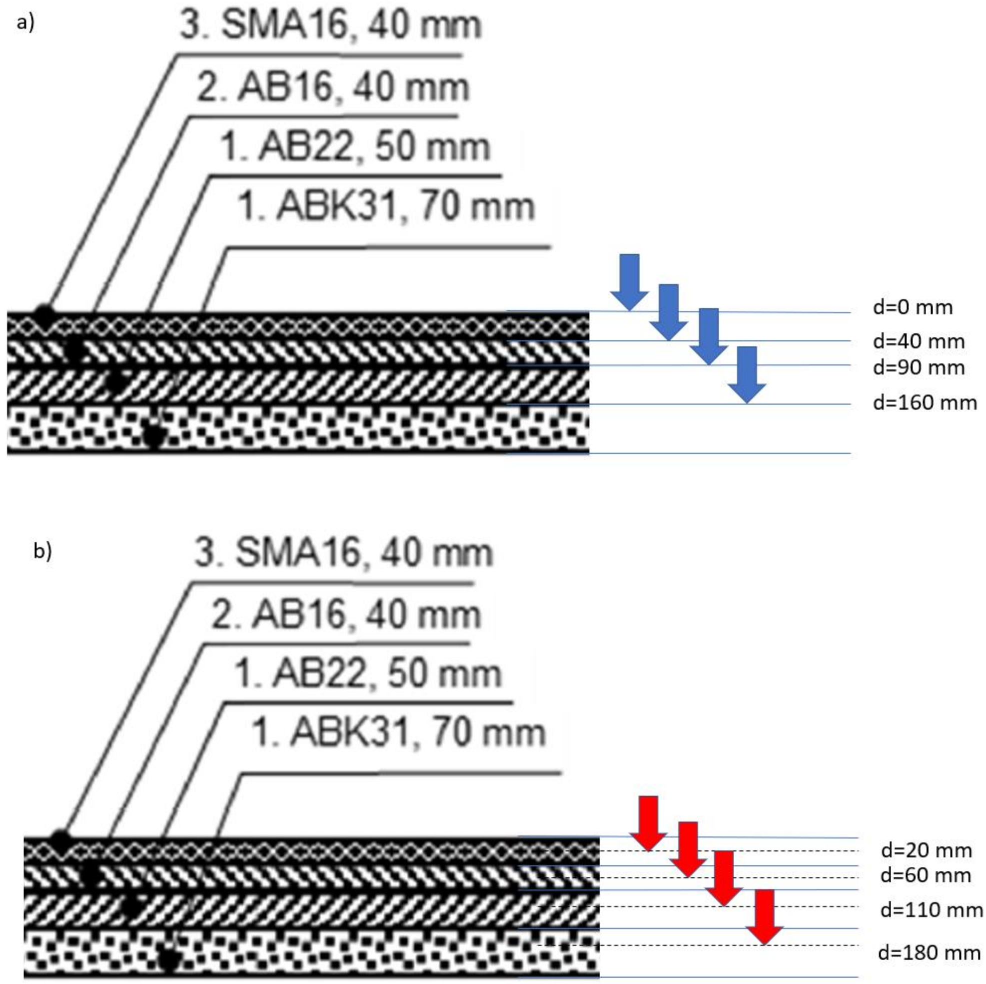


Figure 12. The suggested Design depth (d) in each layer for a) minimum (blue arrows) and b) maximum (red arrows, 20 mm below top of the layer, on the example in the Road Design Guidelines for the traffic class 25,0 (Liikennevirasto 2018) based on suggestions presented by (Pszczola, Rys & Jaskula 2017) for Polish conditions.

7.1.5.3 Example of design PG determination

The average meteorological data is to be gathered from 20 years, but due to the availability of the open access meteorological data from the Finnish Meteorological Institute / Ilmatieteen laitos (downloaded on 2019-07-30) for only the past 10 years, let's consider the example of Helsinki Kaisaniemi with the geographical location of 60.1754° N, 24.9453° E (Figure 13 and Figure 14) using the last 10 year records.

The parameters necessary to conduct calculations using equations 1 and 2 are calculated and presented in Table 4. Based on those results the base PG grade is assigned for each layer in Table 5, and the PG grade upgrade as a result of the traffic type and depth in the pavement is presented.

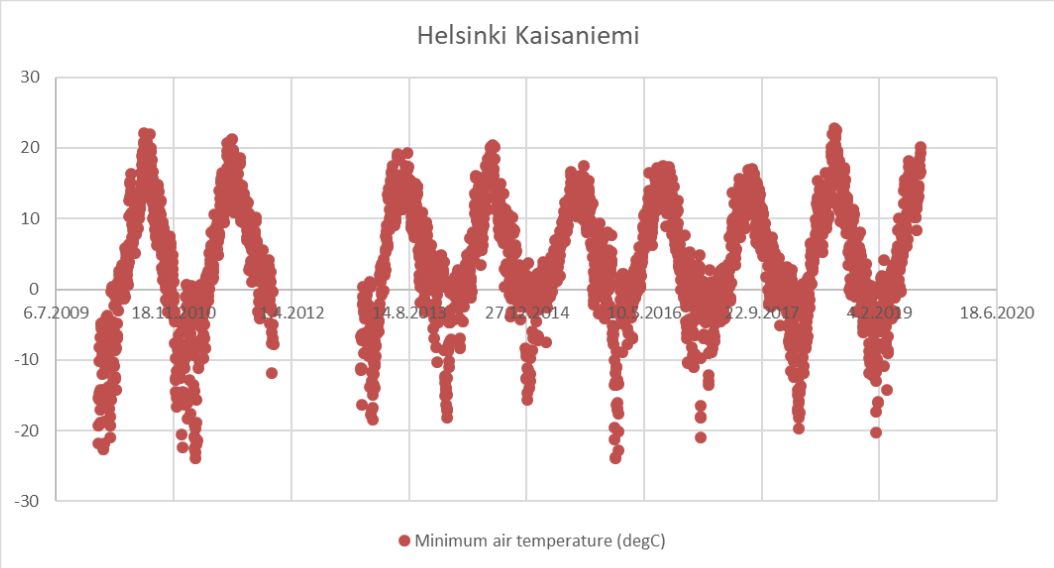


Figure 13. The minimum air temperatures in Helsinki Kaisaniemi between 1.1.2010–31.7.2020 (source: Finnish Meteorological Institute / Ilmatieteen laitos, downloaded on 2019-07-30).

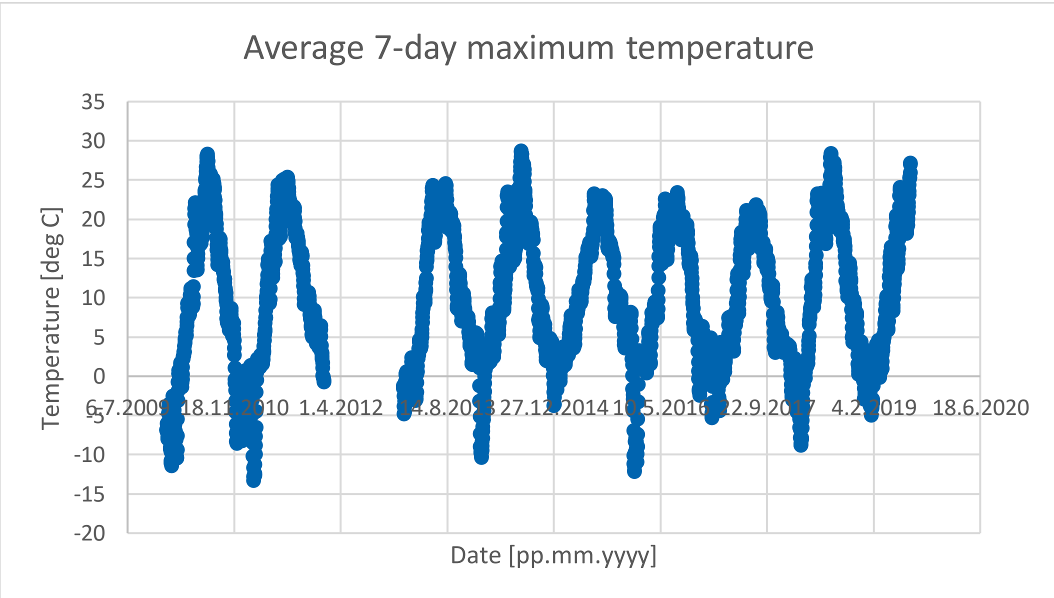


Figure 14. The moving 7-day average maximum air temperatures recorded in Helsinki Kaisaniemi between 1.1.2010–31.7.2020 (source: Finnish Meteorological Institute / Ilmatieteen laitos, downloaded on 2019-07-30).

The assignment of grades (Table 5) is performed as a result of the AASHTO suggestions. Would this be appropriate for Finnish types of mixtures, remains to be established.

In the following step the designer needs to evaluate if the binder meets the rutting, fatigue and thermal cracking performance criteria at the chosen temperatures for a given layer.

Table 4. The processed data for the particular years in database of ITL for the period of 1.1.2010–31.7.2019, calculated at 98% probability (that such temperature occurs once every 5 years).

	2010	2011	2013	2014	2015	2016	2017	2018	2019	Av.	St. dev
$T_{7d\text{-}max}$	28,34	25,42	24,57	28,71	23,27	23,41	21,85	28,42	27,17	**25,6**	**2,57**
T_{min}	-22,6	-23,9	-18,4	-18,2	-15,7	-23,9	-21	-19,7	-20,3	**-20,4**	**2,76**

Table 5. Estimated desired grades of binders to be used in different layers according to the Finnish Road Design Guidelines for Helsinki Kaisaniemi with 98% probability.

Calculated for layers	Finnish Traffic class "Kuormitus-luokka"	Type of traffic	SMA16	AB16	AB22	ABK31
T^d_{max}			47,7	43,5	40,5	37,7
T^d_{min}			-27,9	-25,3	-23,8	-22,5
Calculated design grade based on environment			52-28	46-28	46-28	40-28
Upper Grade bumping for <0.3 mln ESAL	0,1–0,4 PAB/SOP	Normal traffic	52-28	46-28	46-28	40-28
		Slow traffic	52-28	46-28	46-28	40-28
		Standstill (+1)	**58-28**	**52-28**	**52-28**	**46-28**
Upper Grade bumping for 0.3–30 mln ESAL	0,8–25,0 PAB/ AB/ SMA	Normal traffic	52-28	46-28	46-28	40-28
		Slow traffic (+1)	**58-28**	**52-28**	**52-28**	**46-28**
		Standstill (+2)	**64-28**	**58-28**	**58-28**	**52-28**
Upper Grade bumping for >30 mln ESAL	25,0 SMA and above	Normal traffic (+1)	**58-28**	**52-28**	**52-28**	**46-28**
		Slow traffic (+1)	**58-28**	**52-28**	**52-28**	**46-28**
		Standstill (+2)	**64-28**	**58-28**	**58-28**	**52-28**

7.1.6 Dynamic Shear Rheometer (DSR)

The principle of work of DSR equipment in the context of bituminous materials is covered by (Aromaa 2016) in Finnish language and the reader is advised to read through the section 2 of that work. Other good sources in English include Whiteoak (1990) and Mezger (2002) as well as Hunter, Self & Read (2015).

Below are reprinted some of the figures (Figure 13) that allow for better understanding of values collected during the measurement. The values discussed in this work will comprise:

- Complex modulus (|G*|)
- phase angle (δ)
- damping factor (tanδ)
- Loss modulus (G'')
- Storage Modulus (G')

- frequency (f [Hz])
- radial frequency (ω[rad/s]),
- Complex viscosity ($\eta^*=|G^*|/\omega$)
- Shear stress (τ)
- Shear rate ($\dot{\gamma}$)
- Strain (ε, percent displacement to the gap height)
- Zero Shear Viscosity ($\eta^*(\dot{\gamma}\rightarrow 0)$).

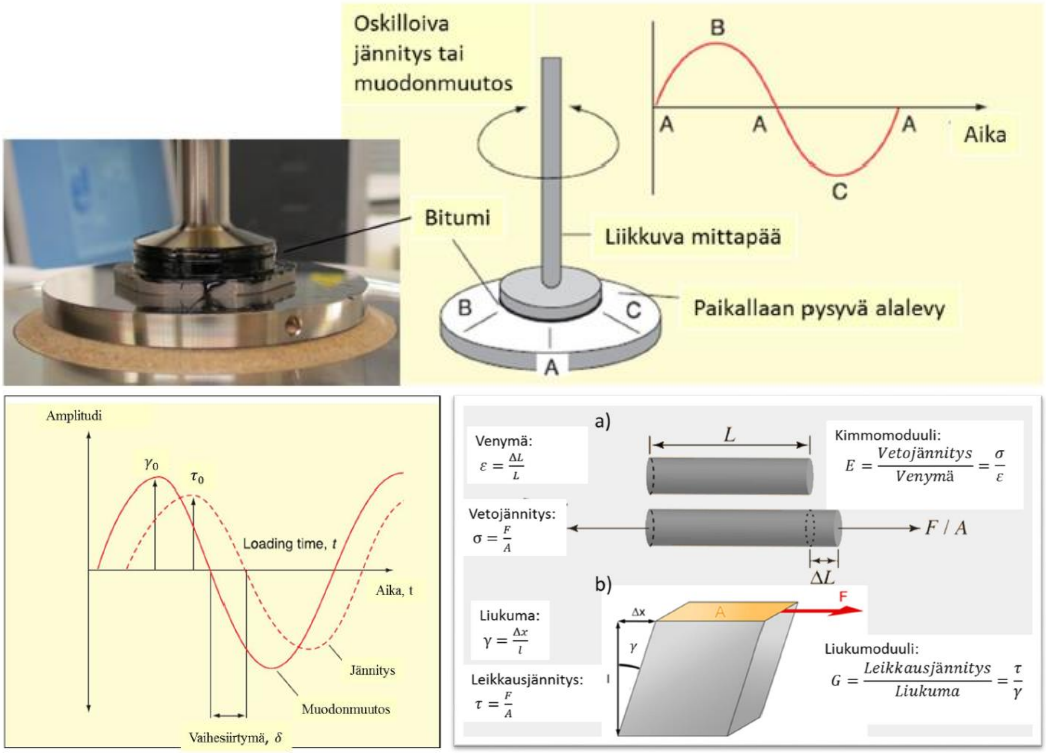


Figure 15. The translation into Finnish of the parameters determined during the measurement by dynamic shear rheometer. Reprinted from Aromaa (2016) after Whiteoak (1990).

Some of the most important things to remember when reading further is that:

- the higher the complex modulus, the stiffer the material is
- the phase angle on the other hand describes the relation between viscous and elastic behavior in the material (the point of equal contribution could be assigned the value of 45° (sometimes referred to as a cross-over point), while 0° means fully elastic behavior and 90° fully viscous behavior (liquid like)).

7.1.6.1 PG specific parameters

As discussed in the section 7.1.5 after the evaluation of the thermal conditions in which the road is expected to perform, a series of tests is to be applied at the temperatures chosen to evaluate its performance. Initially the PG grading was based on the so-called rutting parameter but over time the trust in its rutting predictive characteristics declined. A Multiple Stress Creep Recovery test was developed to test the binders resistance to permanent deformation more precisely. The MSCR is also capable of providing information on recovery of shape after loading, which originally was tested using the direct Tension Test (DTT). It is therefore simpler and more preferred test to assure presence of polymer in the PmB. The fatigue parameter is evaluated from samples which are laboratory aged – typically using the RTFOT followed by PAV system. Choice of aging time and temperature is defined in PG specifications.

7.1.6.1.1 Rutting parameter

Below the binder of the Penetration grade 70/100 on the Finnish market used in the previous projects (Pellinen & Makowska 2018) will serve as an example to demonstrate how the assignment of the PGXX-YY can be done based on the DSR data to start. The DSR data obtained in that project is hereby reprocessed.

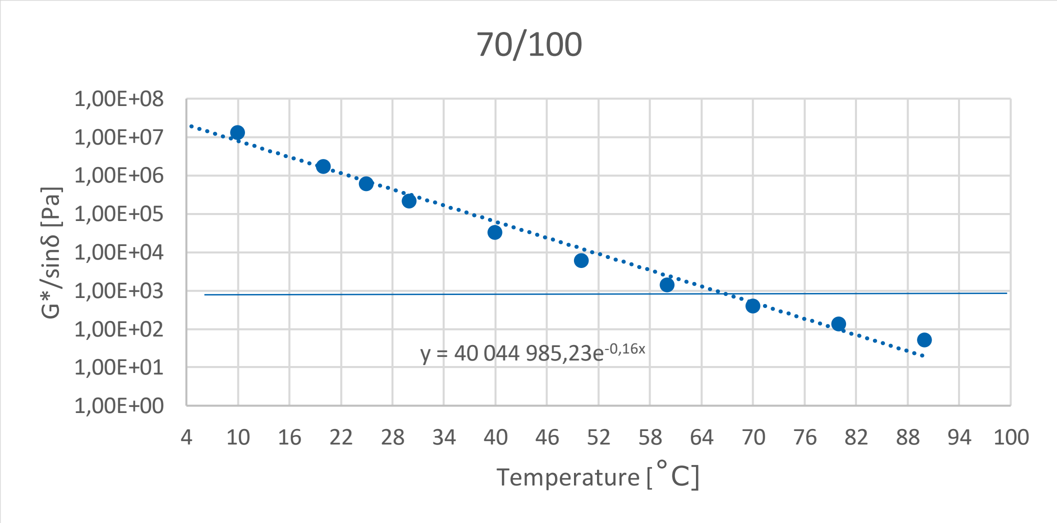


Figure 16. The Rutting parameter measured at 1,59Hz (10 rad/s) frequency across the temperature range for fresh bitumen 70/100 with the limiting criteria of 1000 Pa.

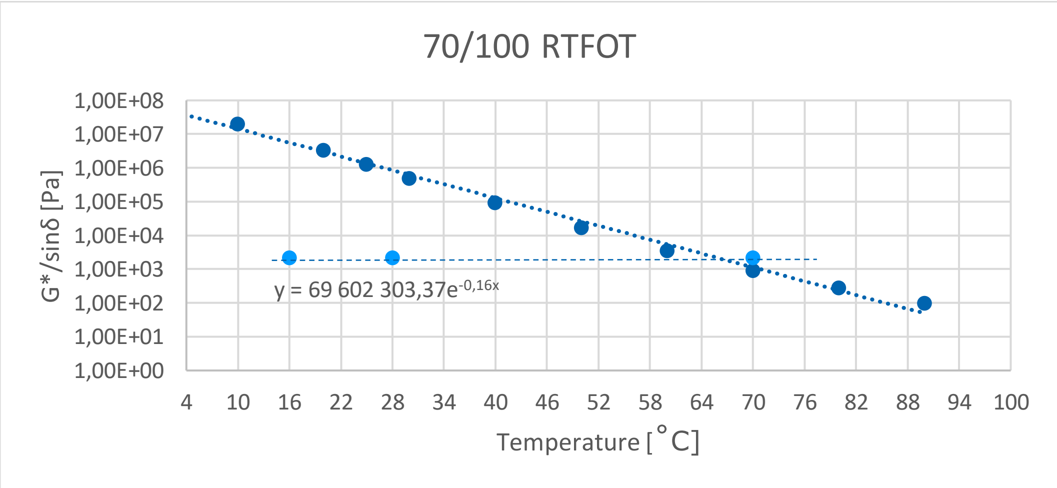


Figure 17. The Rutting parameter measured at 1,59Hz frequency across the temperature range for the RTFOT aged bitumen 70/100 with the limiting criteria of 2200 Pa.

In order to resist rutting, it was proposed that the fresh binder should be characterized with the value of above 1 kPa at the PGXX temperature when fresh, and 2.2 kPa at PG-XX when RTFOT aged, for the rutting parameter (G*/sinδ).

In the above presented case, the binder could be sufficiently assigned the grade PG-XX as PG-58-YY. It is important to underline that, it would meet the requirements of the PG-52 and PG-46 in that matter as well.

7.1.6.1.2 Fatigue parameter

As mentioned before, in lieu of RTFOT+PAV test, our laboratory used the 3xRTFOT aging on the bitumen 70/100.

The fatigue parameter (G*sinδ) should not exceed the value of 5000 kPa at temperatures defined in the specifications for the RTFOT+PAV aged sample, see Figure 18 for an example of the assignment.

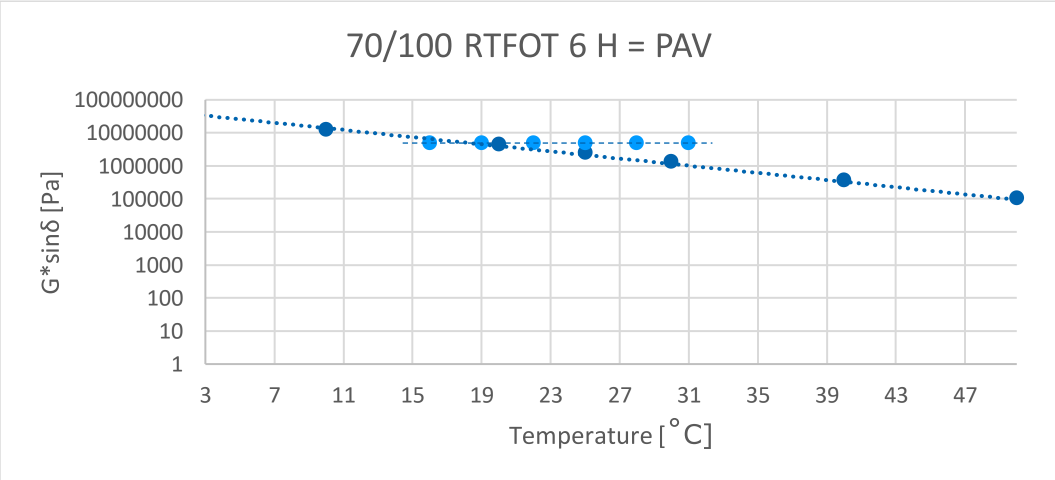


Figure 18. Fatigue parameter measured at 1,59 Hz of 70/100 3xRTFOT plotted against temperature and the limiting criteria of 5000 kPa.

Based on the above DSR data we could determine that our binder after long term aging meets the criteria for fatigue parameter at temperatures above 19. This way using the table presented in Figure 10 we can assign it the grades:

- PG58-28
- PG58-22
- PG58-16
- PG52-22
- PG52-16
- PG52-10

At this point to establish what is the true low grade the BBR and DTT test should be still performed.

The extension to the basic PG system is the test of multiple stress creep recovery (MSCR) which allows to also include the traffic level aspect into the design process and choice of binder.

7.1.6.1.3 Multiple Stress Creep Recovery (MSCR) and extended PG

The weak side of the original PG system is that it was developed just before the polymer modified binders became widespread and thus it does not specifically distinguish between modified and unmodified binders very well. The BBR and DTT tests were added for this reason to PG system to allow for identification of the polymer modification by evaluating sufficient stiffness, resistance to rutting or tensile strength. The shapes of the samples and results of the DTT were indicative of modifier presence. However, the test is relatively laboursome and requires additional equipment in the laboratory.

Because of that, a test called Multiple Stress Creep Recovery (MSCR) was developed (D'Angelo et al. 2007). The test is performed in the DSR equipment, which allows for reuse of the same sample investigated with using 25 mm plate-plate geometry for rutting and fatigue parameter, if only a small sample is available.

The sample is preloaded 10 times for 1 second with the shear stress of 0.1 kPa, followed by 9 seconds of recovery (Figure 19). After such initial preloading, the measurement commences and comprises of:

- 10 cycles of 1 second under load 0.1 kPa and 9 seconds recovery (no applied stress)
- 10 cycles of 1 second under load of 3.2 kPa and 9 seconds recovery (no applied stress).

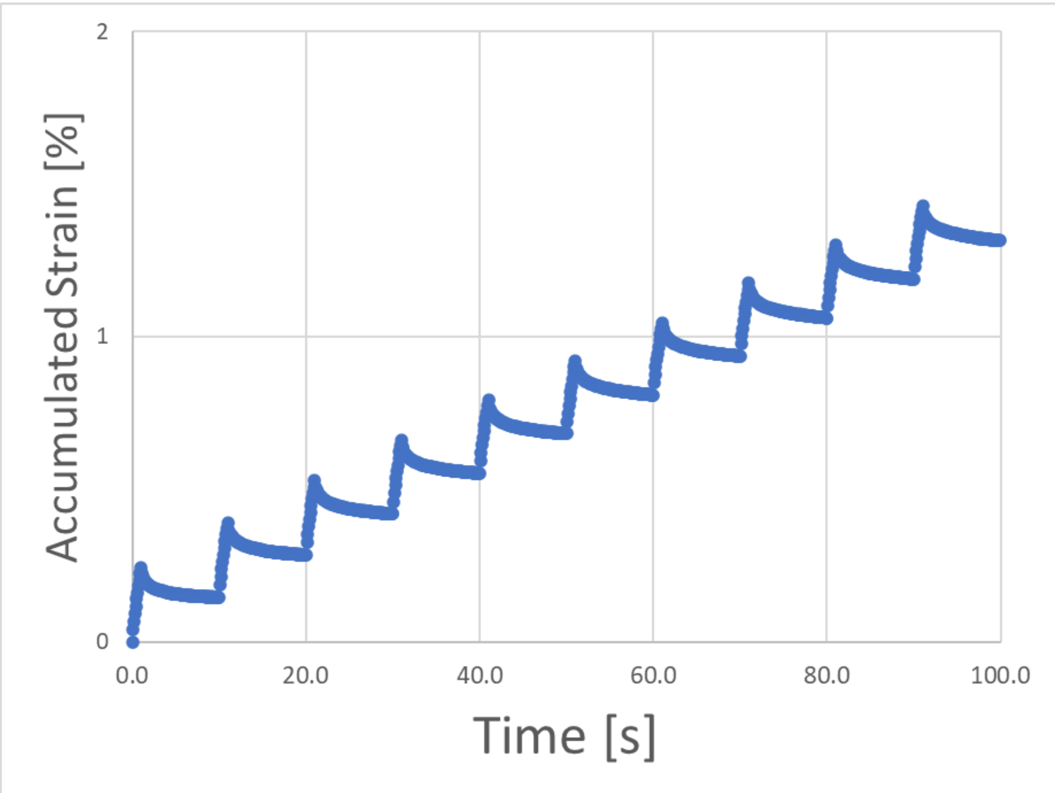


Figure 19. Example of the accumulated strain during preloading cycles demonstrating the viscoelastic response of the binder under stress and its release (own data).

Below is an example of results from the PmB1 and Bit1 bitumen samples from the RILEM2020_RR performed in the Aalto university (Porot, Vanstenkiste et al. 2021). Often the MSCR test is only performed on modified binder. But it is important to understand how the PmB differ from unmodified binders, especially what does the designer gain by using the PmB. Also in the opinion of the author of hereby report, the result of MSCR test at lower temperatures of performance is indicative of the performance mechanism of soft binders in low temperature climate.

Based on the obtained results the classification of the binder as modified or unmodified can be achieved. We can see that the PmB1 is expressing higher recovery of the strain (Figure 20 and Figure 22) at 64 °C than the Bit1 is at 52 °C (Figure 21 and Figure 23). This means that the bitumen Bit1 is more susceptible to deformation than PmB. Should the performance be required to be fulfilled at higher PG-XX, the suggestion would be to choose PmB.

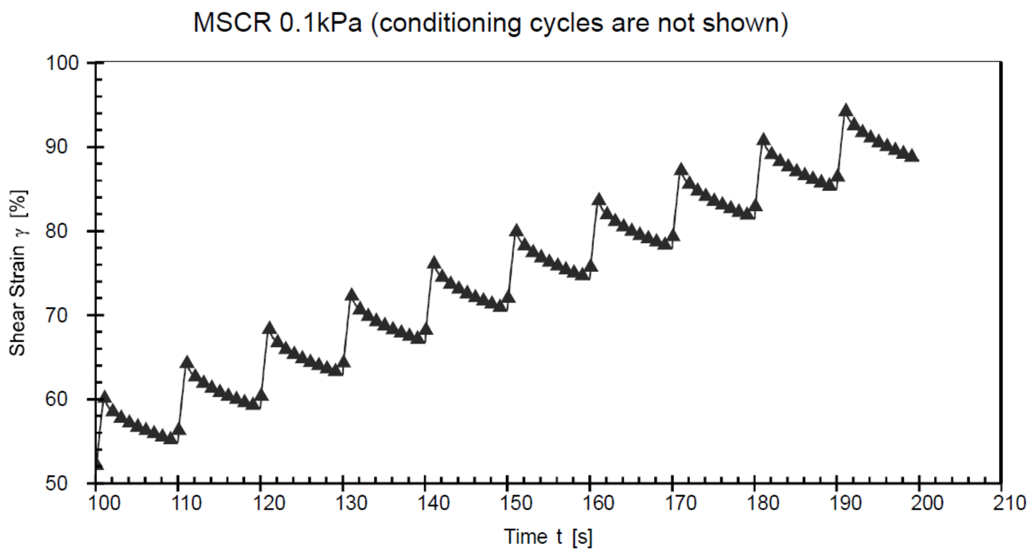


Figure 20. PmB1 at 0.1 kPa loading 60 ˚C.

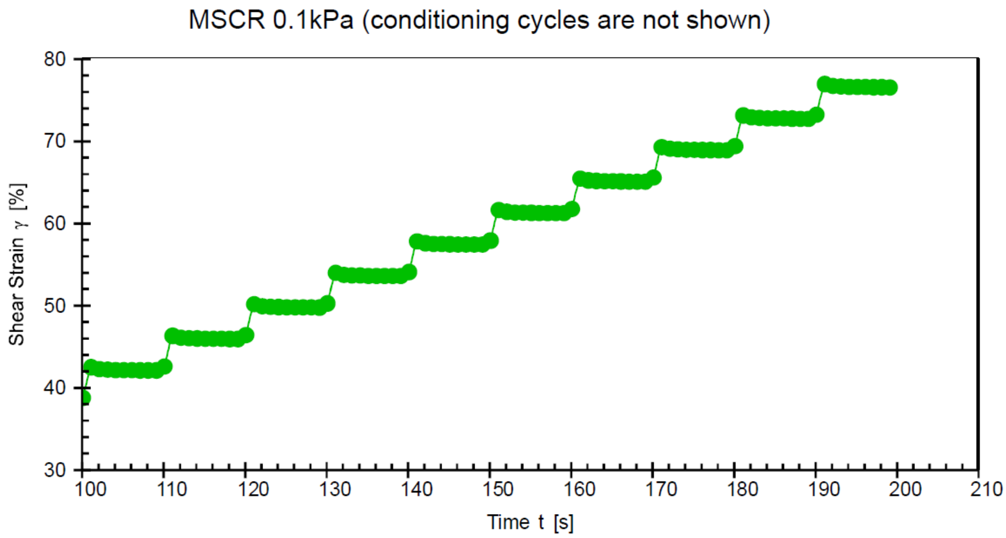


Figure 21. Bit1 at 0.1 kPa loading 52 ˚C.

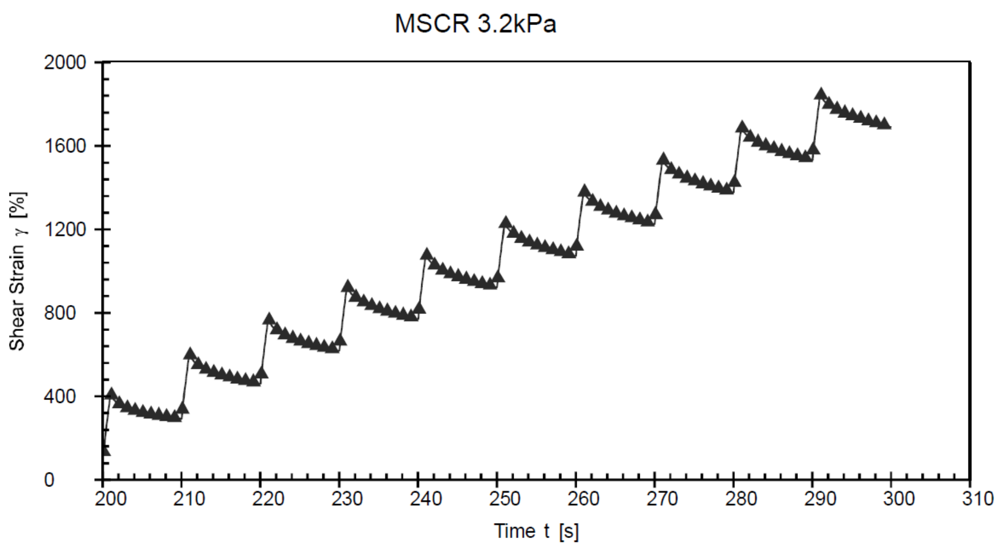


Figure 22. PmB1 at 3.2 kPa loading at 60 ˚C.

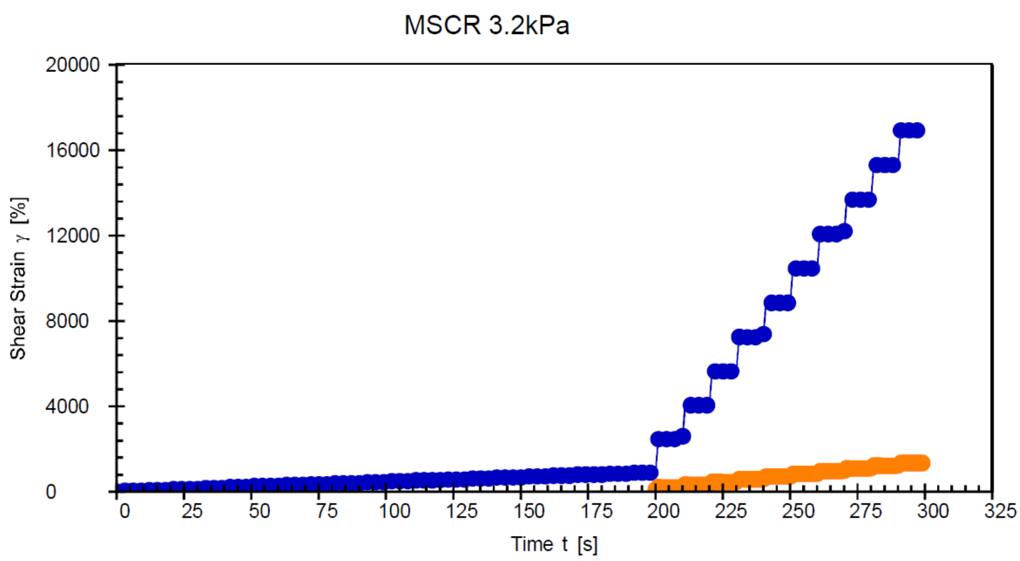


Figure 23. Bit 1 (blue line) and PmB1 at 3.2 kPa loading at 52 ˚C.

Using the data obtained from the measurement a percent recovery (R%) and non-recoverable compliance (Jnr) are calculated (D'Angelo et al. 2007; Aromaa 2016; Bukowski, Youtcheff & Harman 2011) for different levels of stress applied according to the equations provided in respectable standards EN 12697-25:2016 or AASHTO T 350.

There are different relations between R% and Jnr which are good for normal, heavy, very heavy or extreme traffics (Figure 24 and Table 6) (Blazejowski, Wojcik-Wisniewska & Baranowska 2018).

Table 6. MSCR based classification of binders in relation to the particular traffic conditions (after Blazejowski, Wojcik-Wisniewska and Baranowska 2018).

<table>
<tr><td rowspan="2">Type of traffic</td><td rowspan="2">ESAL range</td><td colspan="2">Requirement at XX temperature for PG-XX-YY</td></tr>
<tr><td>Requirement for Jnr3.2</td><td>Additional requirement for Jnr_diff</td></tr>
<tr><td>S – standard</td><td><10 mln and standard speed >70km/h</td><td>≤4.5</td><td rowspan="4"><75%</td></tr>
<tr><td>H – Heavy</td><td>10–30 mln or slow traffic 20–70 km/h</td><td>≤2,0</td></tr>
<tr><td>V – Very heavy</td><td>>30 mln or standing traffic <20 km/h</td><td>≤1,0</td></tr>
<tr><td>E – Extreme</td><td>>30 mln and standing traffic >20 km/h</td><td>≤0,5</td></tr>
</table>

The MSCR test was developed to evaluate the rutting propensity by the means of DSR on a much more reliable or fundamental property understanding level, than the original rutting parameter. The sample amount is much lower than in BBR and the test is quick, currently standardized in EU under EN-16659 Bitumen and Bituminous binders - Multiple Stress Creep and Recovery Test (MSCRT) and in US by AASHTO M322 and AASHTO T 350.

Bukowski et al. (Bukowski, Youtcheff and Harman 2011) report that “In 1996, Mississippi DOT built several test sections on I- 55. Multiple modifiers were used in the sections, including SBS, SB, SBR, and crumb rubber, with a control section of unmodified (neat) asphalt binder. Rutting was monitored for six years. The findings from this study again indicated that the MSCR parameter, Jnr, correlated much better to rutting then the PG parameter, G*/sin.”

It would be worth evaluating if this finding holds true for the Finnish conditions. However, given the lack of suggestion of the temperature that the test should be performed at in European standards, or the temperature of 60 °C as a default for all measurements, suggestion for Finland is to investigate what temperatures of measurement would be the most useful for local use and which values would correlate the best with good field performance, if the test was to be incorporated into specifications in the future.

The test is conducted by applying shear stress to the sample in cycles. At first the stress of 0.1 kPa is applied for 1 second and sample is allowed to relax for 9 seconds. A creep-like curve is obtained and a relaxation behavior is visible by observation of shear strain (deformation and its recovery). This type of loading is repeated 10 times initially as a sample preloading and another 10 times, which is used in further calculations. Afterwards a second loading sequence is applied, in which applied shear stress of 3.2 kPa for 1 second is followed by 9 s of relaxation and this pattern is also repeated 10 times.

What can be calculated from this data is so-called percent recovery (R%) and compliance (Jnr) in respect to applied loads, more information on how the calculations are conducted can be found in (Aromaa 2016). However, currently available programs supplied with the rheometers typically have a pre-installed template packages with preprogrammed algorithms to follow either EN or AASHTO standard. The results are analyzed automatically and a pass/fail criteria is provided for the material at tested temperature. In this context the test is easy in terms of uptake by the industry and informative in terms of resistance of the binder to permanent deformation damage.

The difference between AASHTO and EN standards is in the definition of the sample to be tested and the choice of temperatures. In AASHTO the test is performed at PG-XX (upper temperature of performance), while in EN the operator can freely choose between 40, 50 or 60 °C. It is up to local specifications to define the acceptance criteria for own climate. Additionally, the AASHTO standard defines that sample should be RTFOT aged, while the EN standard does not explicitly require that. For that reason, it is important to remember when defining specifications, that when a MSCR test is required, it should be noted that the RTFOT aged sample should be tested, as the RTFOT binder is the one supposedly similar to the binder in field after construction.

The binder used for the PG determination (RTFOT aged), used in the example in the section 7.1.6.1.2 was tested by means of MSCR test performed at 52, 58 and 64 °C. The results (Table 7 and Figure 24) indicate that the binder is meeting the requirements of the PG58-YY in terms of resistance to rutting for 10,0 AB roads in Finland. However, it may be too soft for the 25,0 SMA roads. If the binder was to be used in the climate region where the PG52-YY would be required, its rutting resistance would be sufficient for 25,0 SMA roads.

Of course, those values were determined in relationship to the mixtures prepared in USA, especially that Mississippi was the state in which the methodology was developed. For Finnish conditions, to understand whether this parameter also influences the rutting during the summertime, own correlations are suggested. However, the test is simple and quite fast (approximately 15–20 minutes per tested temperature). It is the choice of temperature of interest that is of importance when evaluating the results, and therefore the actual climate conditions in which the road will be operated play the role in test design.

Table 7. The results of the MSCR test of the 70/100 binder from 2018.

Temperature	R_01	R_3.2	R_diff	Jnr_0.1	Jnr_3.2	Jnr_diff	Result
Units	%	%	%	1/kPa	1/kPa	%	
52	9,01	3,35	62,79	0,7653	0,8266	8,01	Pass “V”
58	4,22	0,32	92,49	1,9586	2,1362	9,07	Pass “S”
64	0,93	0	100	4,6220	5,0733	9,76	Fail

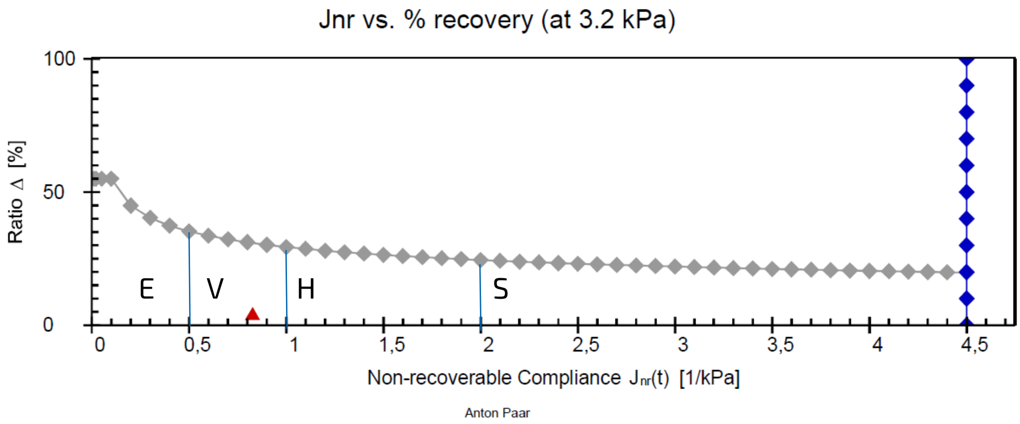


Figure 24. The qualification regions for the pass/fail results for both compliance and percent recovery values. The red triangle represents the results of the 70/100 at 52 °C falling under the grey curve, resulting in the PASS. Measurement at 64 °C is not passing as the J_{nrdiff} is 100%. The J_{nr_diff} criteria for traffic classes S, H, V and E are marked on the graph.

7.1.6.2 Typical ways of utilizing the DSR data for non-PG-related analysis

7.1.6.2.1 Master Curve

To create the master curve a time temperature superposition rule is utilized. The isothermally obtained frequency sweeps are shifted by a vector known as shift factor, to create a smooth curve. This vector is different for data obtained at different temperatures. The values of the shift factors may also change when a different reference temperature is used.

With aging the curve typically moves upwards for the complex modulus and downwards for phase angle as presented in figures below.

Unfortunately, the shift factor and the actual master curve depend on the choice of the reference temperature. The shape of the curve also may depend on this parameter. Additionally, for polymer modifier bitumen and waxy bitumen types, the frequency sweep may not be creating a smooth continuous line, therefore the fitting of the master curve becomes challenging. Such materials are referred to as rheologically not simple. Of course, such deviation from expected curve shape can be an indication of the presence of the modification, which could be considered as an information. However, it is up for a discussion how accurate the created curves are in the case of modified binders. In terms of uptake into the use in the quality control laboratory – the aspect of repeatability being influences by the operator preparing the master curve is on some level marking it as risky technique. If the method is considered, the use of software that eliminate the human factor in the analysis is suggested.

Airey suggests using rather black curves for material type differentiation and processing the measured (as opposed to extrapolated with use of master curve) data for analysis rather than the master curve subjective to the errors in rheologically complex liquids (Airey 2002).

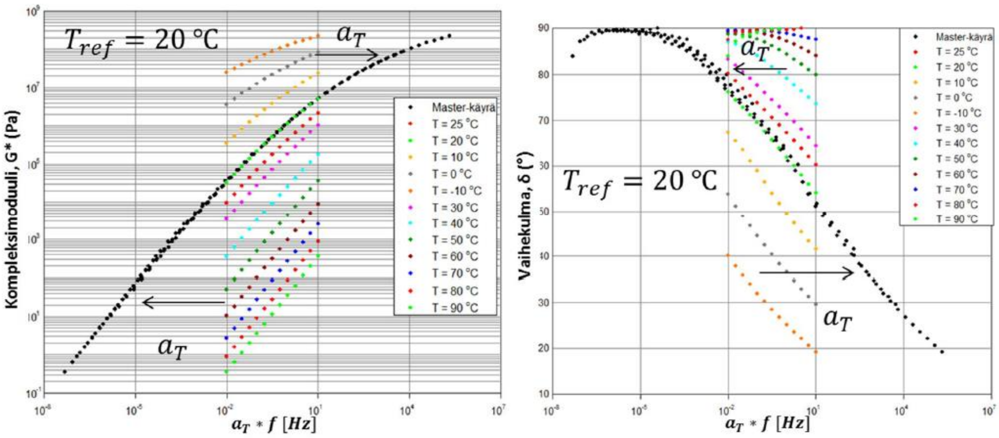


Figure 25. The example of the Master Curve for Complex Shear Modulus (left frame) and Phase Angle (right frame), in which the colorful lines were measured at respective temperatures and black lines represent the prepared master curve (Aromaa 2016).

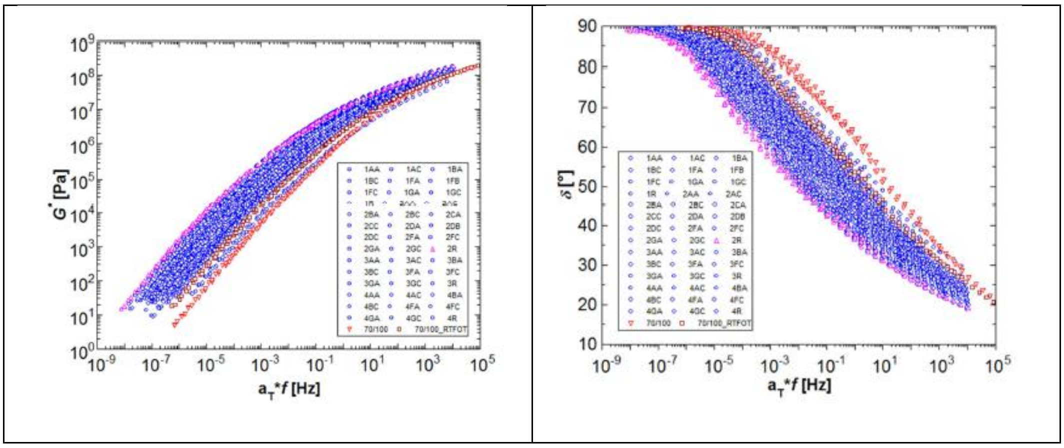


Figure 26. Master curve examples for complex modulus and phase angle, determined for fresh bitumen 70/100 and the binders extracted from aged sections on the Highway number 1 in Finland (Aromaa 2016).

7.1.6.2.2 Black curve

The polymer modified bitumen is a material, which does not follow the Time-Temperature Superposition Principle. Because of that, the master curves should not be pursued. However, Airey (Airey 2002) proposed the use of black curves for evaluation of the data in its pure unaltered form. The black diagram, that is a graph in which measured Complex Modulus (|G*|) is plotted against measured Phase Angle (δ), can be used to

- identify the issues with Compliance during measurement;
- to determine the correct geometry for measurements conducted,
- to identify the presence of polymer modification and the response of the network,
- study the effect of aging (oxidation) of both polymer and bitumen on the rheology
- evaluate if measurement occurred within the LVE region.

Since then, the use of black diagram was also demonstrated (Figure 27) in the studies of filler reinforced mastics (Micaelo et al. 2017), allowing us to explore the difference between stiffening due to network development and due to physical reinforcement of the structure by particles.

Softening point measured from bituminous material blend or mastic increases in comparison to the base bitumen with addition of either polymer and/or filler. However, the addition of particles that do not create a homogenous structure with the bitumen affects the response in the DSR in a different way. Studying the filler reinforced bitumen (inhomogenous dispersion) allows us to see the behaviour of the bitumen as the dominant – the black curve is close to that of the black curve of the reference bitumen (Micaelo et al. 2017). However, for comparison, the addition of the polymer as a modifier, when homogenously dispersed in the structure, causes formation of the network. This network is responsible for the increased elastic response of the system (compare black curves of reference bitumens from Figure 27 a and b, versus all the curves in Figure 27 a). Nevertheless, if the polymer is present as a modifier which did not form a network and remained in form of granules or particles suspended in the liquid, it could be possible that the addition would not be visible in the DSR measurement.

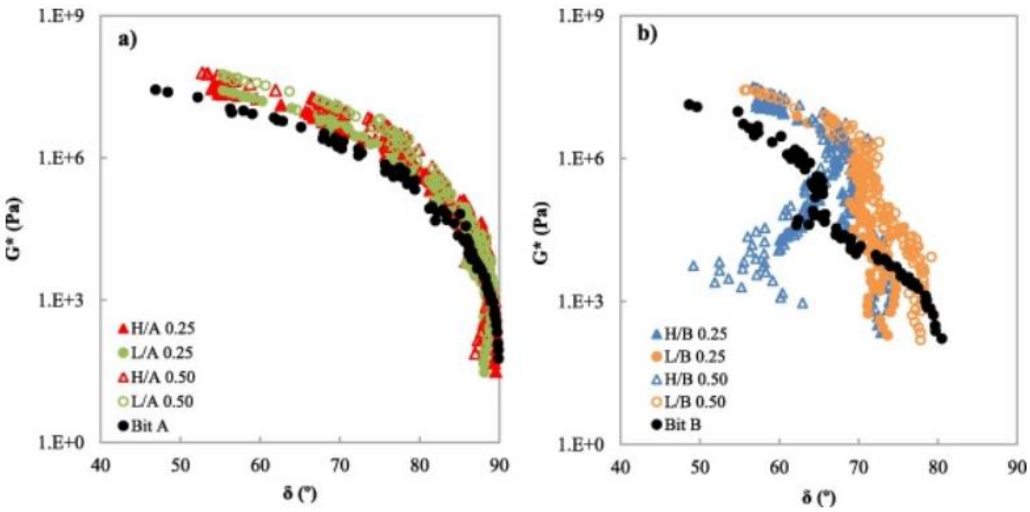


Figure 27. Reprinted from Micaelo et al. 2017 a) the black diagram of non-modified bitumen (black) with two fillers at different ratios (colorful), b) the black diagram of SBS modified bitumen (black) and the mastics of two fillers mixed at various ratios (colorful).

For High Density Polyethylene (HDPE) polymer (Perez-Lepe, Martinez-Boza & Gallegos 2005), the relation between microscopic observations and rheological response are similar to those presented in Figure 27. When the particles of polymer do not create the network, but the structure reminisces the filler particles suspended in binder, the black curve is consistent with the black curve of non-modified bitumen (Figure 28). However, with increasing the HDPE content the dispersion is visible, network is created (despite still not being homogenous), and an observed deviation from the reference black curve is observed. The shift of the onset of the elastic behavior (low Phase angle (δ) at respective temperatures) is observed with increasing content of the polymer.

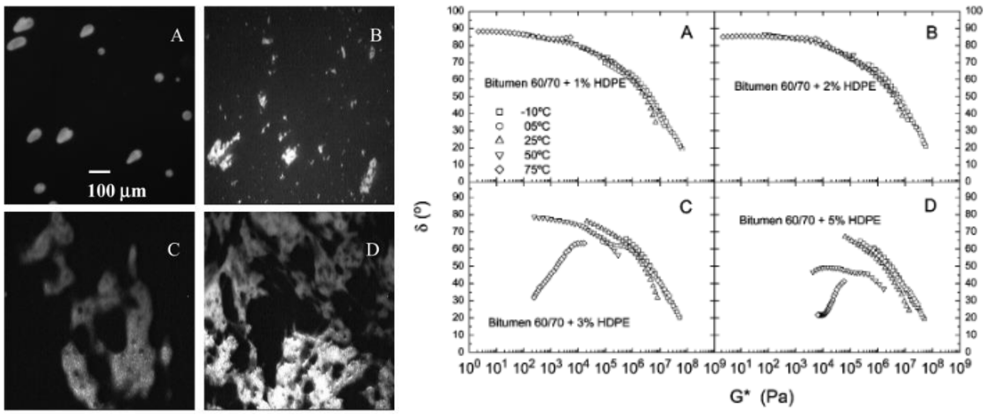


Figure 28. Reprinted from (Perez-Lepe;Martinez-Boza ja Gallegos 2005), on the left the microscopic observations of 60/70 grade bitumen with 1% (A), 2% (B), 3% (C) and 5% (D) of HDPE, while on the left the black diagrams for the corresponding blends.

The relaxation time increases with increasing size of the molecules, which means that to measure the change in the viscoelastic properties inflicted by the large particles/molecules a very low frequency is necessary. Therefore, even if the elasticity in the system is coming from the filler it will not be visible unless extremely low frequencies are used.

The influence of the polymer is visible in the applied range of frequencies in the oscillatory rheological measurements (DSR), but the response has been previously postulated rather to originate in the dual nature of the system composed of liquid and solid (Airey 2002). In the situation when the base bitumen moves towards the Newtonian behavior (at certain temperature) the elasticity of the system measured with the equipment is supposedly coming from the network of polymer. However, if the polymer molecules coalesce and form droplet like particles that are visible under microscope, the material should deviate away from the black curve of reference bitumen only slightly like presented in Figure 27 a, because the polymer acts more as a filler than a network provider. In such case the rheology is not sufficient alone to determine if the sample contains modification and a supportive technique is required to confirm the polymer presence, e.g. infrared spectroscopy (Yut & Zofka 2011).

When a molecule is dispersed (and polymers are macromolecules) the elasticity is visible at frequencies typically measured using the basic DSR system. Which frequencies are the correct to follow behaviour of polymer in terms of relation to their molecular weight, needs to be determined. Unfortunately, there may be deviation from Linear Viscoelastic properties in the material, which could lead to incorrect numerical results, and should be considered during the analysis.

The polymer modified bitumen or unmodified bitumen is exposed to different frequencies of loading in service. Typical traffic loading has been calculated to correspond to 10 rad/s (1,59 Hz), while thermal loading to 0,005 rad/s (0,000795 Hz) (Rowe 2011). As discussed in the later part of this report (Part 3), related to resistance to studded tyre traffic, it was postulated that behaviour at high frequencies or low temperatures may be linked to the rutting by studded tyres – by inflicting the elastic behaviour in the material, which makes high frequency responses of the material an interesting property to study in the future.

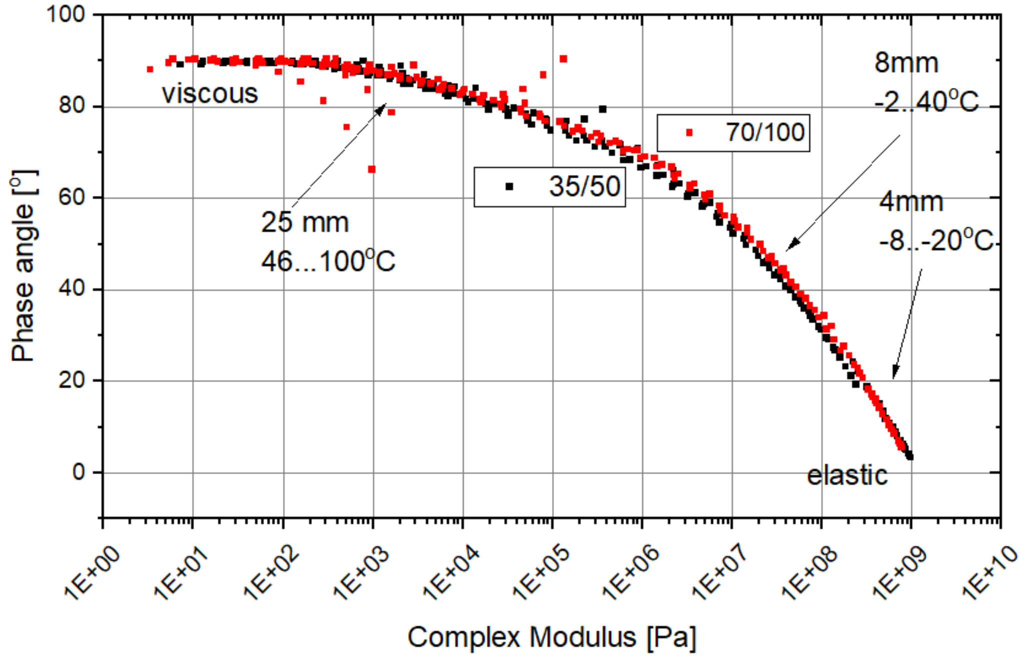


Figure 29. Full black curve for RILEM bitumen 35/50 and 70/100 measured from -20 to 100 °C demonstrating problems with differentiating between binders based on their black curve (own data obtained within the RILEM Round Robin (Porot, Vanstenkiste et al. 2021)).

7.1.6.2.3 Cole-Cole plot

The Cole-Cole plot allows for evaluation of the relaxation properties of the material. The decreased loss modulus at corresponding storage modulus values indicates hardening and brittleness. Because the plot is in the linear scale, the low temperature and high frequency behavior is underlined in those graphs (Aromaa 2016; Saliani et al. 2019).

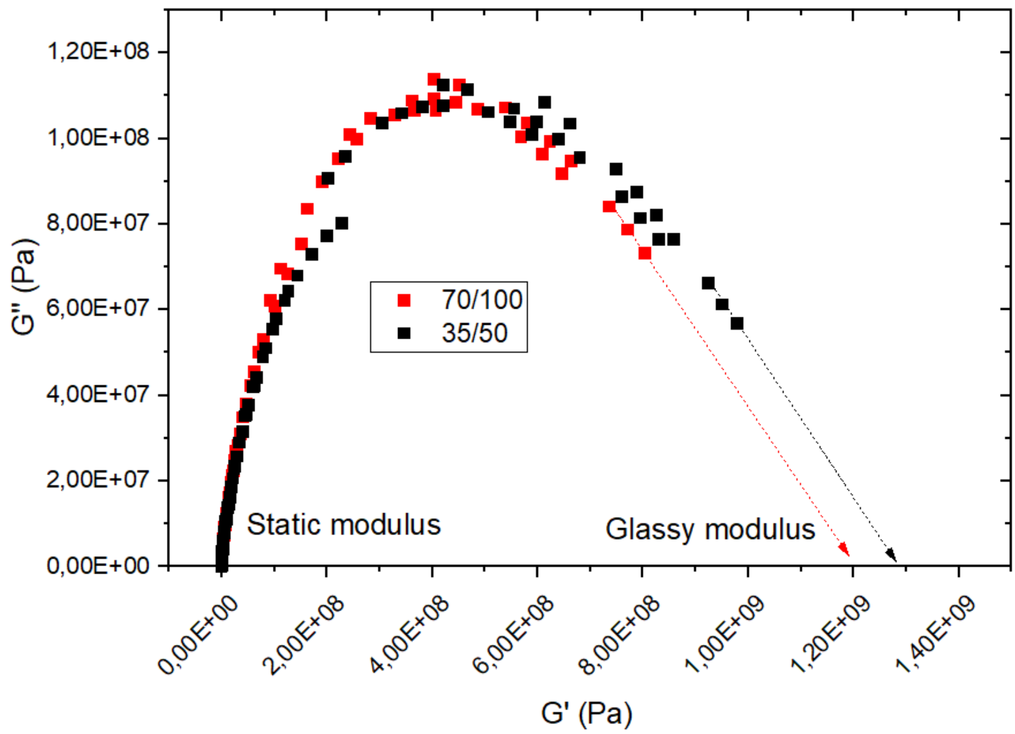


Figure 30. The example of the Cole-Cole plot (own data obtained within the RILEM Round Robin (Porot, Vanstenkiste et al. 2021)).

7.1.6.2.4 Zero Shear Viscosity

Zero Shear Viscosity (η0), which is simply said a viscosity of the material measured when the shear in the sample is approaching zero, stays in relationship with the molecular mass of the material tested. This characteristic can be tested in multiple ways (Mezger 2002):

- In a rotational test when the shear rate is approaching zero at certain temperature
- In an oscillatory test (e.g. frequency sweep) when angular frequency ω is approaching zero
- In a creep test, at the end of the load phase when the tension is released and steady-state flow is achieved.

However, by definition – the viscosity can be measured when the material is in the fluid state, not solid. For the bitumen and polymer, this means that the phase angle (δ) measured at certain temperature needs to be equal to 90° for the Zero Shear Viscosity to be equal to Complex Viscosity. In case of bitumen this is in line with temperatures above the softening point (Makowska;Huuskonen-Snicker, ym. 2018). For the polymer this typically is measurable above the melting temperature. However, in the polymer case, above the melting point (180 °C) a significant oxidation starts to occur and polymer changes color visibly from transparent to brown. Typically, for SBS the ZSV would be determined at 220 °C, but when in the oven the oxidation in the material is occurring at a fast rate. Assumption is that the structure changes due to oxidation and determining the properties from such material is obsolete. It has been noted that for polymers the viscosity in diluted state is provided in the product catalogues. This information is important for application of SBS and SEBS as an adhesive (Sandlund 2004), which typically is applied in thin layers through spraying from a solution. Nevertheless, at a certain concentration in the solution the viscosity of the system (solvent and polymer) should still provide the information about the molecular size of the polymer. The derived relationship between mean molecular mass [M] and viscosity [η] for dilute polymer solutions states that η is proportional to M, with the proportionality constant dependent on the type of the polymer

$$[\eta] \ \alpha \, M^{\wedge}(0.5 \text{ to } 0.8) \quad (3)$$

Comparing the viscosity of the polymer solutions (when difference in SBS is in its molecular mass) provides an information about the degree of difference between various modifiers and should on some level differentiate between products containing differing types of polymers. However, this equation works only for dilute solutions in which molecules are fully dispersed and that may not be a true assumption for PmB systems.

Additionally, the ZSV for PmB and bitumens can be determined from the frequency sweeps performed, by using the Cox-Merz rule in which viscosity in fluid state is equal to complex viscosity (η^*) which relates to complex modulus $|G^*|$ (Mezger 2002; Cox & Merz 1958) as given in following equation:

$$\eta^* = |G^{\wedge *}| / \omega \quad (4)$$

Therefore, testing the complex viscosity (complex modulus at very low frequencies and temperatures where phase angle is close to 90°) should be therefore indicative of differences in the polymer types and concentrations used, if PmB come from one refinery producing different blends.

7.1.6.2.5 Glover-Rowe parameter

Glower-Rowe parameter has been described as part of the work for the Finnish Transport Agency previously and the more detailed review related to it is available in (Aromaa 2016; M. Makowska 2017; Pellinen & Makowska 2018; Makowska, Aromaa & Pellinen 2017; Glover et al. 2005; Rowe 2011).

In principle, the Glover-Rowe parameter (G-R) links the DSR results obtained at very low frequencies (0.005rad/s) (or read from the master curve at the this frequency) to the ductility measurements performed on pavements expressing extreme fatigue cracking. By assigning the threshold value to the G-R for well and bad performing pavements a possibility to evaluate propensity to cracking is presented (Glover et al. 2005; Rowe 2011). It was underlined that the value is derived on the basis of failed pavement and that perhaps deriving the threshold value in the initial or RTFOT bitumen that would correlate with good performance would be of interest (Makowska, Aromaa & Pellinen 2017).

7.1.7 Bending Beam rheomether (BBR) derived parameters

The test and equipment necessary to conduct the measurements using the Bending Beam rheomether is standardized in European Union (SFS-EN 14771:en:2005) under the title of *Determination of the flexural Creep Stiffness. Bending Beam Rheomether (BBR).* The test is designed to characterize the stress relaxation properties of an aged asphalt binder.

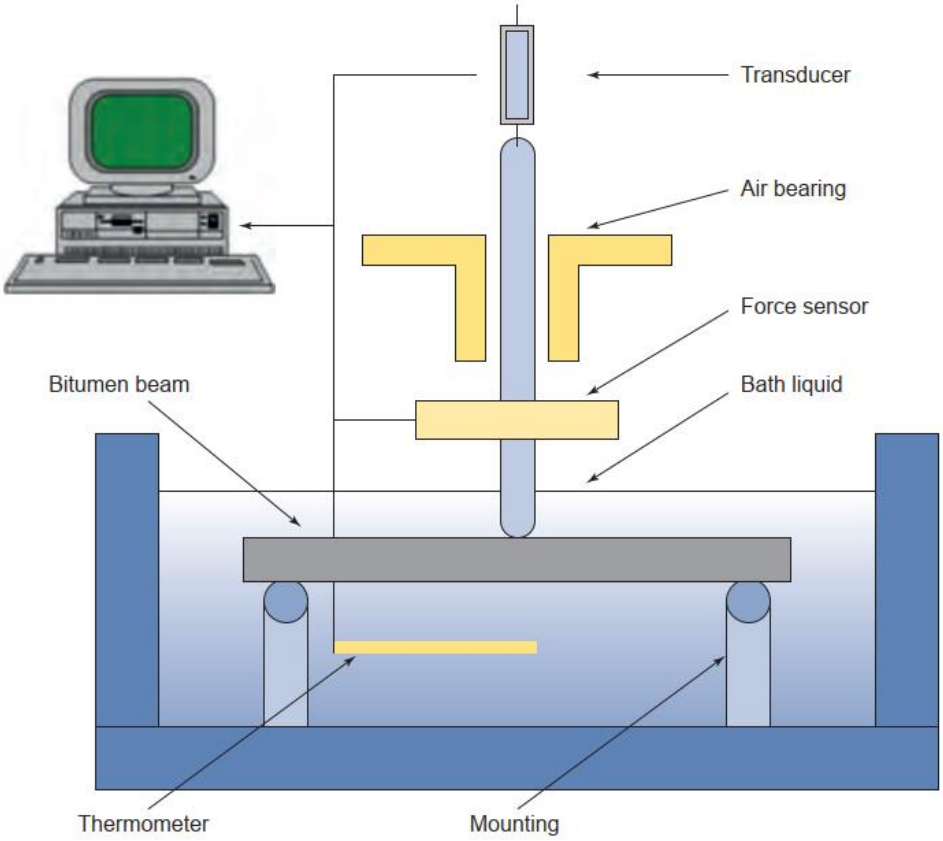


Figure 31. The schematic representation of the test set-up of the Bending Beam Rheomether test (after Hunter, Self & Read 2015).

The binder is poured into a mould to create a beam. The beam is submerged in the liquid non-blending with the bitumen to control the temperature during the measurements. The load of 1 N is then applied from the top in the middle of the beam. The stiffness (S) as a function of time can be calculated from the relationship to load (*P*), deformation (δ_{def}) as a function of time for a simply supported rectangular beam with width *b*, depth *d* and *l*, from the equation:

$$S(t) = \frac{Pl^3}{4bd^3\delta_{def}(t)}$$

The universal testing machine can test the deflection in the time domain, not in the frequency domain. For this reason, the stiffness curve is plotted against the time. The calculation of the stiffness is conducted at 5, 15, 30, 60, 120 and 240 seconds. A master curve is prepared based on those results, by fitting a second-degree polynomial function to the logarithm of the measured stiffness values versus the logarithm of the loading times using formula:

$$log\, S_C(t) = A + B * log(t) + C * log[log\,(t)^2] \quad (5)$$

Where A, B and C are regression coefficients.

The m-value is calculated using the same loading times using the below formula

$$m(t) = \frac{[dlog[S(t)]]}{dlog(t)} = |B + 2 * Clog(t)|, \quad (6)$$

Where B and C are coefficients determined from the previous equation (5).

The value of S at 60 s and the slope of the curve (m-value) at 60 s loading time are the parameters used for the evaluation. The measurement is conducted at multiple low temperatures. For this reason, the number of samples required and the time of the test are quite large. Considering the current lack of specifications related to low temperature in Finland it could be challenging to convince the industry to uptake the technique even if the results have been linked to the performance of the binders (Asphalt Institute Technical Advisory Committee 2019).

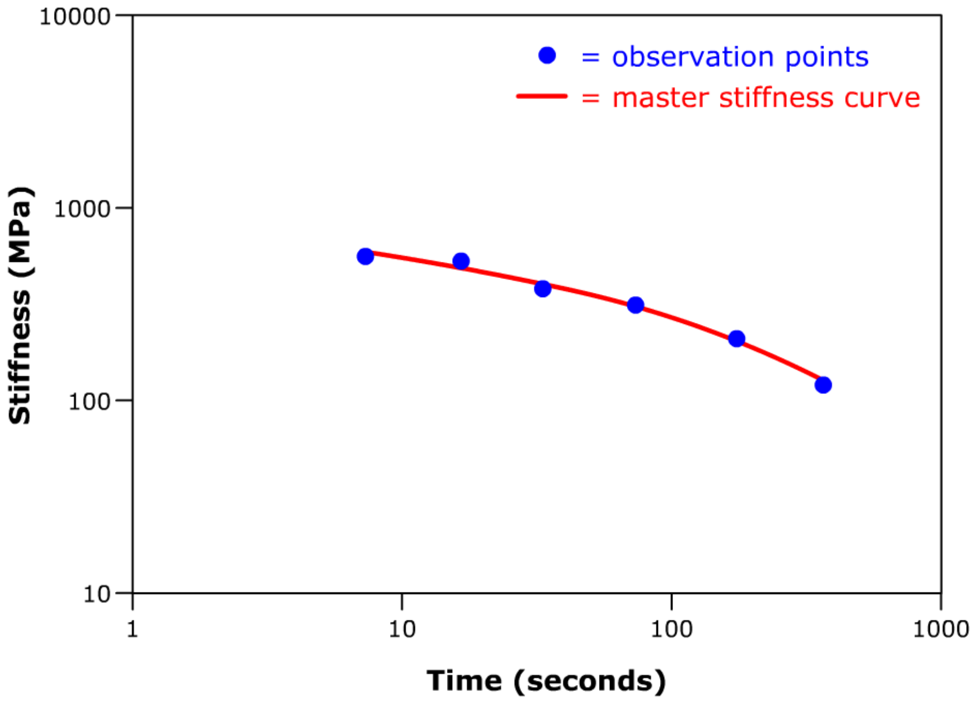


Figure 32. Master curve of the stiffness plotted from the BBR measurements. (www.pavementinteractive.org).

For the binder to fulfill the low temperature performance criteria in the PG grading, the binder after RTFOT and PAV aging needs to have the stiffness at 60 s below 300 MPa and the m-value above 0.300.

7.1.7.1 Creep Compliance

To describe the typical behavior of viscoelastic materials, defined as time-dependent strain $\varepsilon(t)$ divided by constant stress σ_0, the below function can be used

$$D(t) = \frac{\epsilon(t)}{\sigma_0} = D_0 + D_1 t^m \quad (7)$$

Where D_0, D_1, and m – are the power function parameters. (Kim, Sholar and Kim February 2008)

7.1.8 Direct Tension Tester (DTT)

The Direct Tension Tester (DTT) measures the strain and stress in the sample at low temperatures. The samples with high level of modification may express quite high stiffness, but do not crack due to the presence of polymer. The DTT applies tensile load to a dog-bone shaped samples, and load is applied until failure. In combination with BBR data a critical cracking temperature of a pavement is determined (Pavement Interactive Accessed last on 18.5.2021). This methodology is described closer in *SFS-EN13589:2018 Determination of tensile properties of modified bitumen by the force ductility method.*

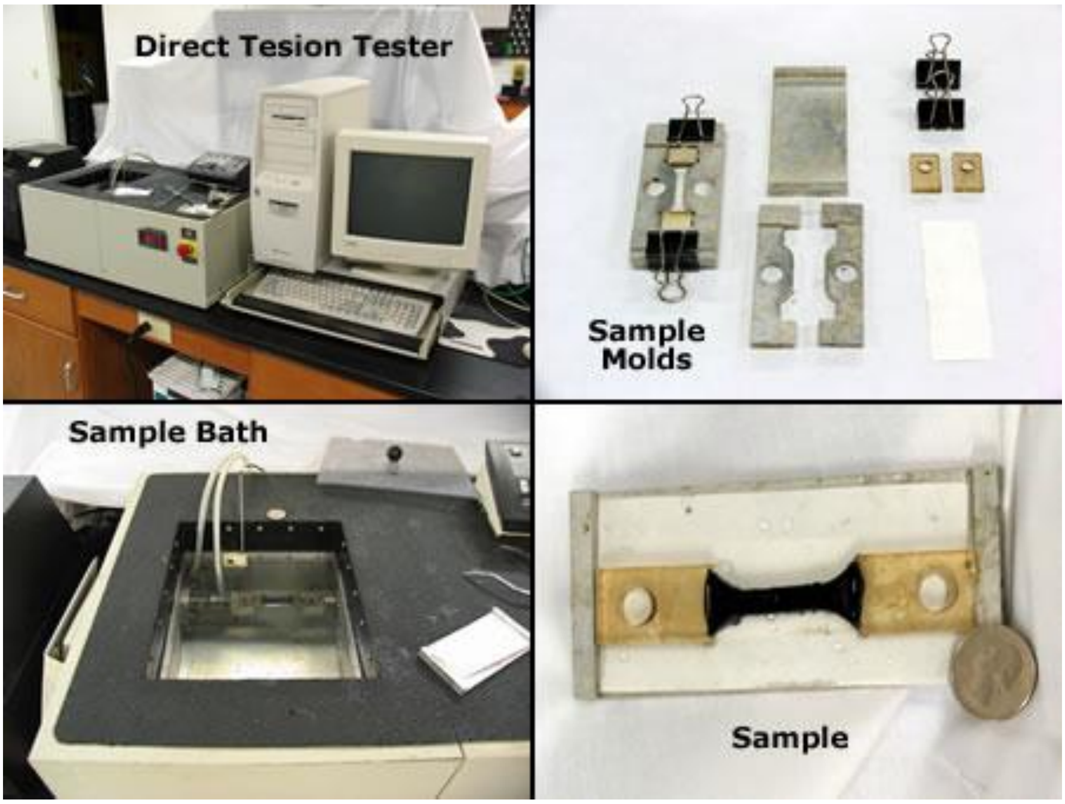


Figure 33. The DTT equipment and sample example (Pavement Interactive Accessed last on 18.5.2021).

Parameters measured in the test comprise failure strain and failure stress. The rate of elongation and peak load are reported as well.

7.1.9 Low temperature cracking resistance (BBR+DTT)

Below procedure combines BBR and DTT test results to determine the low temperature asphalt binder grade by the following steps:

- Calculate the stiffness master curve from BBR test data.
- Create the creep compliance curve by taking the inverse of the stiffness master curve.
- Convert the creep compliance curve to a relaxation modulus. This is a fairly involved and numerically complex conversion.
- Calculate the thermal stress. This is also a fairly involved and numerically complex conversion.
- Multiply the calculated thermal stress by a “pavement constant” (chosen through research as 18). This pavement constant converts the laboratory-determined thermal stress to an equivalent field cracking temperature.

Compare this calculated thermal stress to the failure stress from the DTT.

When initially grading an asphalt binder, two DTT test temperatures are used and a curve is drawn between these two temperatures (with just two points the curve is really just a line). The point at which this DTT curve intersects the BBR thermally induced stress curve (Figure 8) is defined as the *critical cracking temperature* of a pavement that uses the tested asphalt binder.

When prequalifying an asphalt binder for a specific PG grade, only one DDT test temperature is needed. If the DTT determined failure stress is above the BBR thermally induced stress curve then the binder passes the low temperature prequalification.

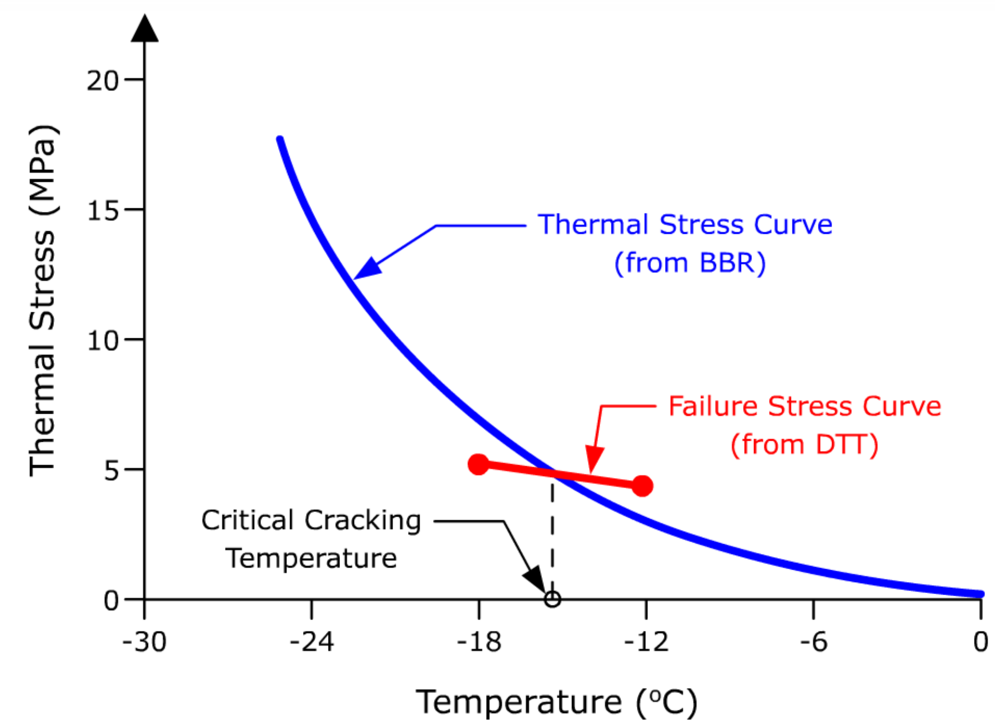


Figure 34. Determination of the critical cracking temperature of the binder using DTT and BBR results presented on a schematic graph (Pavement Interactive Accessed last on 18.5.2021).

7.1.10 4MM DSR instead of BBR?

Conducting the testing on the binders using BBR and DTT require quite a large amount of material. In the times when the recycled asphalt pavements are encouraged for the use, the assurance of the binder properties meeting the requirements set for the binder to be used in the pavement in the specific location, one would need to extract a significant amount of bitumen to perform the BBR.

At the same time, the technology related to the DSR equipment progressed, and it became plausible to run the measurements at low temperatures using the 4 mm geometry. The problems with equipment compliance at low temperature were also overcome (Sui et al. 2010; Läuger & Stettin 2016; 2010), by introduction of the compliance correction factors (AASHTO T315-10).

The sample in DSR can be measured multiple times at multiple temperatures. A relatively small amount of sample is required to perform the test, in comparison to BBR. For both of those reasons a methodology using the DSR with 4 mm geometry at low temperatures was proposed to replace the BBR (Sui et al. 2010; Hajj et al. 2019).

The error of the stiffness measurement of the stiffness and m-value from the BBR measurement is allowed on 7.2% for Stiffness and 2.9% for m-value according to AASHTO procedure description. Hajj et al. (2019) found that the error in those values obtained by the means of 4 mm geometry is on a similar level. It is worth to mention that according to the EN instruction for the BBR, the repeatability of the method is within 9% for stiffness and 4% for m-value, while reproducibility is on the level of 27% for stiffness and 13% for the m-value. **The discrepancy comes from the master curve building procedure chosen by the operator.**

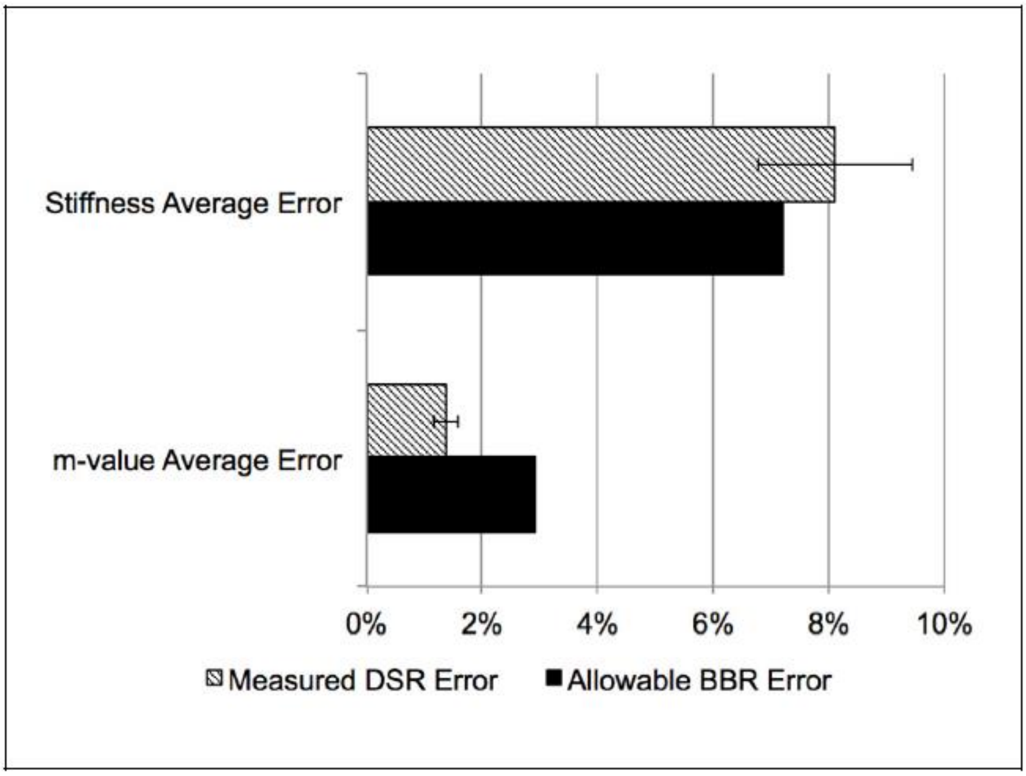


Figure 35. Single operator error using the 4 mm DSR method compared to the allowed error levels of the BBR procedure (after Hajj et al. 2019).

Multiple methods of recalculating the complex modulus and phase angle to the stiffness and m-value have been proposed in the literature. Hereby, a method considered the simplest for the direct uptake is discussed.

Anderson et al. (1994), under the assumption of phase angles close to the 0 at low temperatures Proposed an equation by using which calculation of the S(t) is possible using the complex modulus and changing the frequency domain into time domain from equation:

$$S(t) = \frac{3|G^{*}(\omega)|}{1+0.2\sin(\delta)} \quad (8)$$

where

ω – radial frequency, [rad/s]
$|G^*|$ – shear complex modulus, [Pa]
δ – phase angle [°].

The stiffness in this case would be computed for various loading times and m-value determined in a similar manner as from the stiffness vs. loading curve in the BBR test.

On the other hand, Oshone (2018) provided the more linear relationship for the stiffness and m-value:

$$S(t) = 1.28|G^*(\omega)| + 19.2 \tag{9}$$
$$m = 0.008\delta + 0.1 \tag{10}$$

As is apparent from the equation (10), the phase angle corresponding to m-value of 0.3 is estimated at 25°. For this reason, perhaps the assumption of the Anderson model is not valid. Preferred is the use of correlation derived from actual measured values of Stiffness and m-values to complex modulus and phase angle. However, one is advised, that each of the correlations is developed on the certain set of binders, which may be incomparable to the binders considered in own study, for example if the correlation is derived on relatively stiff binders 20/30–50/70 and one would like to apply it to binders softer than 70/100–160/220, especially 650/900, an uncertainty towards absolute result of calculation should be exercised. The calculated value of Stiffness at 60 s loading and m-value, from DSR data is in this sense only indicative of the properties. It is advised to determine the correlations for the binders of interest in order to achieve more certainty.

Below are examples of the transfer of complex modulus values from 4 mm DSR scan after compliance correction into the Stiffness according to Oshone model and m-value according to Oshone model (Hajj et al. 2019; Oshone 2018) (Figure 36, Figure 37) performed on Aalto's laboratory data from samples evaluated within the MOREBIT project.

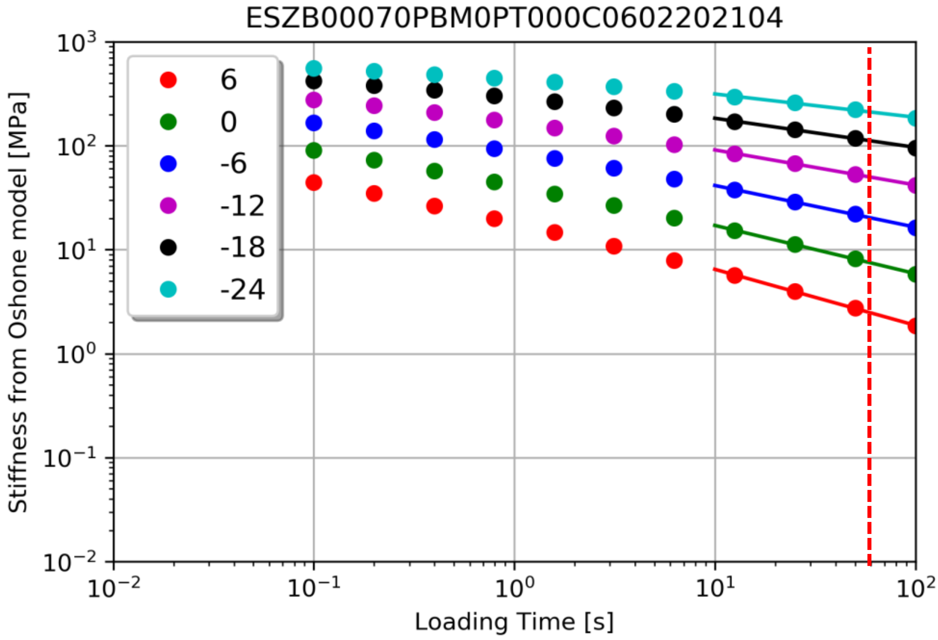


Figure 36. Stiffness of sample bitumen 70/100, calculated according to Oshone model from compliance corrected Complex Modulus values measured using 4 mm DSR between 0.01–10 Hz demonstrating interpolation principle, with red dashed line representing the 60 s loading time mark (data courtesy of MOREBIT project).

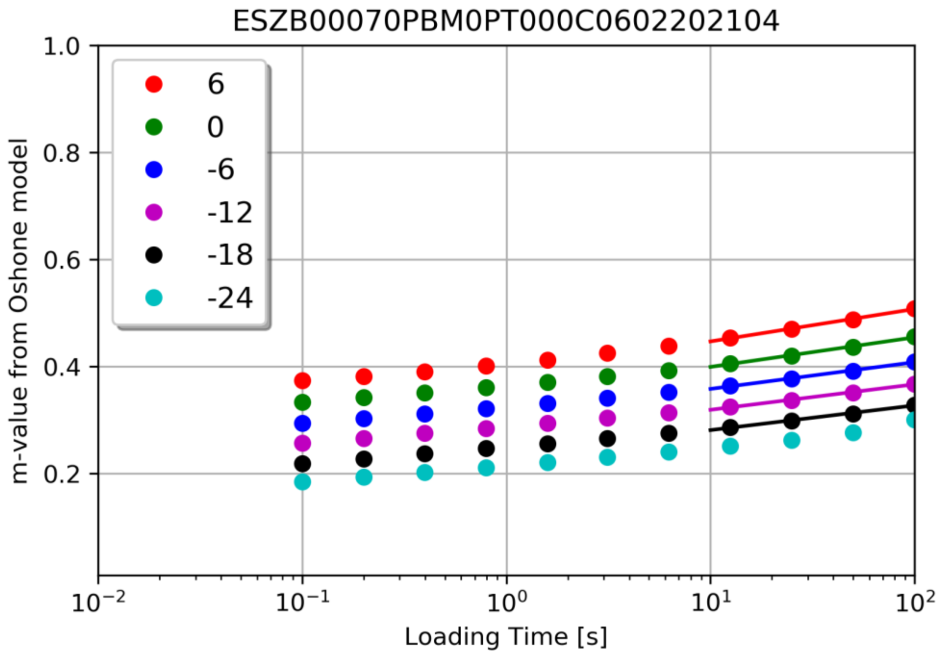


Figure 37. The m-values of sample bitumen 70/100, calculated according to the Oshone from compliance corrected phase angles model values measured using 4 mm DSR between 0.01–10 Hz demonstrating interpolation principle, with red dashed line representing the 60 s loading time mark (data courtesy of MOREBIT project).

7.1.11 DELTA TC

7.1.11.1 What is delta T_c (ΔT_c)?

For the determination of the low temperature performance in PG system, the critical temperature from BBR needs to be below the temperature corresponding to the temperature YY. However, even if below the YY temperature, the exact critical temperature from Stiffness criteria and m-value criteria can have different values (Asphalt Institute Technical Advisory Committee 2019).

The so-called delta T_c (ΔT_c) refers to the difference between the critical temperature determined by Stiffness ($T_{c,S}$) and critical temperature determined by m-value ($T_{c,m}$) from the BBR measurements.

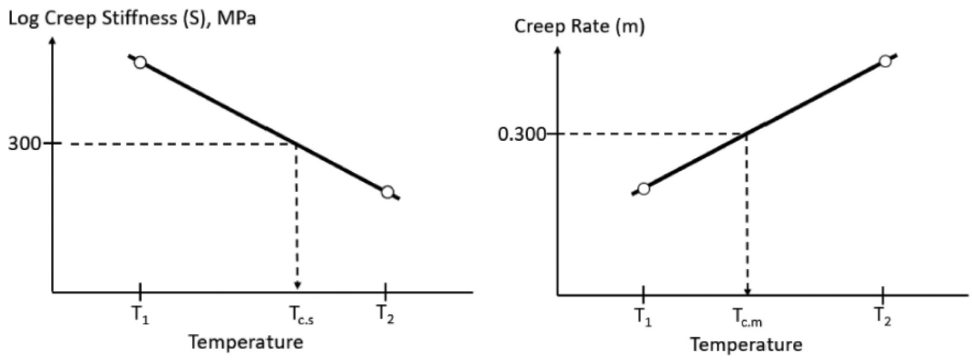


Figure 38. The graphical explanation of the critical temperature, printed after Asphalt Institute Technical Advisory Committee (2019).

The temperature can be determined graphically or through calculation based on linear interpolation principle described in below equations:

$$T_{c,S} = T_1 + \left(\frac{(T_1 - T_2) * (log300 - logS_1)}{logS_1 - logS_2}\right) - 10 \quad (11)$$

$$T_{c,m} = T_1 + \left(\frac{(T_1 - T_2) * (0.300 - m_1)}{m_1 - m_2}\right) \quad (12)$$

where

S_1 – creep stiffness at T1, MPa
S_2 – creep stiffness at T2, MPa
m_1 – creep rate at T1
m_2 – creep rate at T2
T1 – temperature at which S and m passes °C, and
T2 – temperature at which S and m fails, °C.

The ΔT_c is then defined as:

$$\Delta T_c = T_{c,S} - T_{c,m} \quad (13)$$

7.1.11.2 What is the meaning of ΔT_c?

If the sign of the result of the ΔT_c is positive, this means that the binder grade is governed by its creep stiffness (+ΔT_c). When the sign is negative, this indicates that the grade is governed by the creep rate m (-ΔT_c) (Asphalt Institute Technical Advisory Committee 2019).

During the BBR test, as the temperature drops, and the stresses inflicted by thermal shrinkage emerge, this results in the increase of stiffness (Figure 39). The volume decrease caused by molecular assembly, increases density of the material and this leads to increase of the stiffness, but that may lead to the thermal cracking due to shrinkage over large distances. However, if the binder is sufficiently viscous at those low temperature, the molecular movement within the material leads to the relaxation of stresses and decrease of the stiffness over time. This is described as the slope of creep, as the material starts changing shape under the given load.

When the stiffness is measured by BBR the desire is for the material at design temperature not to exceed the 300 MPa, as this value indicates brittleness and stress buildup in the material. When the m-value is determined, the desire is to establish the creep rate equal to 0.3 to assure good relaxation properties during the thermal loading, but also to prevent rutting. Too high of the m-value would indicate material very viscous, while too low m-value indicates no relaxation in the material. In the second case the m-value would be optimal at higher temperature, and that would result in negative ΔT_c.

Therefore, the more negative values of the ΔT_c are associated with binders more prone to thermal cracking and block cracking.

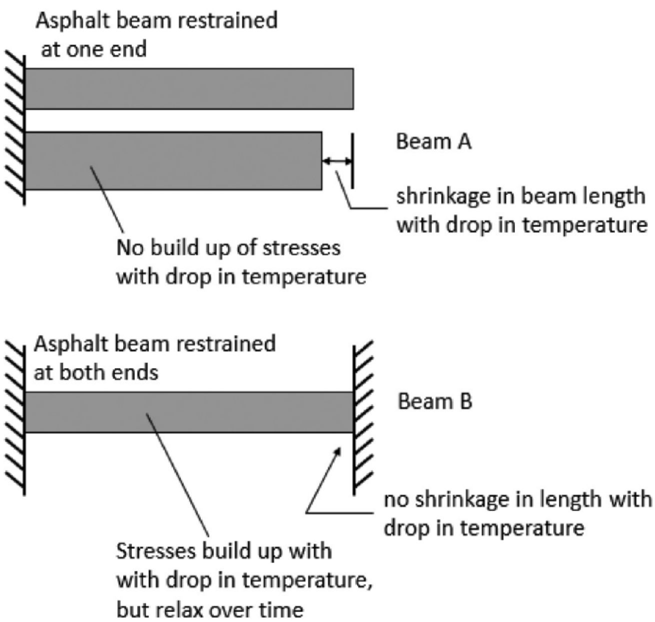


Figure 39. Graphical explanation of the stress buildup during the low temperature exposure of binder and its effect on the thermal cracking (Asphalt Institute Technical Advisory Committee 2019).

7.1.11.3 Example using above tested binder

The laboratory of Aalto University participated in MOREBIT project to which various bitumen samples were delivered by producers and contractors. The below example is provided using two different bitumen samples of penetration grade

70/100 and two different bitumen samples of penetration grade 160/220. **It is important to note that the calculations are performed on the unaged samples at this point, as the study is preliminary to evaluate the results for materials considered to study as a follow up of the BITU2020 project.**

Afterwards the data was mathematically processed using the Oshone model (equation 9 and 10) to create the stiffness graphs in the time domain, using previously presented equations. Hereby still the limiting criteria of 0.3 for m-value was used to demonstrate how the measurements correspond with each other. It was noted that for better correlation with the actual m-value measured by BBR, the value of 0.28 should be used for m-values derived by 4mm-DSR (Hajj et al. 2019), but at this point we use the 0.3 due to the actual lack of knowledge of how our climate products correlate to the BBR results.

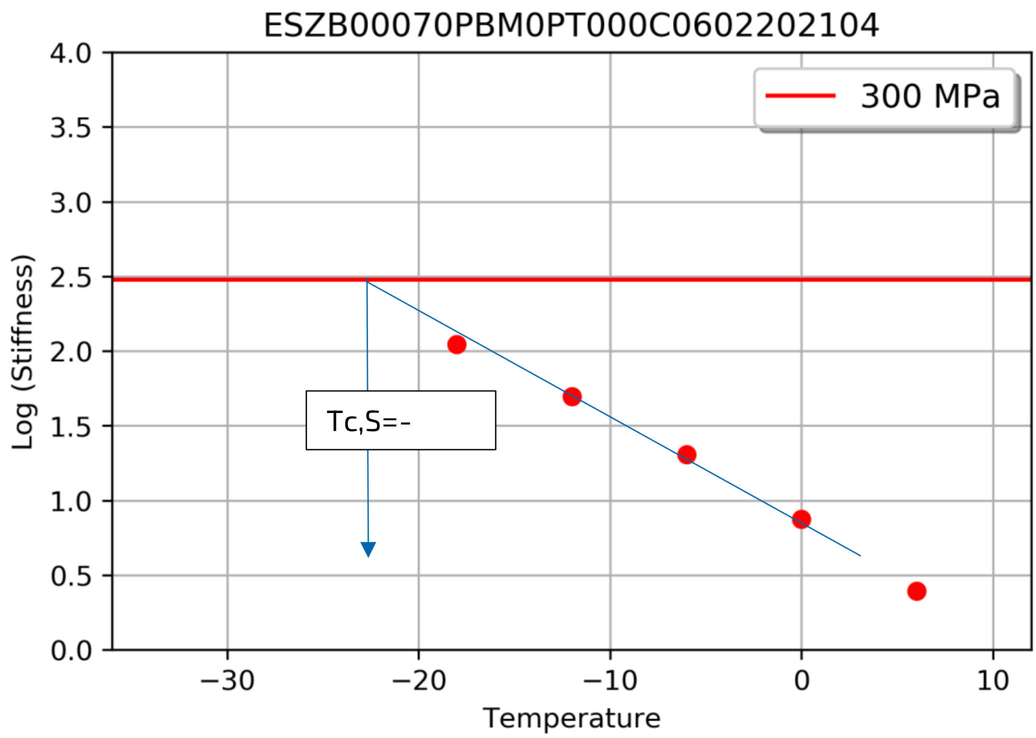


Figure 40. The sample bitumen 70/100 (data courtesy of MOREBIT project), demonstrating methodology to derive critical temperature for Stiffness using data obtained from 4 mm DSR measurements presented in Figure 36.

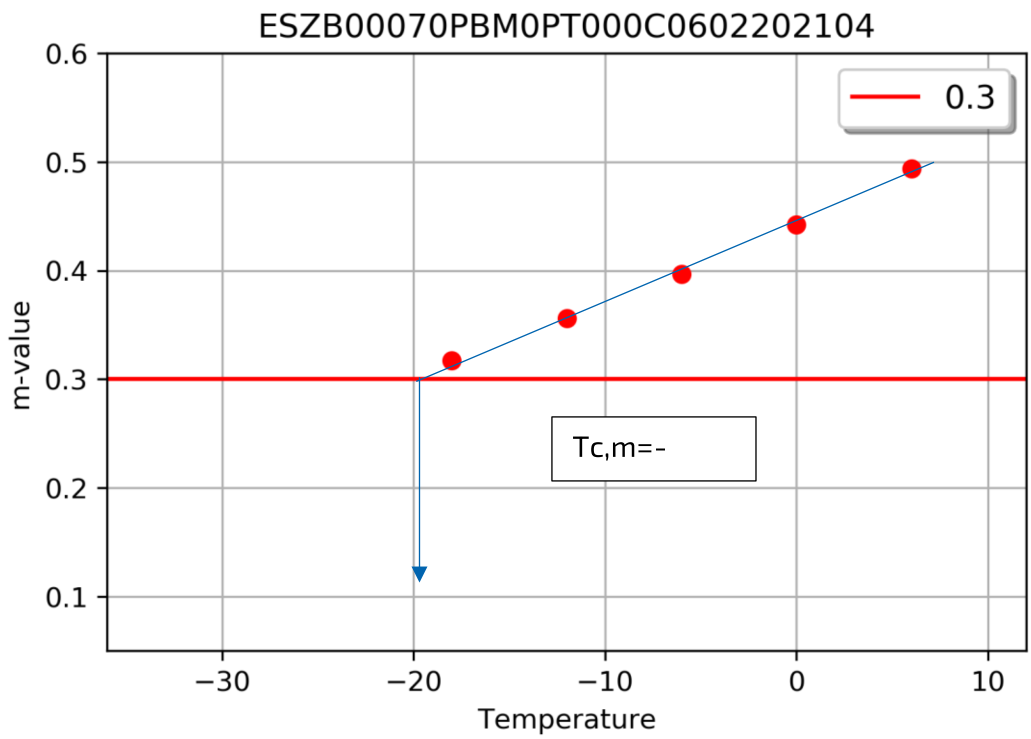


Figure 41. The sample bitumen 70/100 (data courtesy of MOREBIT project), demonstrating methodology to derive critical temperature for m-vale using data obtained from 4 mm DSR measurements presented in Figure 37.

Using the data from Figure 40 and Figure 41 we can obtain $T_{c,m}$ and $T_{c,S}$ necessary for the calculation of delta Tc (ΔT_c).

The ΔTc in this case is equal to -3.8 °C for fresh bitumen studied as an example in MOREBIT project (contemporarily ongoing as a follow up study to the BITU2020 project). This binder is stiffness governed, thus more prone to cracking. If the value is significantly on plus (above 5 °C) the binder would be rutting prone (Asphalt Institute Technical Advisory Committee 2019).

The value may be different when we start aging the binders. The differentiation between the binders is more apparent when RTFOT+PAV or RTFOT+2xPAV is applied (Asphalt Institute Technical Advisory Committee 2019). However, the criteria set for ΔT_c of aged binders suggests that the value should not be below -2 °C for good long-term performance (or resistance to thermal cracking) (Asphalt Institute Technical Advisory Committee 2019).

Below (Figure 42) are some of the binders on Finnish market measured from raw state, compared against each other in the comparison to the good performance criteria. The Asphalt Institute report indicates that the ΔTc is in fact independent of the climate, and that for values of below -5 °C the cracking will commence regardless of the climate conditions in which the pavement would be in.

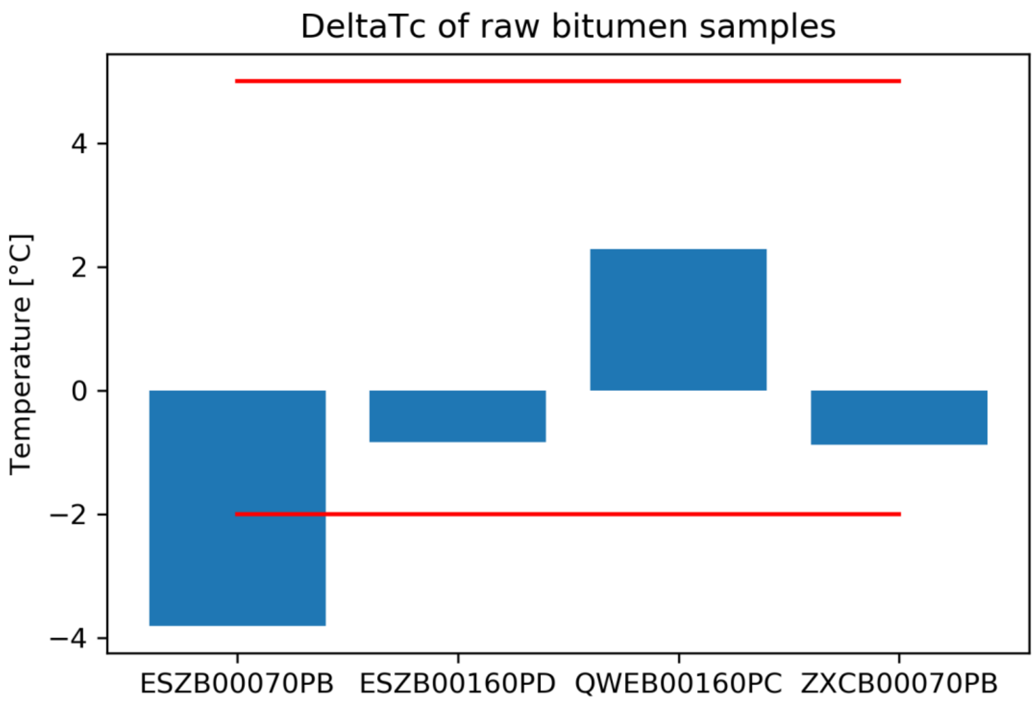


Figure 42. ΔT_c of raw bitumens on Finnish market determined using presented methodology plotted against the criteria of good performance indicating differences between the products (data courtesy of MOREBIT project).

7.1.11.4 Distinguishing between well and badly performing products

In the studies in which a comparison between field performance, recovered binder and laboratory aged binder characterized by the means of ΔTc, it was determined that the ΔTc < -2 °C or ΔTc > 5 °C indicates a binder which is likely to be stiffness or creep controlled, respectively. This means that binder is more susceptible to thermal cracking or rutting, respectively.

It was also determined that the PAV time of 20 h, applied in the PG system may be in fact too short to predict long term behavior of binders. The time of 40 h was providing more accurate to correlate well to the field conditions observed after 8 years in service (Asphalt Institute Technical Advisory Committee 2019). For this reason, binders were tested after 40 h PAV, and it was noted that many of the binders after this type of aging were below the cut-off of the ΔTc = -5 °C, even the ones which were supposedly not containing additives proven to be resulting in bad performance (waste engine oil – REOB – at levels above 8%). The industry was concerned about long testing time, so the suggested time was still set at 20 h PAV and cutoff kept at -5 °C. This caused the unwanted products to be excluded from use, while allowing for a shorter testing time (Asphalt Institute Technical Advisory Committee 2019).

Considering the Finnish reality in which the pavements can be deemed in necessity for hot in-place recycling after 4–5 years in service on high trafficked roads (KVL > 25000), or after 8–9 years on low trafficked roads (KVL < 2500) (Suikki & Spoof 2017), the evaluation of binders propensity to aging is suggested hereby for consideration, to be performed differently for different application. In the case of Class A roads the resistance to long term aging may not be the main parameter of interest, but on roads class B–D (Finnish Pavement Technology

Advisory Council 2017) the extended PAV times are expected to correlate better to the performance of the pavement in Finland, based on the results reported by Asphalt Institute Technical Advisory Committee (2019). Despite the fact, that the report claims that the climate had little effect on the likelihood of occurrence of block cracking if the ΔTc was anyway estimated at below -5 °C, it is advised to correlate the field behavior in field with the values, before deciding on setting up a quality requirement.

However, this elaborate report (Asphalt Institute Technical Advisory Committee 2019) is missing information related to the initial properties of the binder and the performance of the pavement which contains RAP material at extremely high levels typical for Finnish conditions, such as after the hot in-place recycling maintenance. In the end, despite being partially rejuvenated by addition of approximately 20% of fresh binders into the final mixture – the original binder constitutes large portion of the final blend in such case. The original binder (used for the paving during the first cycle) is constituting at least 50% of the total binder after two recycling maintenance cycles, around 15 years after the initial laydown. For the binders used on the Class A roads, it would be suggested to test the binder with 20 and 40 h in PAV, to evaluate which of the aging times relates better to the performance of binders in Finnish conditions.

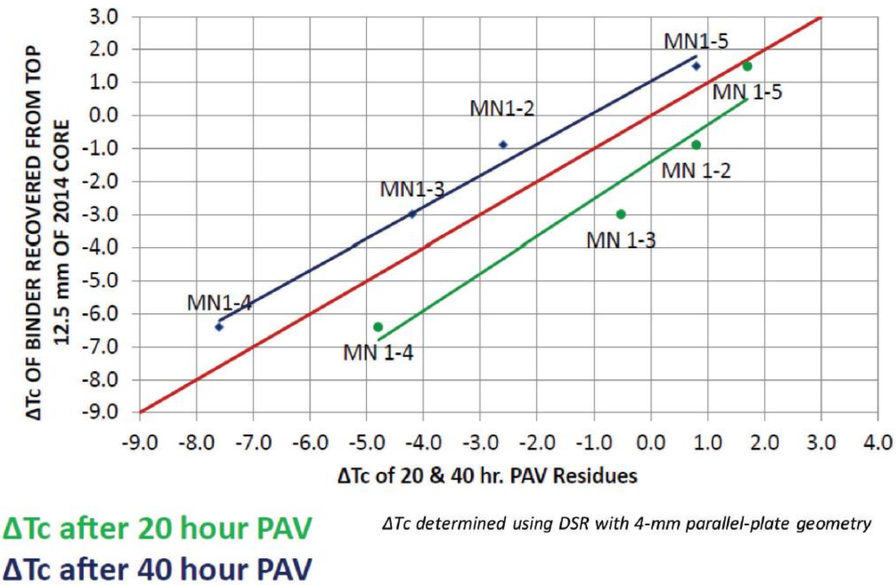


Figure 43. The comparison of the ΔTc parameter on the binders recovered from construction sites after 8 years in service and the laboratory aged samples for 20 and 40 h by PAV of the corresponding binders (Reinke et al. after Asphalt Institute Technical Advisory Committee 2019).

What is interesting to note, is that the parameters of the total binder in RAP are influencing the ΔTc of the binders extracted from the asphalt mixtures containing RAP. **The reports suggest that depending on type of RAP and binder, the effect may be visible already at 20% RAP content, and for that reason caution would be advised and collection of rheological data suggested also for binders extracted from RAP or mixtures containing RAP.** See Figure 44 after (Asphalt Institute Technical Advisory Committee 2019).

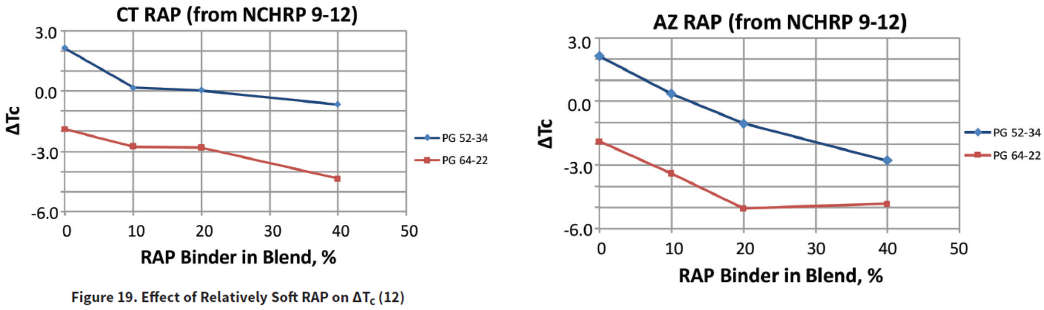


Figure 44. Adding relatively soft RAP into the blend is decreasing the ΔTc on within acceptable values up to 30%RAP (left), but for stiffer RAP the addition already at 20% RAP level may negatively affect the parameter (Asphalt Institute Technical Advisory Committee 2019).

7.1.11.5 Effect of modifiers on ΔTc

The Asphalt Institute (2019) commissioned testing of binders without and with multiple modifiers or binder extenders. The results indicate that the visbroken binder expresses highest susceptibility to thermal cracking as characterized by ΔTc after 20 h of PAV aging (see Figure 45). Interestingly, some of the polymer modified bitumen samples resulted in much worse ΔTc after 40 h of PAV aging (see Figure 46). As was discussed before, the threshold for 40 h PAV should probably be different or adjusted depending on performance need, but the decision to limit the time of measurement to 20 h (that correlated to predict bad performance at -2.5°C), resulted in leaving the limiting value at -5 °C (the value established as discriminative after 40 h of PAV). The level at which the criteria should be set is therefore not straightforward, as both interests of bitumen producer and Transport Agencies are subject to a compromise.

Interestingly, the presence of waste engine oil residue (RE-OB) in the bitumen was previously postulated to also contribute to lower resistance to thermal cracking and to be predictable by the means of BBR test repeated after specific time allowance for physical hardening to occur (Johnson & Hesp 2014). As demonstrated below, the ΔTc of such materials at low concentrations was not distinguishable, but at around 20% per binder content it was significantly affected (Figure 47). Some Departments of Transportation (DOTs) have acceptable experience with REOB modifier up to 8%, but recorded problems with values above that concentration. The ΔTc was used as a differentiator between those products. Overall, the tested binders were grouped into two types, of which one is m-controlled gel asphalt. It was observed that the air blown and REOB modified binders belong to this group. The gel type asphalts were also postulated to respond worse to the addition of REOB (see Figure 47) (Asphalt Institute Technical Advisory Committee 2019).

The Eurobitume commissioned the evaluation of 168 binders on the European market and determined that binders can be differentiated between based on obtained results (Robertus 2017), with values ranging from about -17 to 4 °C. Because the procedure and result are linked to performance of the binder, the method is therefore of high interest when the future standardization is discussed.

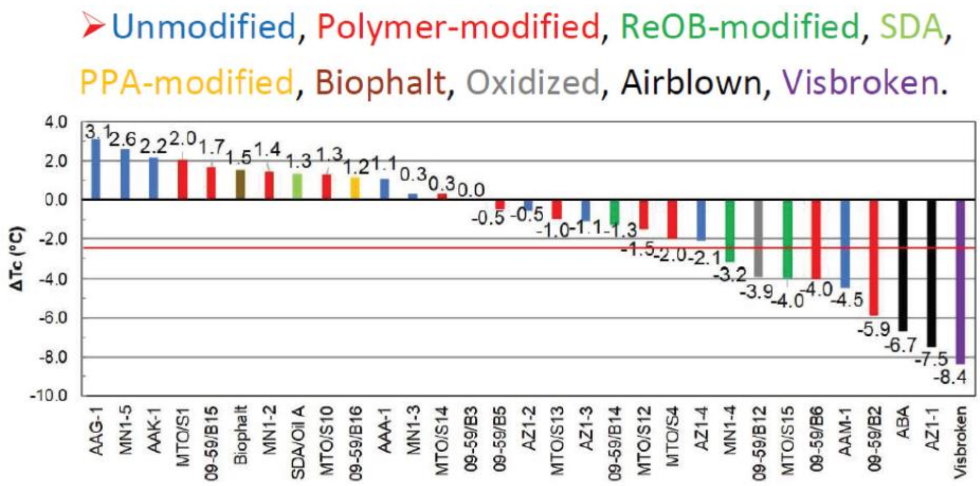


Figure 45. The delta Tc after regular PAV 20 h of various binders from NCHRP 9-60 Research Project Database (Asphalt Institute Technical Advisory Committee 2019; Planche et al. 2019).

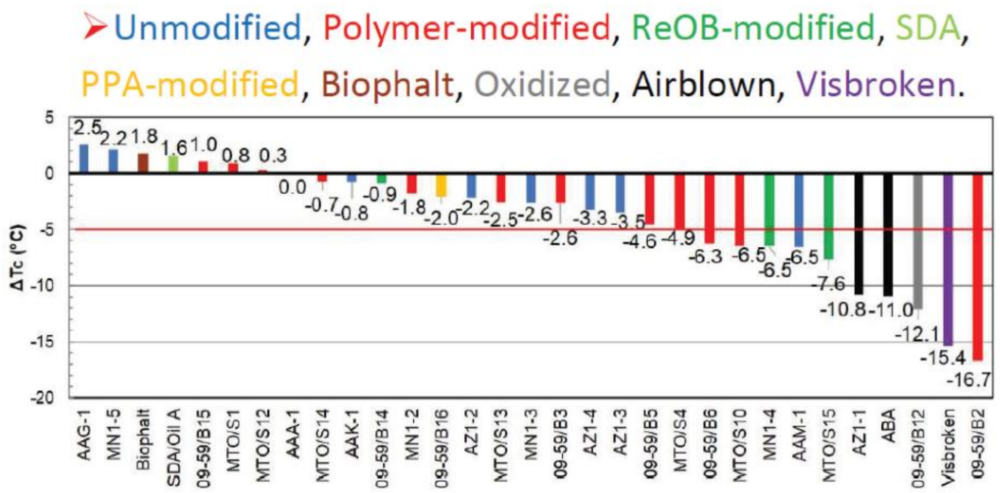


Figure 46. The delta Tc after extended PAV 40 h of various binders from NCHRP 9-60 Research Project Database (Asphalt Institute Technical Advisory Committee 2019; Planche et al. 2019).

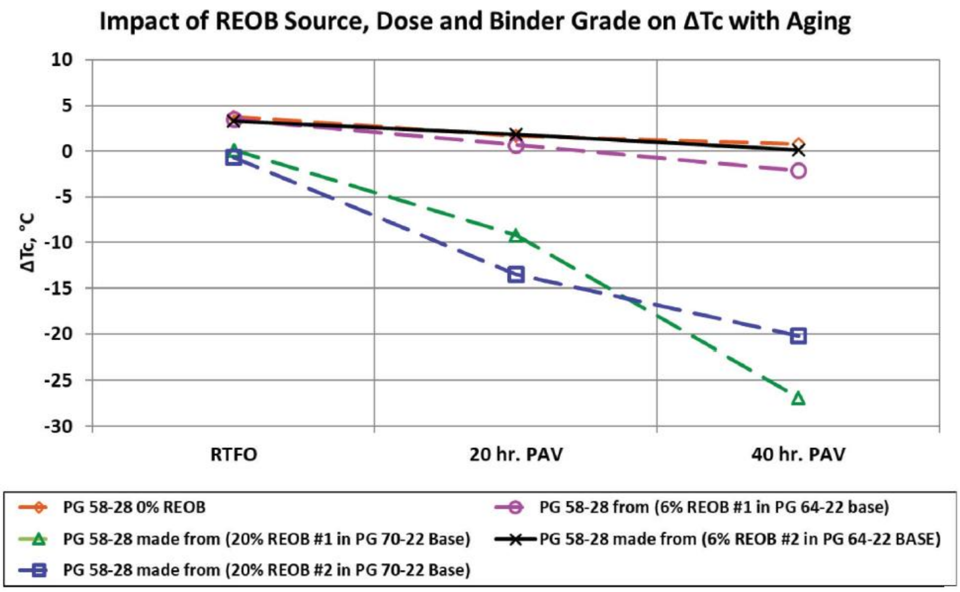


Figure 47. The effect of Re-refined engine oil bottoms (REOB) addition on the delta Tc indicates that extensive modification may lead to the bad low temperature performance (Asphalt Institute Inc. and European Bitumen Association–Eurobitume 2015).

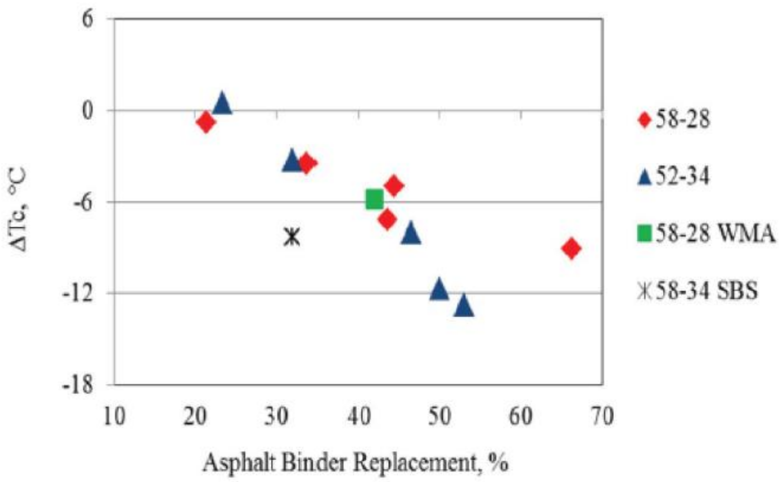


Figure 48. The effect of the binder replacement by the binder from RAP on the delta Tc parameter (Asphalt Institute Technical Advisory Committee 2019).

The ΔT_c was then compared with distresses in the pavement. More negative value of ΔT_c correlates with more distresses observed on the surface (Figure 49 and Figure 49) (Asphalt Institute Technical Advisory Committee 2019).

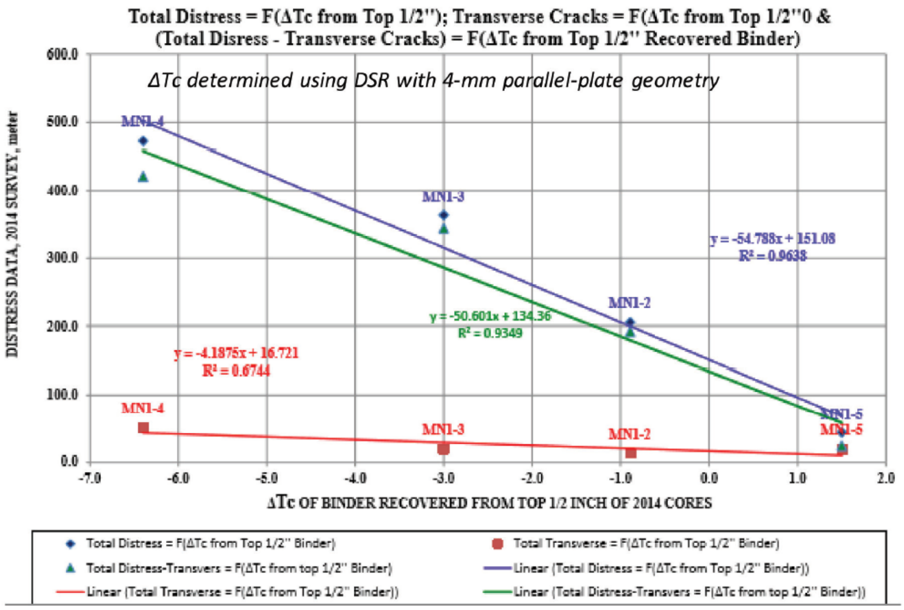


Figure 49. Relationship between ΔTc and distress data of the pavements (Asphalt Institute Technical Advisory Committee 2019).

7.1.12 Finnish experiences with PG-grading

In 2003 a report on co-operation between Finland and Italy was published. The University of Ancona, Politecnica di Torino, Valli-Zabban on the Italian side, as well as Finnish National Road Administration, Fortum Oil and Gas Oy and Helsinki University of Technology on Finnish side, joined forces to investigate the applicability of PG-grading for the bitumens in Europe (Levomäki et al. 2003). A set of three unmodified binders 40/50, 50/70 and 160/220, as well as five polymer modified bitumens from both producers, were investigated.

In 2002–2003 the amount of PAV equipment in Finland was equal to zero, and there was one DSR equipment. One of the objectives was to investigate if in fact the investment into the PAV should be conducted.

It was found that:

- the DTT from PAV aged samples correlated very well with DTT result of the fresh samples
- the limiting stiffness temperature correlated very well with Fraass breaking point of fresh binder
- a correlation between viscosity at 60 °C was correlated to the rutting parameter from PG-grading
- the SHRP fatigue parameter was related to the Penetration value at low temperatures.

Based on this a conclusion was reached that aging tests do not bring any additional knowledge and the DSR results could be predicted from typical laboratory measurements, therefore a suggestion was to continue to make the evaluations from the raw bitumen using the tests required for the Penetration grade characterization (Levomäki et al. 2003).

From among the tested binders the ΔTc values after each aging procedure were calculated hereby even if they were not computer at the time of the report preparation, and the data is summarized in Table 8. One of the binders is expressing quite different behavior in all the measurements, namely PMB3.3., but besides that the resistance to long term aging was hard to estimate based on the unaged binder result. After RTFOT and PAV treatment indeed it is more visible, which binder retains the properties and which could be susceptible to long term aging. It is worth noting that with each aging cycle, the ΔTc parameter seems to be decreasing or staying on similar level (considering the repeatability of 0.8 °C for the BBR measurement) (Asphalt Institute Technical Advisory Committee 2019).

Table 8. The results of the BBR tests for the binders tested by Levomäki et al. (2003) and in orange the marked values exceeding the ΔTc ≤ -5.

Binder	Unaged			RTFOT			RTFOT + PAV		
	Tc,S	Tc,m	ΔTC	Tc,S	Tc,m	ΔTC	Tc,S	Tc,m	ΔTC
160/220	-22,9	-21,5	-1,4	-23	-23,3	0,3	-21,2	-19,3	-1,9
PMB3.1	-27,2	-22,2	-5	-26,7	-22,5	-4,2	-25,1	-20,9	-4,2
PMB3.2	-27	-22,5	-4,5	-27,4	-22,7	-4,7	-25,9	-18,1	-7,8
PMB3.3	-28,5	-22,4	-6,1	-35,9	-28,2	-7,7	-31,6	-20,5	-11,1
B40/50	-14,1	-16,6	2,5	-13,9	-14,3	0,4	-10,5	-11	0,5
B50/70	-17,1	-19,2	2,1	-16	-17	1	-14	-14	0
PMB30/50	-15,1	-14,5	-0,6	-14,7	-12,9	-1,8	-13,8	-8,6	-5,2
PMB50/70	-19,8	-20,1	0,3	-19,4	-18,9	-0,5	-18,7	-16,9	-1,8

7.1.13 Binder-Fast-Characterization Test (BTSV)

The motivation behind the development of the test is the substitution of the Softening Point (SFS-EN 1427) measurement through DSR equipment, to distinguish between complex (e.g. polymer modified) and simple binders. The Softening Point repeatability for PmBs is less reliable than for the straight run bitumen samples. Additionally, the difference in viscoelastic properties between such binders is not reflected by a temperature of Softening alone. For this reason, the Binder-Fast-Characterization-Test (BTSV) is attempting to look at both change in complex modulus and phase angle in a two-dimensional space, while sustaining a similar sample preparation procedure as a well-known Softening Point measurement (Schrader & Wistuba 2019).

Methodology uses DSR equipment with 25 mm plate-plate geometry with the gap of 1 mm. The procedure requires:

- Increase of the temperature in a continuous way from 20–90 °C with the rate of ΔT = 1.2 °C/min (approximate test time of 60 minutes)
- Constant shear stress of 500 kPa and frequency of 10 rad/s is applied in an oscillatory movement
- Recording of three parameters every 2.5 s: temperature (T), complex shear modulus (G*) and phase angle (δ) – test can be aborted if G* < 1kPa.

Using the collected values, a temperature and phase angle are collected for the condition of G* = 15kPa. The temperature at which the complex shear modulus of the material reaches 15 kPa is then referred to as T_{BTSV}, and the phase angle corresponding to G* = 15 kPa is referred to as δ_{BTSV}. The two parameters are then plotted against each other as in Figure 50, and based on the analysis of multiple products, ranges of values were assigned to different types of products. As can be witnessed from the figure, for the same softening point equivalent T_{BTSV} different bitumen products can be recognized by looking at a second parameter, namely phase angle (Schrader & Wistuba 2019).

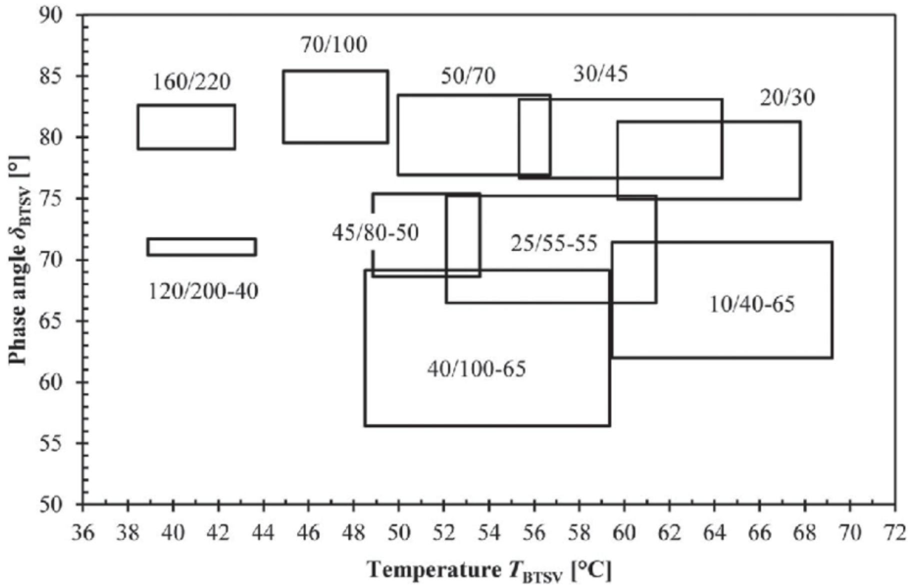


Figure 50. Example of differentiation between binder grades within the BTSV space allowing to separate polymer modified from straight run bitumens (Schrader & Wistuba 2019).

This test, along the PG-grading idea of characterization of binder by DSR, is allowing to differentiate between the binders of different composition. The principle suggests that the position of the specific point in the black space (G* versus phase angle, measured at fixed frequency), similar to Glover-Rowe parameter principle, should be able to differentiate between binders. Such approach using one equipment and one measurement procedure allows one to test the unknown binder without assumptions in respect of its structure, as opposed to the different set of standards being required to characterize the Penetration graded binders and Polymer modified Binders in European specifications. Such approach would be of interest in product recognition using the machine learning, due to the visible separation into the rheological classes and products when performing just one measurement.

7.1.14 Effect of additives on the rheological properties of binders

Typically, when we speak about the additives, their effect is discussed by the prism of the property that they enhance in the asphalt. For example, the addition of the adhesion promoting additives is usually analyzed by their influence on for example ITSR. The influence on rheology is rarely discussed in the context. The effect to low temperature properties or rutting are typically outside of the scope of the investigations. On the other hand, the modifier can be marketed as three different products influencing each performance criteria separately. For this reason, for example one can think of adhesion promotors being marketed as warm mix promoting additives on two different markets, depending on the aspect which generates demand for the product.

Therefore, the reader of hereby report should exercise caution over the results reported in the literature that do not look at the effect to the asphalt concrete performance in a holistic way, i.e., considering more than one performance parameter. The user should be weary to notice that by enhancing one parameter, something else may be affected as well – either positively or negatively, but the effect should be investigated to fully evaluate the construction risks.

Remisova and Holy (2017) looked at the effect of adhesion promoting additives on the traditionally tested rheological parameters, such as Penetration, Softening Point and Kinematic Viscosity, of three different binders 35/50, 50/70 and PmB 45/80–75. The tested additives with their description and potential application are listed in Table 9. The additives were added to the binders at mixing temperatures ±5 °C.

Table 9. The adhesion additives investigated by Remisova and Holy (2017).

Name	Type of compound	Improves	Applied amount
Sasobit	Fischer Tropsch paraffin, wax	Adhesion to aggregate	3% by binder weight
Licomont BS100	Fatty amines based derivative	Temperature stability -30 and 80 °C, adhesion to aggregte Decreases bitumen viscosity	3% by binder weight
Wetfix BE and Wetfix AP-47	Fatty amine derivative	adhesion	0.4% by weight of binder
CWM	Surface active additive decreasing surface tension between bitumen/aggregate	Covering the aggregate evenly, compaction properties at lower temperatures, adhesion to aggregate	0.4% by weight of binder

The effect of wax addition is witnessed by:

- Decreases Penetration (significantly)
- The Softening Point increases
- Decreases the kinematic viscosity.

The effect of addition of Licomont BS100:

- Increase of penetration for non-modified
- Decrease of penetration for modified
- Increase of softening point for all tested binders
- Decreased Kinematic Viscosity

The effect of addition of the other modifiers:

- Increase of Penetration
- Decreased or similar Softening Point

- Decreased Kinematic Viscosity

As is visible in the research of (Remisova & Holy 2017), the effect on the rheology of the final blend is not only a function of an additive, but also of the type of binder. Adhesion promoting additives often change the viscosity at higher temperatures, which causes them to be marketed occasionally as Warm Mix Additives. By achieving lower viscosity at mixing temperatures, the producer of asphalt may reduce the production temperature without jeopardizing the mixing process.

Likewise, in comparison between evaluated waxes and other modifiers, we can see different effect to the softening point – namely either decrease or increase. Therefore, those products are marketed as rutting improving or alternatively compaction improving. The producer should be advised, that the two are standing on the opposite side. Something that improves compaction, may cause the mixture to be more susceptible to rutting.

Interestingly the presence of waxes had the decreasing effect on Penetration in that studies, and that should be therefore followed by investigation on the effect of addition to low temperature performance. If the material is overly stiff at low temperatures, this may lead to thermal cracking.

On the above example it is worth to underline, that the addition of each additive was optimized by the producer of said additive. The use of additives without cooperation with their producers (at least) or own product development department in terms of determining the optimal content of them in the mixture design, may lead to the over-addition. For this reason, the approach of suggesting the use of minimum X% of additive in all of the mixtures (e.g. when publishing specifications) is posing a risk of the material failure in respect of the low temperature properties or rutting resistance, and without appropriate decrease of the mixing temperature, perhaps even drain-down of binder.

7.2 Spectroscopy

7.2.1 The interaction between photons and matter

The definition of the spectroscopy can be generalized as interaction between the radiative energy as a function of its wavelength or frequency. The electromagnetic waves can be described by Photon Energy (E), wavelength (λ) and frequency (f). The relationship between those parameters can be described by the below equation:

$$E = \frac{hc}{\lambda}, \qquad (14)$$

where

c is the speed of light (299792458 m/s),
h is the Plank constant ($6{,}62607015 \times 10^{-34}$ J·s = $4{,}13566733(10) \times 10^{-15}$ eV·s)

and the relationship between frequency and wavelength is given by the equation

$$f = \frac{c}{\lambda} \qquad (15).$$

This rather simple relationship indicates that the higher the wavelength, the lower the radiative energy is, and vice versa. In Figure 51 different radiation types are compared to the size of the matter that they interact with. The gamma ray radiation is of the wavelength similar to the atomic nuclei. The energy of the gamma ray wave is capable of ionizing the atom, by providing enough energy to the atom to eject the electron from the orbitals. The difference between incident Photon energy and Photon energy after interaction with the atom, is related to the electron energy in the atom. And the energy necessary to eject the electron relates to the positive charge in the atom due to the number of protons in it. For that reason this wavelength can be used to study the nuclei.

However, it is important to note, that techniques connected with X-ray as incident light are challenging to study atoms with mass lower than 16 Daltons (atomic units), that is, smaller than oxygen. This includes carbon, nitrogen and hydrogen – all components of organic molecules such as those in bitumen. Such techniques as X-ray Diffraction or X-ray fluorescence are much more reliable for heavier atoms. However, if a need to determine more accurate information about the carbon, oxygen and nitrogen in the molecules exists, especially such that are absorbed on some surface, X-Ray photoelectron spectroscopy has been for example proven as a very powerful technique in the studies of the doped carbon nanotubes (Terrones, Souza Filho & Rao 2008).

As the wavelength increases, the interaction between photon and matter is changing. The high energy waves are considered as the ionizing waves. The gamma ray, affects to the electrons of the highest energy in the atoms – for example those located closest to the nuclei of the atom. When the wavelength of the photon increases, its energy decreases and only the electrons on the orbitals further from the atoms core can be excited. Those electrons are bound to the atom in a much weaker way, as their distance from the core of the atom is larger. Simply said, the pulling forces of the positive charge in nuclei have lower effect on the electrons located at the outer orbitals.

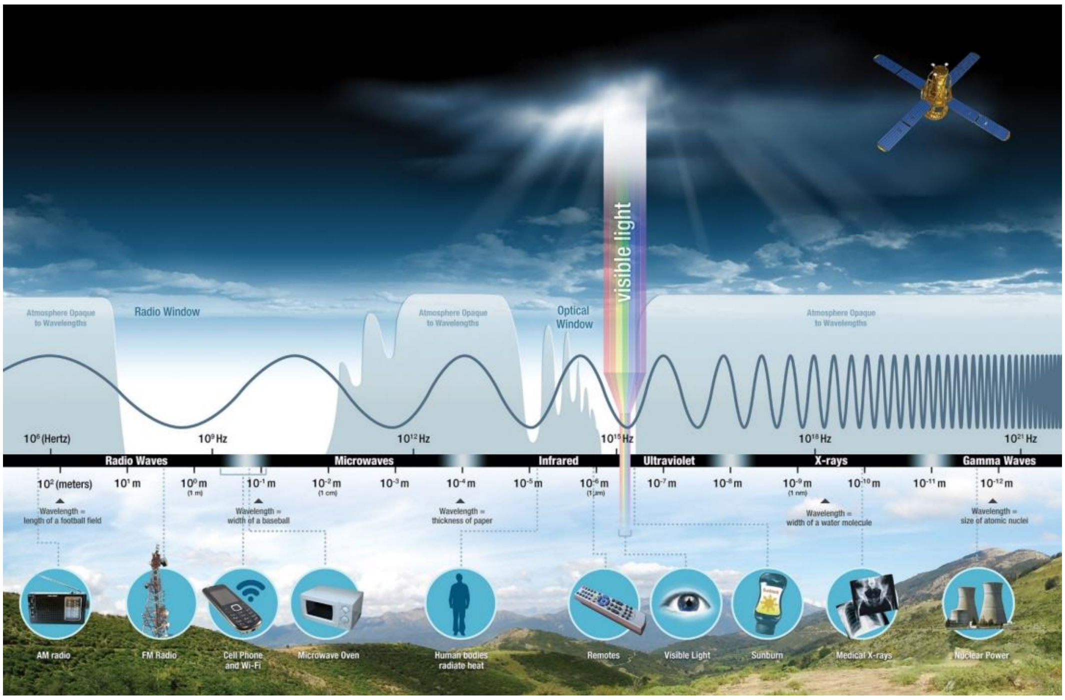


Figure 51. The wavelength of the various radiation types and their use (NASA Accessed last: 19.5.2021).

When the wavelengths are from the range of ultraviolet and visible light range, that is λ between 100 and 1000 nm, a different type of electron excitation is observed. When we speak about analysis of atoms, we speak of atomic orbitals (s, p, d, f) and transitions of electrons between those orbitals can be measured spectroscopically using photons with energy between 1.24 eV – 12.4 eV. When molecules are considered in interaction with photon, the absorption of the energy can be equal with the transitions between the molecular orbitals, especially the bonding (σ, π, δ, ϕ) and antibonding (σ*, π*, δ*, ϕ*) molecular orbitals (MO). Each orbital can be occupied by two electrons. When the molecule has an occupied only bonding MO, it usually is stable and non-reactive. For example, hydrogen atom having only one electron, forms a molecule of H_2 with σ and σ* MO. However, only the σ is occupied by the two electrons while σ* is unoccupied.

A shift of the electron to the antibonding MO can be considered excitation of the molecule, leading to its higher reactivity. Again, the location of the antibonding MO is further away from the core of the molecule (multiple nucleis) and therefore bound in a weaker way to the molecule. Such electron on antibonding MO can "jump" (or be withdrawn) to a different molecule or atom or positive ion (system missing an electron, and therefore with higher pulling forces towards electrons – referred to also as acceptor). This energetical gap, between the highest occupied molecular orbital (HOMO) and lowest unoccupied molecular orbital (LUMO) is often within the range of photon energy with the wavelengths of the ultraviolet and visible light range (UV-Vis), especially for organic molecules (Williams & Fleming 1966). The light can be absorbed and stored in form of the excitation, but to reduce the energy a reaction between molecules is likely to occur to reduce the energetical state.

For this reason, the UV-Vis light illumination on the sample, especially many of the organic molecules, initiates reactions or acts as a promoting factor, for example oxidation reactions of organic molecules or polymerization. **In case of bitumen for example, the oxidation reaction propels with higher rates when the bitumen is exposed to UV-Vis light** (J. C. Petersen 2001).

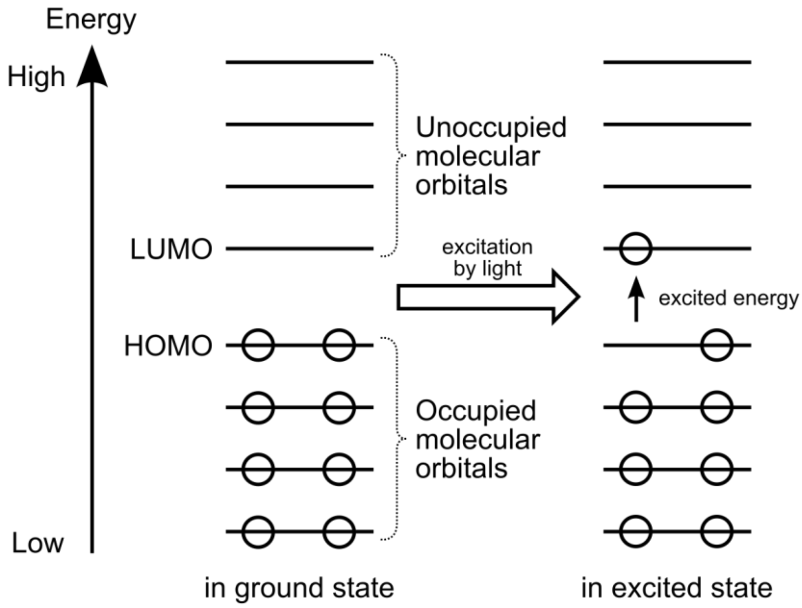


Figure 52. The transition between HOMO and LUMO upon the irradiation by UV-Vis light (Wikimedia Commons 2021).

Alternatively, if the reaction cannot occur between two molecules, the excited state is relaxed by a release of the energy and shift of electron to bonding MO. The likely form of energy release from the system irradiated initially by the photon can occur by fluorescence (or luminescence). The gaps between orbitals can be calculated, and the energy difference between them estimated. Based on this information the determination of the transition in the molecule responsible for both absorption and fluorescence is plausible and a deduction of structure can be aided by this spectroscopic method.

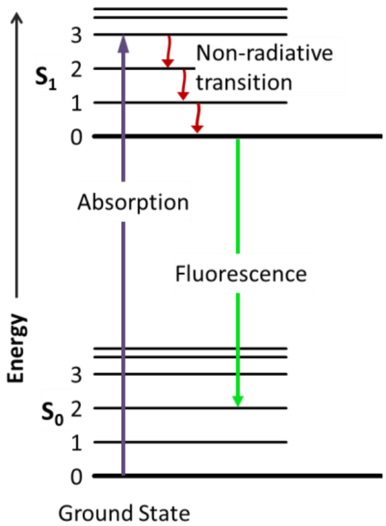


Figure 53. The Jablonski diagram of fluorescence demonstrating that not all transitions are occurring with release of the visible light photon, causing the

fluorescent photon to be of lower energy than the absorbed photon (Wikimedia Commons 2021).

When the wavelengths move towards the micrometer range (that is within the infrared range discussed a little bit further), the energy is no longer sufficient to move the electrons, but the energy can be used on other things such as movement or vibration. To help in imagining by analogy, consider that you would need more energy to pull somebody's hand out of their body than to spin them around in dance. Analogically, UV light can withdraw the electron from molecule, while the IR light can twist or spin the molecule.

During the irradiation by photons of even longer wavelengths such as from near infrared (NIR) and infrared range (IR), the energy supplied is no longer sufficient to excite the electrons within the molecule (continuing with the analogy – "to dislocate their bones"). However, every molecule has vibrational modes and energy in the system can be consumed by executing one of the allowed vibrations, for example symmetrical and asymmetrical stretching, scissoring, rocking, wagging or twisting. The energy absorbed into the system is transferred into a kinetic movement, which in turn turns into heat due to the internal friction between the molecules.

Analogically to the difference in energy required to spin a bridge, an elephant and a mouse around their axes, the size of atoms in the molecule, the number of atoms and combination of atoms in the molecule will determine the energy levels required for the molecule to be excited to the vibrational mode. Again, by analogy it is easier to spin a couple of dancers around than a train of 30 people, and there is much more combinations of movements between corresponding people in the train, than just when the couple is considered. A large bridge perhaps cannot be spun around, but it can vibrate, shrink and stretch. the amount of energy absorbed by the couple to spin or a bridge to vibrate would be very specific to their shape and size, and thus allow us to deduct which of the object actually was studied, by analysing the amount of energy absorbed in the system.

Small molecules, such as gases, have therefore very discreet infrared spectra with limited types of movements allowed within the molecule and insignificant effect from other molecules in the measured fluid. If we compress the gas into the liquid phase the interaction between molecules and the space between them will affect to the allowed vibrational modes (allowed movement). If we also combine small molecules into the larger ones, such as ethane to octane, and we again move from gas to liquid, not only geometric aspects play role in allowed vibrations, but the effect of other atoms in the structure starts to be of significance. Consider this analogy: a dancer can lift his partner above own head with energy X. Would the dancer need less or more of energy to lift the same partner if the partner was in fact being held by a hand by a third partner?

7.2.2 Types of spectroscopies

As explained in previous section, the spectroscopy is a study of the interaction of light with matter. However, a microscopy can be also described with similar definition. Therefore, to distinguish between microscopy and spectroscopy, certain additions to the definition are needed.

The spectroscopy is describing the level of interaction of photon as a function of its wavelength or frequency. Essentially, during spectroscopic measurements the energy of the photons before and after the measurement are measured, and the change in the intensity of the photons is followed. Sometimes the incident light and scattered light angle are considered.

The change in the intensity can be measured as absorbance, transmittance, reflectance, diffraction, fluorescence, Auger electron spectroscopy or Raman scattering. Let us consider differences between those techniques, methodologies or applications of the studies of photons.

Energy submitted to the system can be either absorbed, non-absorbed therefore transmitted through the material, or emitted from material after relaxation. Based on scintillating those phenomena we distinguish Absorbance, Transmittance and Fluorescence.

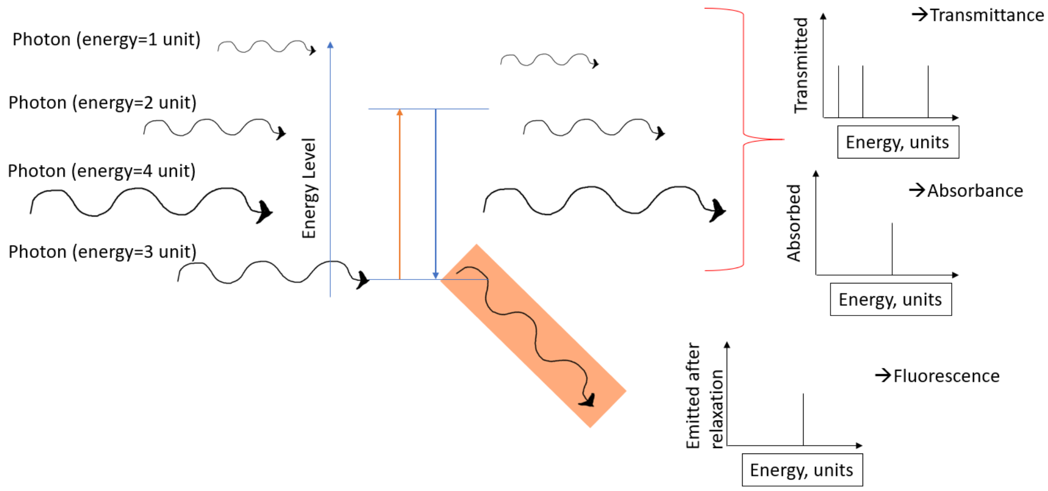


Figure 54. The schematic explanation of differences between Transmittance, Absorbance and Fluorescence based on the method of energy interaction with the matter.

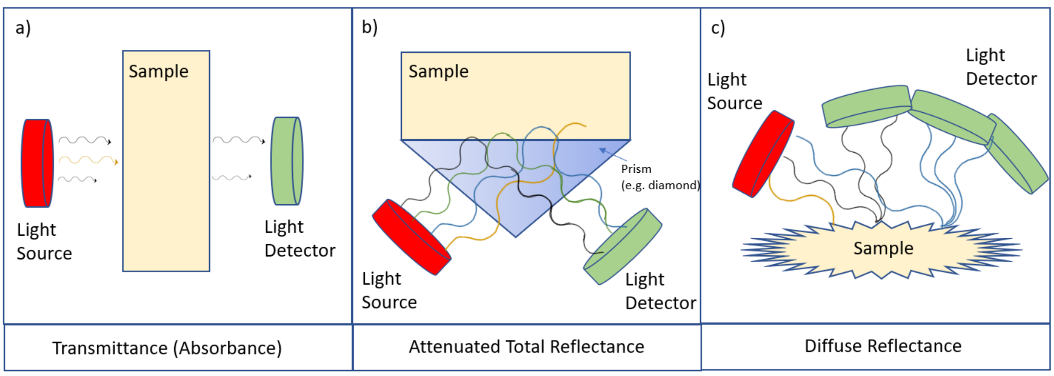


Figure 55. Spectroscopic techniques available for measurement of Absorbance as a result of different equipment design: a) Transmittance, b) Attenuated Total Reflectance (ATR) and c) Diffuse reflectance (DR).

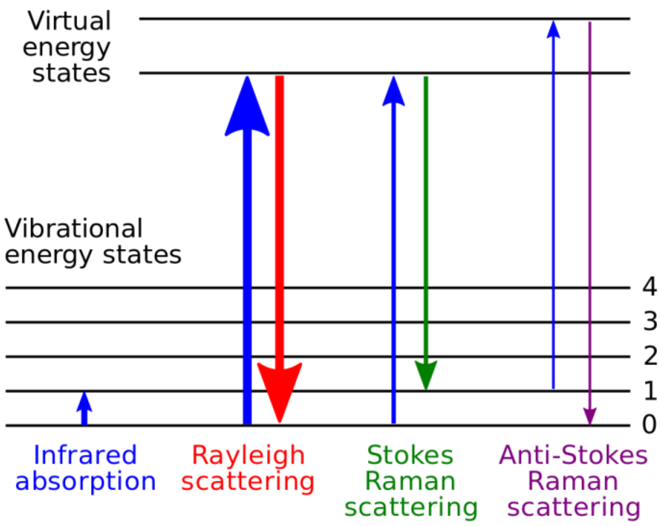


Figure 56. The energy level diagram explaining the transitions responsible for infrared and Raman spectra, explaining the difference in spectroscopic technique based on the type of relaxation after excitation.

7.2.2.1 X-Ray Photoelectron spectroscopy (XPS)

One of the spectroscopies used to determine atomic composition and bonds in between atoms in the tested materials is X-Ray Photoelectron Spectroscopy (XPS). The distance of each molecular orbital from the center of the atom is characteristic for each of the atoms. The more protons, the stronger the pulling of electrons is, therefore an atom with 8 protons like oxygen and atom with 6 protons like carbon are having the 1s orbital at a different distance from the core, as a result the energy of electrons on their 1s orbitals is different.

During XPS a monochromatic beam of well characterized focused X-Ray light is illuminated on the tested surface. The photons of the beam lead to ejection of the electrons from various orbitals. The kinetic energy of the electron beam is then measured. By comparing the original photon energy to incident light energy, considering own corrections inflicted by parameters of the equipment, one is able to calculate the binding energy of each recorded electron.

As an effect, the spectrum is obtained, as demonstrated in Figure 57 and Figure 58, which relates the number of counts to certain energy. Analyzing such result allows one to understand which atoms constitute the material and relatively in which ratio. Additionally, if an atom is a part of a molecule, e.g. compare metallic Fe and salt FeCl3, due to the presence of extra electrons the energy of molecular Fe electrons may be lower than in atomic Fe. It now has extra electrons but the same number of protons, thus it is now pulling each electron with lower force. The Cl atom after contributing its free electrons from the highest orbital contains now less electrons with the same proton number, leading to contraction, and a necessity to supply higher energy in order to eject the electrons at lower MO in Cls structure. Effectively, pure elements have different binding energies than compounds of those elements. Using the data from catalogues and tabulated values for each molecular bond, one can deduce the molecular composition of tested specimen, or characterize bonds present in the material. This technique is good to use if it is not known what is the composition of the material studied, especially its surface.

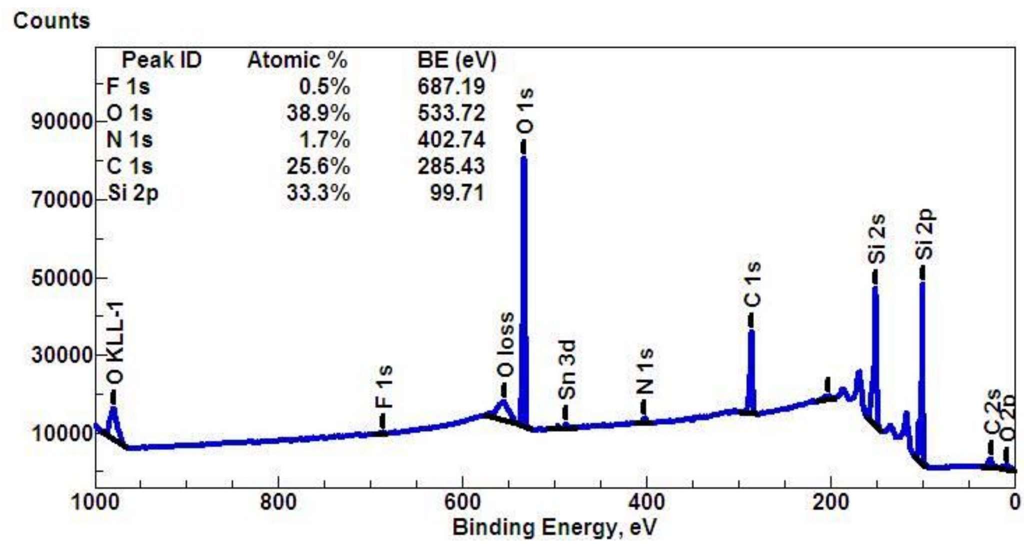


Figure 57. The example of the wide-scan XPS spectra of silicon wafer in which number of electrons emitted from studied surface is plotted in function of the electron energy, and from which a first evaluation of atomic composition can be conducted by assignment of the peaks to corresponding energy levels of various atoms and the atomic percentages. Picture source: Wikicommons (2021).

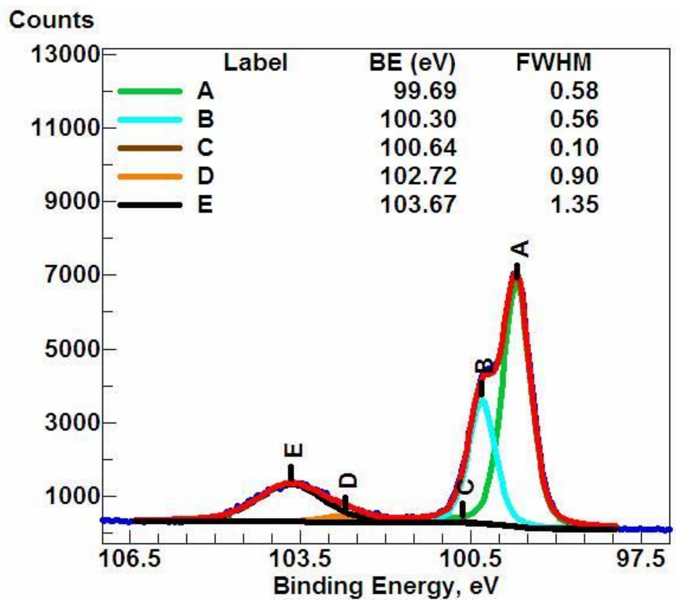


Figure 58. High-resolution spectrum of an oxidized silicon wafer in the energy range of the Si 2p signal. The raw data spectrum (red) is fitted with five components or chemical states, A through E. Each of the chemical states can be assigned to a certain bond of Si with other atoms, and from this a composition of the measured material can be deduced. Picture source: Wikicommons (2021).

7.2.2.2 Ultraviolet and visible light (UV-vis)

The ultraviolet (UV) and visible light (vis) wavelengths span from around 10–400 nm and from 400–750 nm, respectively (see Figure 59). The range between 750–1400 nm is defined as the near infrared (nearIR). The nearIR spectra can be measured by special separate equipment or a combination of UV-Vis-NearIR equipment with nearIR-IR equipment.

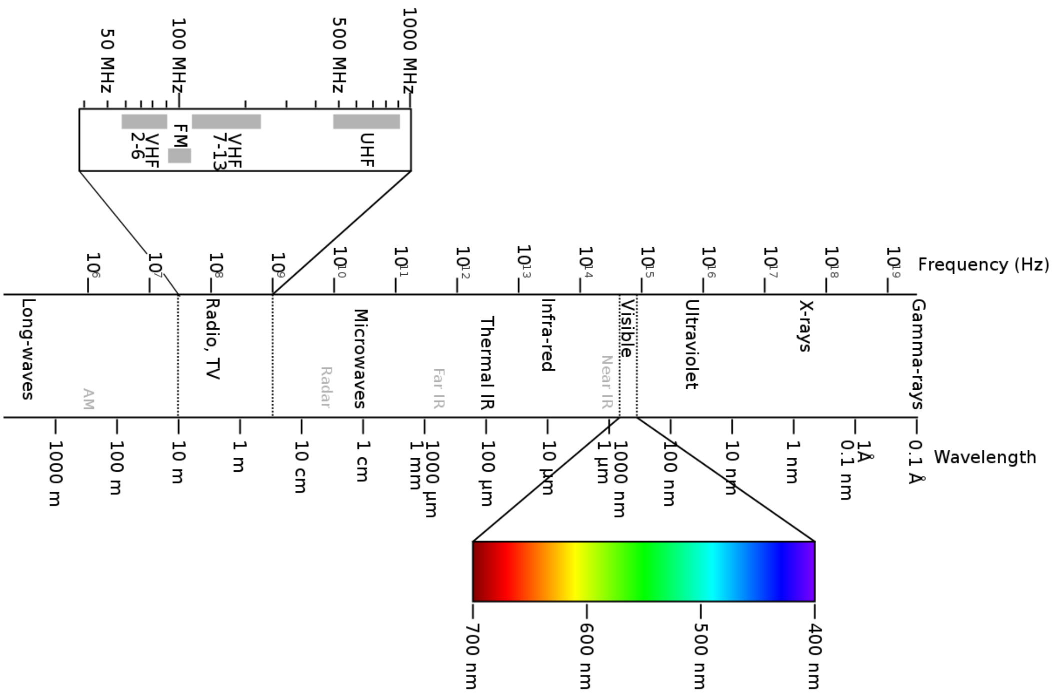


Figure 59. Electromagnetic spectrum range. Source: Wikimedia commons.

Within the UV-Vis range a material can be measured using few methods: absorption (covering absorbance, transmission, reflectance) and fluorescence.

The first explores the energy that the molecule absorbs in order to move the electrons from the highest occupied molecular orbital (HOMO) to the lowest unoccupied molecular orbital (LUMO), see Figure 52. The HOMO in stable molecule is called bonding orbital (BO) and the LUMO is called Antibonding Orbital (AO). When such energy is absorbed, a molecule is in excited state. In such case a molecule is more likely to react with its environment to pair the electron with other excited molecules to form another molecule. As a result of such reaction the final molecule achieves lower energy state, and a portion of energy is released. Hence, once the molecules are in the excited state the reactions are exothermic, i.e. release energy.

The energy of the light is stronger in the UV range than in visible range as the wavelengths are shorter. UV light is often used to start a reaction between molecules, e.g. polymerization or oxidation. Once the excitation is provided, the energy released by the reaction itself propagates the process further. In the context of bitumen, the oxidation of bitumen occurs slower or along a different path in the absence of UV-vis light (J. C. Petersen 2001).

Alternatively, the excited molecule can emit a photon of energy and return to its original ground state. This phenomenon is called fluorescence. Just like the light illuminated at the molecule and absorbed during excitation, the light emitted during fluorescence is of the UV-Vis range, thus even possibly visible to human eye. Some of the bitumen molecules are expressing the fluorescence in the UV range (not visible to human eye) and thus their presence and absence can also help distinguishing between different products (Blomberg & Turpeinen 1992).

For isolated molecules UV-Vis spectroscopy gives an additional information about the types of bonds within the molecule. As will be explained in the following chapter on infrared spectroscopy, and in Figure 61, the chemical bonds to be expected within the bitumen are coming from conjugated rings (polyaromatic structures), ketones, aldehydes, pyridines and quinones, among others. All the mentioned bonds can be characterized by the absorbance in the UV-Vis region between 200–400 nm.

Because oxidation of bitumen was proposed in the past to not only introduce heteroatoms into the structure of molecules – leading to formation of quinones, carboxylic acids, ketones, sulfoxides, etc. – but also leading to aromatization, the analysis of the spectra coming from UV-Vis spectroscopy may be challenging due to the overlap of the signal from those products with other organic structures. However, it seems that the increasing number of aromatic rings within the structure should shift some of the absorption peaks towards the higher wavelengths, i.e. 400–500 nm (Williams & Fleming 1966).

In fact, the differences between the bitumen types and sources, and the effect of the aging on the UV-Vis spectra, was demonstrated to be of significance in respect of the large conjugated systems (Soenen & Redelius 2014). Additionally, a phase angle was correlated to the absorbance intensity at various wavelengths, see Table 10. The methodology could be evaluated for the purpose of screening the products in terms of their elasticity at lower temperatures and higher frequencies, as is desired. The UV-Vis measurement is relatively faster than the DSR one.

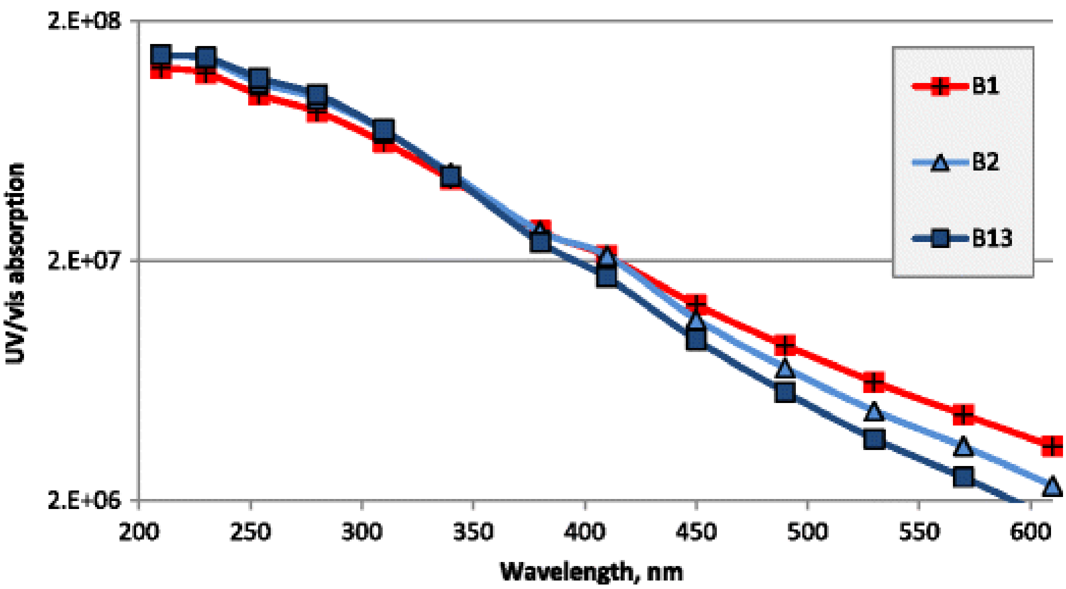


Figure 60. The UV-Vis absorption in logarithmic scale for three different bitumen (Soenen & Redelius 2014).

7.3 Infrared spectroscopy for bitumen analysis

7.3.1 Principle

The principle behind the infrared spectroscopy is similar as in any absorption spectroscopy and uses the interaction between the radiation and matter. A ray of light with various photons of different energies is interacting with the matter. The photons of the energy E_x that match the energy of transition between non-agitated and agitated state get absorbed by the molecules and the energy is consumed to perform certain movements as discussed in previous sections.

Vibrational and rotational movement in molecules can consume the energy associated with infrared light at resonant frequencies. For example, the possible vibrations for the methylene group (–CH2–) involve: symmetric and antisymmetric stretching, scissoring, rocking, wagging and twisting, of which each absorbs a different amount of energy. In terms of analogy, if you imagine a couple of dancers, they require a different amount of energy to perform slow dance movements, e.g. waltz, than fast dance movements, e.g. jive, but both dances are possible for this couple. A couple without previous practice may perform less intensive dance when you provide them the energy, and the quality of the dance in our case would be analogous to the intensity of absorbance. If the couple has 30 other people attached to them as they dance, they can still do it, but the movement will be smaller with the same energy or requires more energy to perform the same move. If you then imagine a couple of kittens, no matter how much energy you are going to provide them, it is unlikely they would perform either waltz or jive. Similarly, for bonds in molecules, molecular bonds without the possibility to move/vibrate, will not absorb the energy.

Table 10. Overview of coefficients of determination between phase angles at the indicated test conditions, versus log (UV-vis absorption) at the indicated wavelength. Reprinted from Soenen & Redelius (2014).

10 Hz							
Wavel. (nm)	0 °C	10 °C	20 °C	30 °C	40 °C	50 °C	60 °C
340	0.51	0.59	0.59	0.49	0.32	0.17	0.12
380	0.62	0.73	0.80	0.82	0.66	0.48	0.40
410	0.57	0.65	0.76	0.86	0.78	0.73	0.60
450	0.46	0.53	0.64	0.81	0.83	0.84	0.82
490	0.34	0.39	0.51	0.69	0.77	0.82	0.83
510	0.25	0.28	0.39	0.56	0.69	0.78	0.81
0.021 Hz							
Wavel. (nm)	0 °C	10 °C	20 °C	30 °C[a]	40 °C[a]		
340	0.58	0.60	0.34	0.25	0.12		
380	0.78	0.77	0.67	0.54	0.41		
410	0.75	0.73	0.81	0.71	0.62		
450	0.63	0.61	0.81	0.86	0.85		
490	0.50	0.47	0.73	0.82	0.85		
510	0.38	0.34	0.65	0.76	0.82		

The highest R^2 are in italic
[a]At 30 and 40 °C, 0.021 Hz, sample B17 and B21 were excluded from the correlations.

The photons of different energies than the quants resonating with moves specific for specific molecule pass though the material, i.e. get transmitted. By measuring the energy levels in the ray of light passing through the material, one can determine which energy levels get transmitted. Using the reverse way of thinking, one can determine which of the photons got absorbed. For this reason, infrared spectra can be displayed either in Transmittance or Absorbance.

It is worth to note, that the inorganic materials may express the absorbance within the infrared light region as well, but those substances are recorder and reported typically in the Far IR region, i.e. frequency of 200–400 cm^{-1} or 25–50 µm wavelength. There are some modes of inorganic molecules that overlap to the organic spectra, for example in silica or calcium carbonate. This information is crucial when analysing the binders extracted from asphalt (with the risk of containing the impurity.

7.3.2 Application for the bitumen studies

Infrared spectroscopy is often used during the studies of pure molecules, to determine their structure and functional groups present. When one considers simple materials, such as water, ethanol, heptane or toluene, the whole studied liquid is having a concentration close to 100%, which we refer to as pure substance. Analysis of the spectra helps scientists to understand what the geometric structure of one molecule in such pure substance is by identification of allowed moves the molecule may perform.

However, the deduction of molecular structure of compound in a mixture, especially without knowing its concentration in the mixture, becomes a bit complicated. Infrared can be used in the studies of two-component mixtures, e.g. heptane:toluene mixed 50:50 or 30:70 by volume or anything in between, in which the contribution of each molecule to the spectra is related to the concentration of the molecule in the studied mixture (see Beer-Lambert law, IUPAC 1997 and section 7.3.3.2).

However, as explained before, bitumen is a mixture of thousands of molecules. Using infrared spectroscopy to determine the structure of all bitumen molecules separately is futile or extremely time consuming. Nevertheless, scientists managed to divide the bitumen into groups of molecules by eluting bitumen into different solvents. Such obtained groups of molecules share certain properties and functional groups. Scientists then compared their contribution to the spectra with some of the model basic molecules that contain similar functional group (J. C. Petersen 2001). The functional groups found in bitumen include: carboxylic acids (and independently their salts), anhydrides, ketones, 2-quinolone types, sulfoxides, pyrrolic types, and phenolic types (Figure 56).

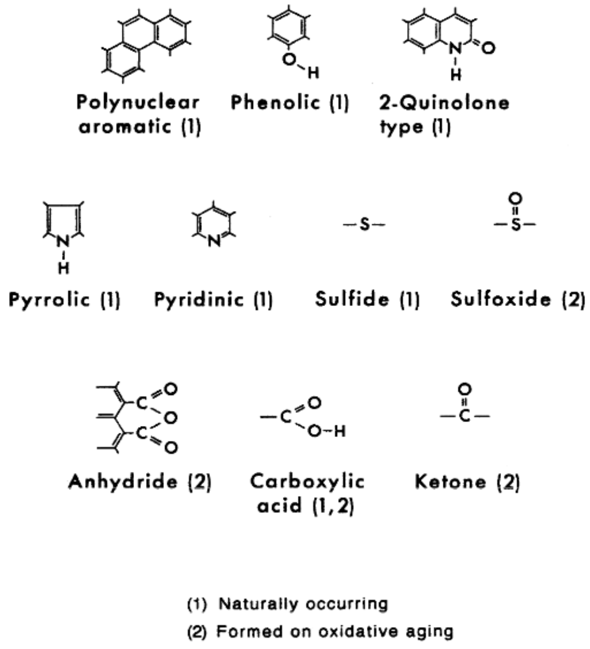


Figure 61. Chemical functionalities in asphalt molecules normally present or formed on oxidative aging (originally from J.C. Petersen 1984, after J. C. Petersen 2001).

Different areas in the spectra, as a result of analytical chemistry investigating the interaction with different solvents, were assigned to the different functional groups (J. C. Petersen 2001, see Figure 62 for the example assignment). Additionally, the groups more prone to adsorb on certain minerals, or groups related to increased moisture damage in asphalt were studied along the analytical process (Petersen, Barbour & Dorrence 1974) (Plancher, Dorrence & Petersen 1977). The effect of aging on the spectra was also studied and attempted to be correlated with changes in rheology of tested binders (Jemison et al. 1992; Jin et al. 2011; Liu et al. 1996).

In the beginning of the 90s, after introduction of the Superpave gradation system for the binders, the polymer modified binders became more common. It was also observed that the polymer modification is providing a signal in the spectra, and as such – the spectra can be used as a differentiator between modified and non-modified bitumen types (Yut & Zofka 2011). Further developments in research activities focused on linking rheology with infrared spectroscopy, in either forward (predicting ageing) or backward manner (evaluating age and origin of the pavement (Zofka, Chrysochoou et al. 2013), or fresh product differentiation using machine learning (Zofka & Błażejowski 2018).

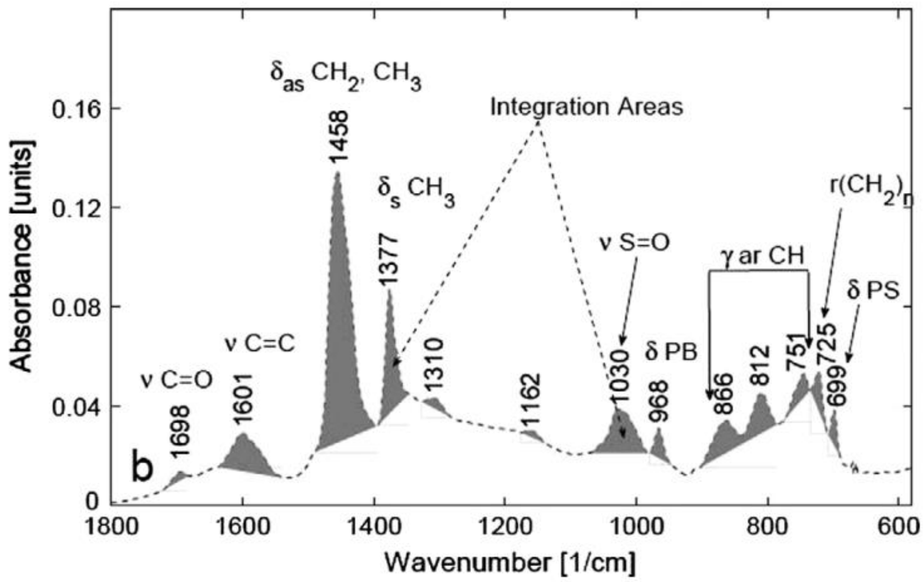


Figure 62. Typical spectra of oxidized bitumen modified with SBS polymer (Zofka, Maliszewska & Maliszewski et al. 2015).

The differences in aging patterns, i.e. the increase of the intensity in particular regions due to oxidation reaction changing the structure of molecules, were observed in the past (Yut, Bernier & Zofka 2012; Jemison et al. 1992). However, typically the studies found in literature investigate only one laboratory derived data and one or few binders. Interesting effort happened within the RILEM community were the Round Robin testing effort was conducted using the FTIR-ATR methodology on fresh and aged samples, and indeed it confirmed that the differences in aging pattern are perhaps more differentiating than the comparison of the original spectra of raw material (Hofko, Porot & Falchetto Cannone 2018), see Figure 63.

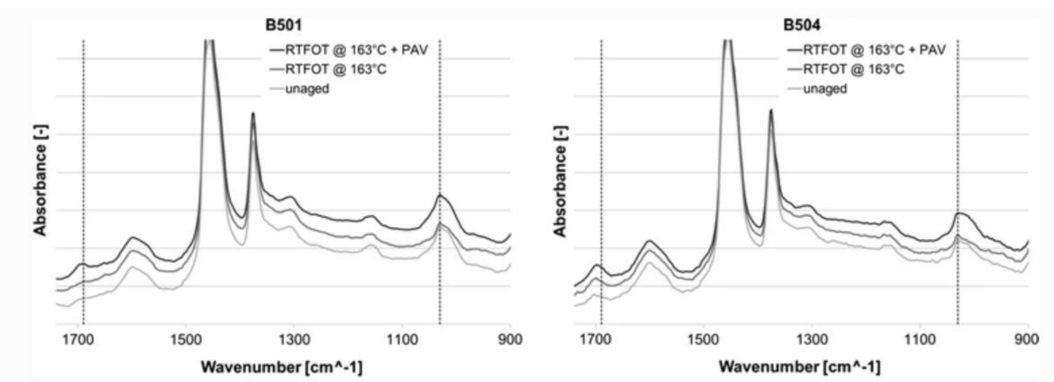


Figure 63. Example of the differences in aging patterns, followed using the infrared spectroscopy, between two different binders (Hofko, Porot & Falchetto Cannone 2018).

7.3.3 Infrared Spectroscopy Techniques

7.3.3.1 Transmission

Infrared spectroscopy can be executed in various ways (Figure 55). Originally the technique was developed in the form of transmission spectroscopy in which a liquid was placed between two non-IR-absorbing walls and a beam of IR light was passed through the system (Williams & Fleming 1966). Execution of transmission spectroscopy requires the skills in chemistry and background in safety with dangerous solvents, special ventilation cabinets, storage for chemical solvents, as well as special respiratory protection, because solvents may have effect on the health of the operator. For this reason, the uptake of the technique to the asphalt industry was on a low level.

Moreover, in respect of bitumen, preparation of transparent liquid requires significant dilution. The effect of dilution may cause the asphaltenes to precipitate in some cases. The different behavior of bitumen in various solvents and at various concentrations was actually at the core of determining the types of functional groups in bitumen. However, the original **transmission** spectroscopy is expected to not be highly applicable to the realities in asphalt laboratories.

However, the absorbance may be measured also via reflectance techniques. One of the most used in asphalt industry is Attenuated Total Reflectance (ATR) method in which a sample of bitumen (without solvent) can be placed on top of the prism (Zofka, Maliszewska & Maliszewski et al. 2015; Yut & Zofka 2011; Yut, Bernier & Zofka 2012; Hofko, Porot & Falchetto Cannone 2018; Karlsson R. 2003; Makowska, Hartikainen & Pellinen 2017). The focused beam of light is shined under specific angle through a specific medium of known reflectance, e.g. diamond or ZnS crystal (Karlsson & Isacsson 2011; Karlsson R. 2003). The part of the beam will be reflected, a part scattered under different angles, and part absorbed in material on the prism. When the background is collected without the sample on top, the machine determines the composition of the beam due to internal reflection (anything that is inflicted by machine itself). After the sample is placed on top of the prism, the comparison between background and sample spectra reveals what part of the beam was in fact absorbed by the sample. The result can be then either displayed as light reflected (Reflectance) or light absorbed (Absorbance). The Absorbance spectra without correction is not identical to that from Transmission type measurements. However, mathematical processing allows one to obtain the Absorbance spectra on par with the one obtained from the Transmission experiments.

7.3.3.2 ATR qualitative or quantitative?

The basis behind use of FTIR as a tool for quantitative analysis is grounded in the Beer-Lamber law, which according to IUPAC (1997) states that the absorbance of a beam of collimated monochromatic radiation in a homogenous isotropic medium is proportional to the absorption path length (l [cm]) and to the concentration (c [mol/dm^3]). The law is given by equation:

$$A = \kappa c l \tag{16}$$

where

κ is molar (decadic) absorption coefficient [dm^3/(mol*cm)].

To understand the equation through analogy to coffee used previously:

- If we fill the same cup fully with coffee of different strengths, the color of the coffee we see will differ, as absorption of light by the coffee depends on the concentration of coffee essence in total liquid (Figure 64).
- If we fill the cup fully, to half and just on the bottom, the color of the coffee is different, despite the same concentration – because the more coffee in the cup, the more light is absorbed by the coffee particles (Figure 65).
- If we fill the cup with coffee, milk or tea, the color is different, because each of the substances has its own molar coefficient specific to the chemical structure of the liquid studied even if all are dissolved in water as a solvent.

Figure 64. An example of how concentration of chemical in the solvent affects to the absorbance of light, pictured are tea concentrates at different levels of dilution with water from weakest to strongest (left to right) in which the thickness of the liquid is kept constant.

Figure 65. An example of how thickness of solution affects absorbance of light, pictured are tea concentrates of equal strength, where from left to right approximately 5 mm, 20 mm and 40 mm thick film is poured.

Therefore, if the blend is homogenous and measurement is performed in Transmission mode, with increasing concentration of the modifier dispersed in medium, the absorbance in the system should increase, if spectra is measured from a known thickness of sample. Using this relationship, a calibration line can be developed as presented in Figure 66. Such calibration line should assure if there is linearity within the range of interest and measure minimum and maximum expected concentrations in studied materials. At the very high and very low concentrations the linearity may be skewed by the relation to the background and thus extrapolation from the calibration line is not advised.

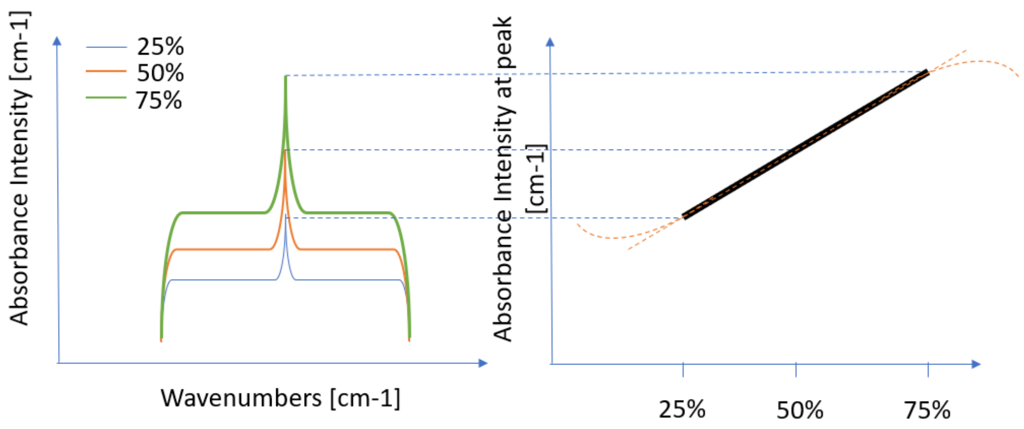


Figure 66. Example of the process of determining calibration line for component concentration in the mixture, using a characteristic peak's intensity difference, where on left there is a schematic spectra of 3 mixtures with varying concentrations of compound, and on the right the maximum absorbance intensity is correlated to the concentration of compound.

However, the bitumen is a non-transparent material. The Attenuated Total Reflectance (ATR) tool is preferred for measurements of it to avoid solvents, and laboratory related issues, as well for simplicity and robustness of measurement. But the method comes with its own limitations. But when systems in which modifiers are added into the bitumen, their relative concentration can still be evaluated using the Beer-Lambert principle, as demonstrated in the past (Karlsson R. 2003; Makowska & Pellinen 2016).

The ray of light in ATR set-up penetrates **only to a certain depth** in the material and the signal reflected is recorded (Harrick 1967). The resulting spectra are an average of the sample properties for the certain distance between the prism and a fixed depth in the material tested. However, a different wavelength (or energy of photon) can travel to different depths before reflecting happens.

Additionally, other aspects affect the depth to which the radiation penetrates (d_p) and that is defined by equation:

$$d_p = \frac{\lambda}{2\pi(\sin^2\theta - {n_{21}}^2)^{1/2}} \quad (17)$$

where

λ is the wavelength of incident light
θ is the angle of incidence of light
n_{21} is the refractive index ratio between sample and the prism (Harrick 1967).
In the ATR measurement, the angle is kept constant, thus is not a variable. Other parameters in the equation do affect the result.

Unfortunately, the change in refractive index (n_{21}) with changing concentration of polymer and other components, may affect to the Absorbance values as it may affect to the d_p. For example SBS is reported to have refractive index of 1.53 (Scientific Polymer Products, Inc 2018), while bitumen is estimated to have a refractive index between 1–1.5, depending on the density (Chamkalani 2012). Therefore, adding the polymer may affect the molar coefficient in the equation 16 and refractive index in equation 17, due to the measured material being of different composition. In such case the wave of light may go deeper or shallower into the material.

Why is this important? See Figure 67 in which a schematic spectra collected from three mixtures is taken, but with the assumption of refractive index changing in mixture with extremely high concentration of modifier, leading to the lower wave penetration into the material. As a result, a thinner film of the mixture would be measured, and as we saw with thin film of the tea in a cup (Figure 65), the whole spectra's intensity would be lower, not only that of the characteristic peak that one would follow for the purpose of the calibration line determination (Figure 66).

To be scientifically correct when applying the Lambert-Beer law to ATR, the diffraction index should be determined for each mixture tested to determine how deep into the sample the ray penetrates before refraction occurs. This is the correct approach to account for the path length variable in the equation 16, which in this case is defined as d_p. The d_p will affect to the intensity of the absorbance recorded and spectra needs to be normalized. However, this approach is very laborious.

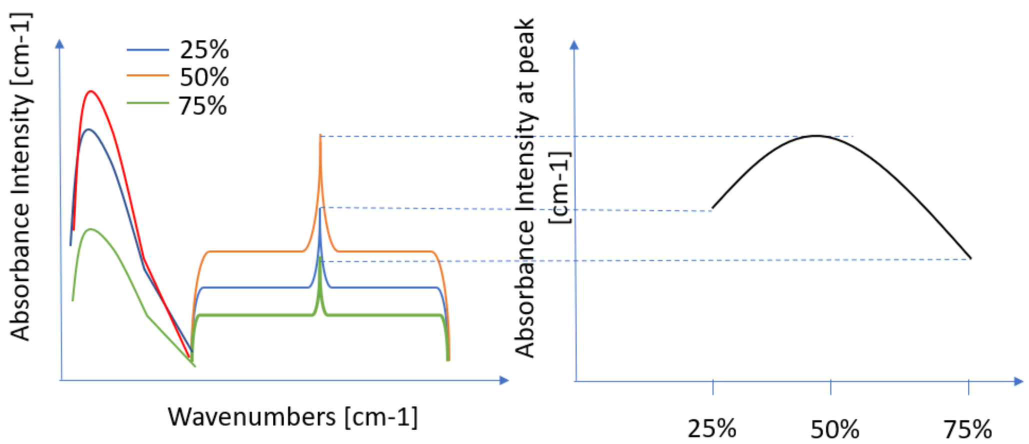


Figure 67. Schematic demonstration on how the change in diffraction index affects to the calibration line, depth of penetration being lowered at high concentration causes total absorbance being lower, not only the characteristic peak.

Additionally, if the material is not homogenous and precipitation of some particles through dispersing medium are changing with time (e.g. gravitational segregation of polymer in bitumen), then the repeatability of measurement may be low even from one sample because both the concentration and path length may be changing during the measurements.

For this reason, analysis of complex mixtures in which relationship between 2–3 characteristic peaks need to be deconvoluted (e.g. increase of carbonyl due to aging, increase of polymer due to segregation) a more sophisticated analysis is required. **For this purpose, the machine learning solutions are expected to provide a quick analytical tool.**

7.3.3.3 Diffuse IR spectroscopy

In case of diffuse reflectance infrared Fourier-Transform spectroscopy (DRIFTS), the sample is powdered into the fine particles, smaller than the size of the incident wavelength (good if below 5µm) and blended at about 10% by weight with potassium bromide powder (which is transparent for IR). However, the sample is not necessarily compacted as can be the case in pill preparation for transmittance studies of solids.

As shown on Figure 68 the infrared light beam is illuminated on the surface of such powder under certain angle. There always occurs specular reflection (mirror like reflection), which in this case is not of particular interest. The light beam reflects from some particles and travels internally to the depth of the material. During the path from particle to particle some wavelengths get absorbed by the sample and some become reflected. After the reflected beams travel back to the surface, the angle under which they emanate from the sample is random (Figure 68), depends on the granularity and reflectance parameters of the sample. All the beams after reflection are gathered using a collimated window above the sample, which reflects them to the detector.

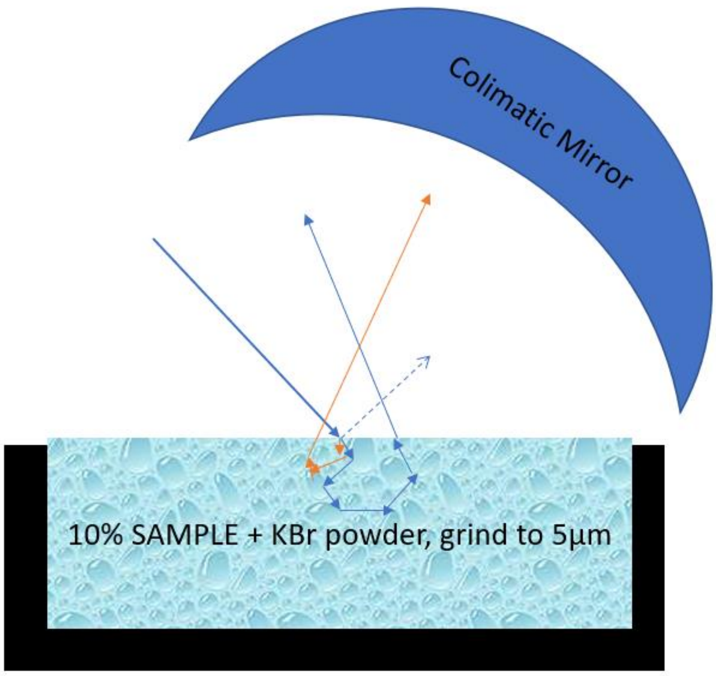


Figure 68. Schematic of principle behind the Diffuse Reflectance Infrared Spectroscopy measurements, with incident light coming from one side, the specular reflection under the same angle being ignored, and the ranged of diffused beams being concentrated via colimatic window before transmitting it to the receiver.

Because the measurement is performed from the top and can be placed on top of the studied sample, it automatically poses an interesting solution for IR studies of asphalt samples in the road conditions. The diffuse spectroscopy was investigated in SHRP2 program (Zofka, Chrysochoou et al. 2013; Chrysochoou, Johnston & Yut 2014; Salomon & Yut 2016). The aim of the study was to evaluate what is the possibility to use infrared in field.

Sample pavements were drilled with a regular drill to powder the asphalt material into the fine powder. Such obtained powder was gently pre-compacted but not over-compacted in the prepared containers. Additionally, a series of tests was performed on "in-place" pavement without drilling, just from the surface as is. The method is simple and requires 1 h of training for the operator. The equipment is portable. (Zofka, Chrysochoou et al. 2013; Chrysochoou, Johnston & Yut 2014; Salomon & Yut 2016).

The best findings were obtained for studies of RAP content in fresh mixtures (Chrysochoou, Johnston & Yut 2014; Salomon & Yut 2016). Based on the calibration data of RAP and fresh mixtures, a rather reliable estimation of the RAP content was demonstrated (Salomon & Yut 2016). However, a suggestion was to include more roads into the future studies. The content of the polymer in the bitumen and the composite could be established qualitatively with some reasonable doubt (present or not), but due to the very low concentration of the polymer in the whole composite, the qualitative evaluation is rather not plausible from the asphalt concrete (Zofka, Chrysochoou et al. 2013; Salomon & Yut 2016).

However, as can be seen from Figure 69 and Figure 70, the spectra obtained by means of ATR and DRIFTS expresses characteristic absorbance within similar ranges, but the shapes of the peaks and the clarity of signal is on a diametrically different level. In the case of going towards the DRIFTS type analysis, a separate procedure for data analysis and improvement of the signals would be advised still. There is a work ongoing (Salomon & Yut 2016) on that subject but no easily available published data was found during the preparation of hereby report.

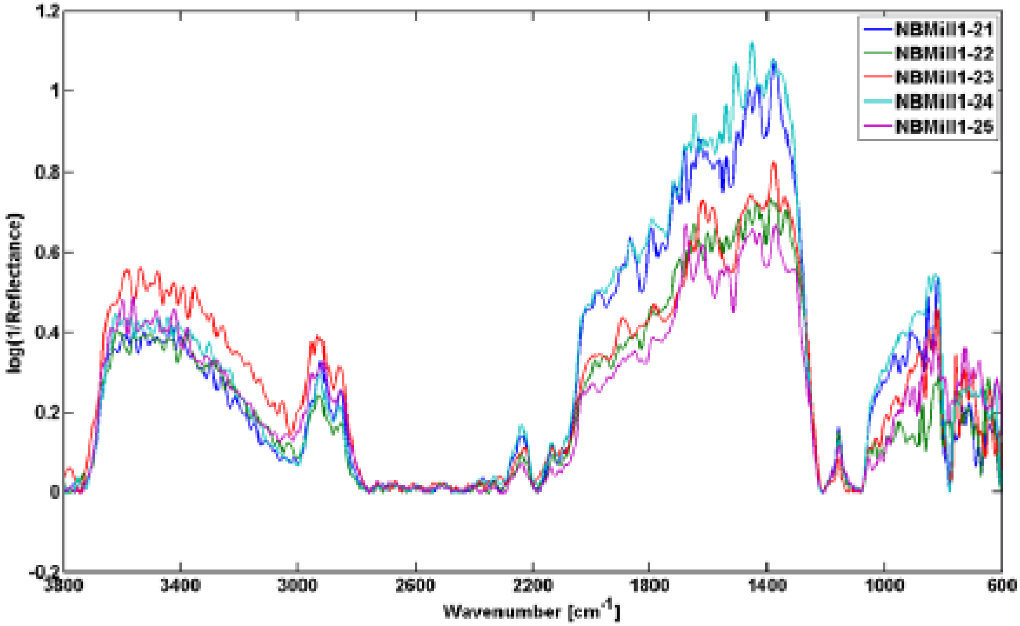


Figure 69. The example of the DRIFTS spectra of milled RAP after 1–2 years in storage from New Britain, Connecticut, USA (Chrysochoou, Johnston & Yut 2014).

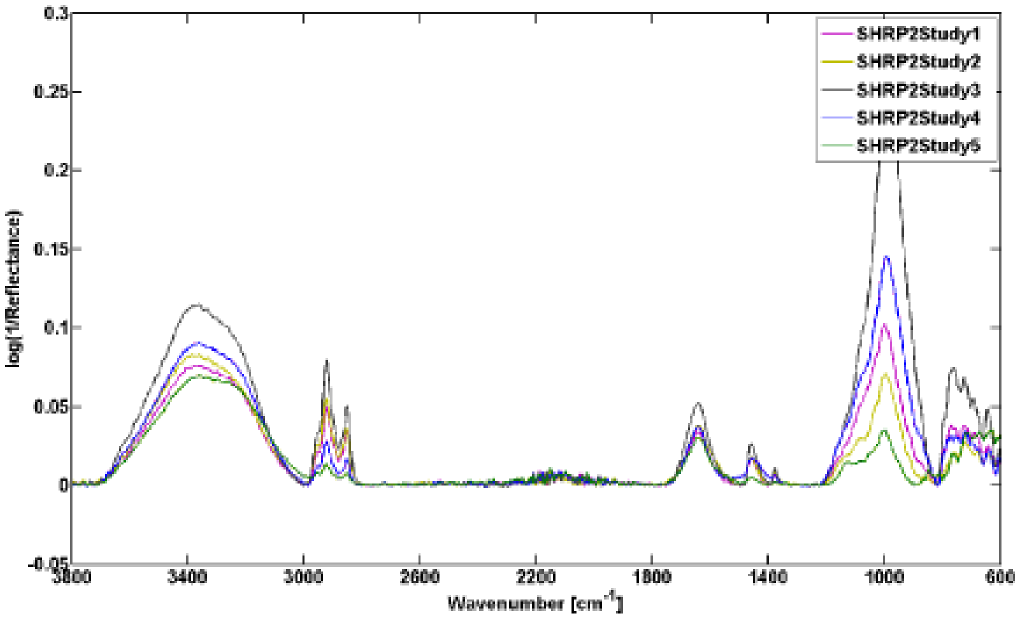


Figure 70. The ATR spectra of milled Class I RAP from Poland, Maine, USA (Chrysochoou, Johnston & Yut 2014).

The SHRP2 reports provide the manuals for the operators who desire to uptake the method into use (Zofka, Chrysochoou et al. 2013; Chrysochoou, Johnston & Yut 2014). The principle based on the RAP from piles included pre-compaction of the samples or sieving the RAP to obtain the very fine material.

This application is a solution that could be easily adapted in every plant and by every contractor. The methodology still requires some calibration to plant's own parameters. However, the success and homogeneity of dispersion of RAP in the structure could be evaluated much more easily than currently and non-destructive control of recycling process could be taken to a new level.

The phase IV of the SHRP2 report indicated that the following of the RAP oxidation in piles is plausible, as well as the following of the oxidation of the pavements in field. Perhaps using the information about the level of oxidation of the pavement connected with performance and rheological parameters could be build into the digitalized database allowing us in the future to evaluate the state of the road in a much more rapid way.

It is postulated by author of hereby report based on review of literature related to the technique (Carmona-Quiroga et al. 2009), that the suggested path to improve results would be to investigate if including the dilute KBr at higher concentrations and trying to reduce the grain size of the samples during measurement would improve the resolution.

7.3.4 The chemical composition and rheology influences resistance to aging

Quite early on, it was observed that the viscosity affects the resistance to aging (review in J. C. Petersen 2001). When one compares the values of retained penetration, even in the Finnish Asphalt Specifications (Finnish Pavement Technology Advisory Council 2017), it becomes apparent that the softer binders are allowed to stiffen by relatively more than the stiffer binders. Of course, the comparison is done by logarithmically displayed viscosity in form of Penetration, so comparison of stiffening power by percentages is a complex issue.

Nevertheless, it was observed that softer grades of binders oxidize faster than the stiffer grades (Yut & Zofka 2011). The effect is most likely due to the diffusion of oxygen into the binder. As is known from Stokes equation, the diffusion of one component through the medium depends on the viscosity of medium (after M. Makowska 2017). Figure 71 demonstrates how different origins of the binder influence the rheological changes with time, but also how the aging process affects the change in a chemical manner (J. C. Petersen 2001). The ketone content or "-CO-" peak is therefore not the only thing which can be affecting the rheology and performance.

Using FTIR, one can determine between binders with abundance of aromatic and abundance of aliphatic components, by comparison of peak ratios in corresponding regions. Why does it matter? Long chain aliphatic components are also referred to as **waxes**. Their influence on rheology is well-known, and results in stiffer, more solid like behavior at lower temperatures and lower viscosity at higher temperatures.

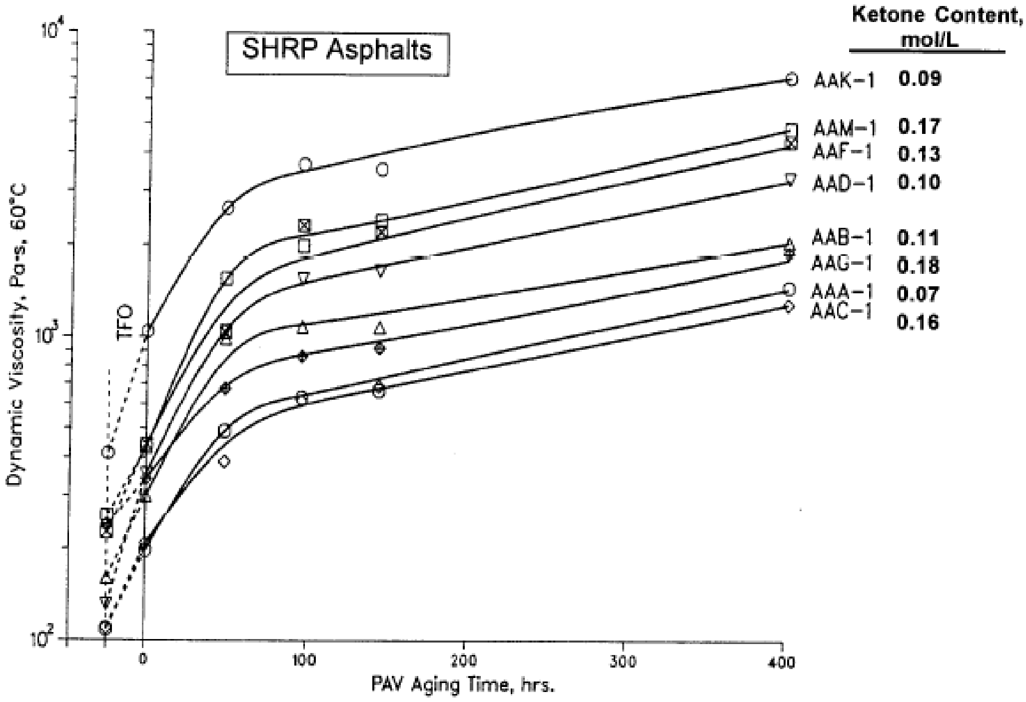


Figure 71. Age hardening kinetics of eight core SHRP binders studied in oxidative PAV at 60 °C (J. C. Petersen 2001).

As is schematically presented in Figure 72, three bitumen types of which as witnessed by typical rheological markers like Penetration, Softening Point and Dynamic Viscosity at 60 °C, could be for example classified as 70/100 (green), 50/70 (blue) and 20/30 (orange). However, above the melting point of the solid waxes, which can be above 60 °C, the waxes turn into liquid paraffin – a liquid of very low viscosity. Such liquid then acts as a softener and as a result at higher temperatures the viscosity of highly waxy 20/30 bitumen can be lower than that of 70/100. The thing is, that the majority of thermal oxidation occurs during the short-term aging, or during asphalt mixture preparation (Whiteoak 1990). If more oxygen can diffuse into the binder, the aging pattern can be different.

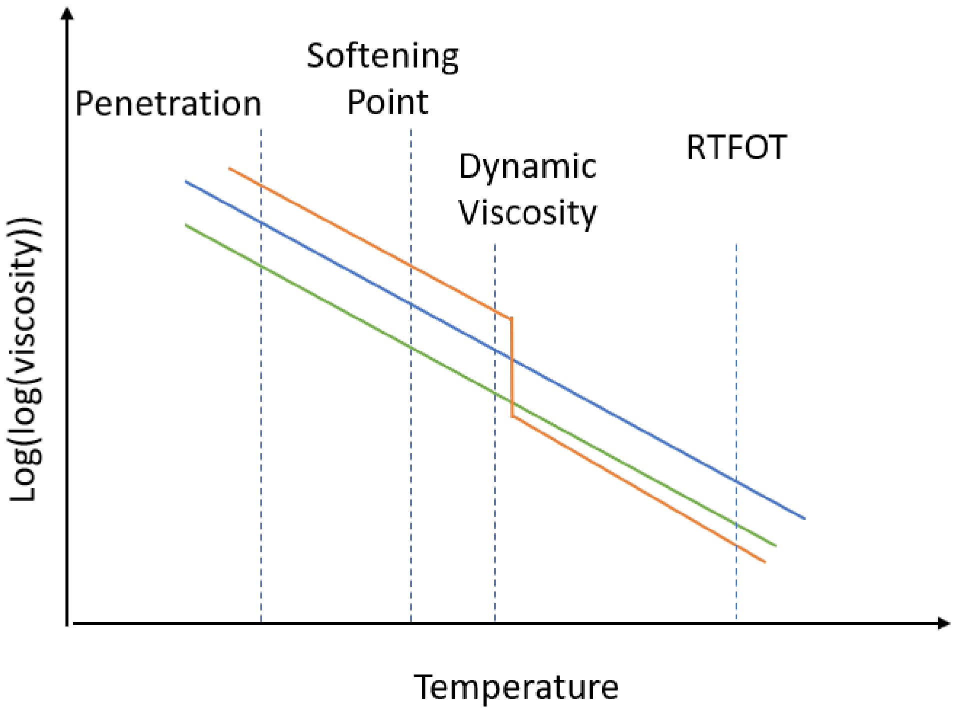


Figure 72. Schematic representation of differences in rheological behavior between waxy (orange line) and non-waxy bitumen types (blue and green line).

It would be of great value to be able to recognize the waxy bitumen type in a fast manner before production. Waxes are typically used as additives to Hot Mix Asphalt mixtures in order to lower the production temperature and produce asphalt in Warm Mix Asphalt manner. Such approach allows reduction of carbon dioxide (CO_2) emissions connected with production in plant. However, as has been witnessed by Hofko, Porot, Falchetto Cannone et al. (2018), producing the asphalt with the same bitumen at lower temperature could also lead to lower stiffening in short term aging. Perhaps exposing the binders to lower temperatures could extend the lifecycle of the pavement on top of the clear reduction in emissions and costs, if we were able to identify which binders are naturally good for WMA.

7.3.5 Analysis of bitumen during aging The ATRAC system

The studies analyzing the aging of bitumen typically focus on the analysis of the binder before and after aging, while aging is conducted outside of the equipment used for actual characterization. Yut et al. (2012) proposed that a sample of bitumen could be aged during the observation by FTIR-ATR. Their solution, called ATRAC (ATR based air-flow conditioned sample) is presented in a schematic picture presented in the Figure 73.

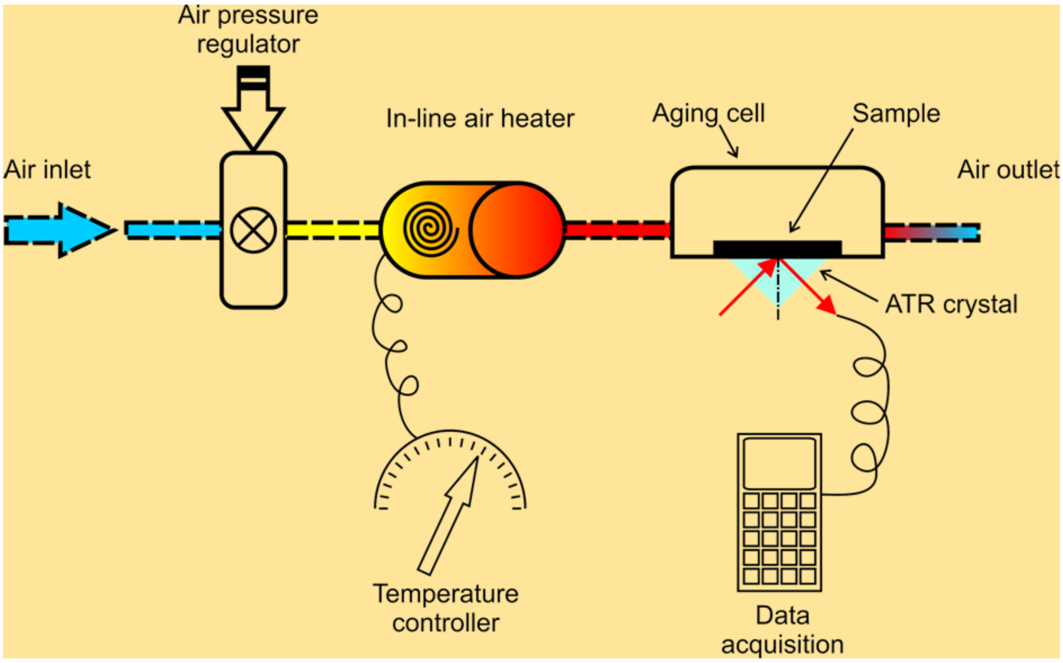


Figure 73. Schematic representation of the ATRAC set-up (Yut, Bernier & Zofka 2012).

Figure 74. The picture of the setup used for in-situ aging by the method called ATRAC (Yut, Bernier & Zofka 2012).

In the above system, the bitumen sample is placed on top of the ATR prism. The chamber with inlet and outlet for air is placed on top of the ATR system. The air supplied to the chamber is heated prior to the entrance to the chamber and contact with the sample of bitumen. This type of observation by infrared during aging will be referred further on as in-situ. The sample is not removed from the equipment and the risk of losing the information due to loss of material during transfer or on the walls of equipment (as in the case of RTFO or PAV) is therefore minimal.

The spectra was collected at certain intervals. The reaction rate (called as oxidation rate, rCA) is determined from change in carbonyl area (dCA) during the measurement by using the area under the carbonyl peak (CA) (as defined in Figure 62), and its change with time (dt), given by the equation:

$$rCA = dCA/dt \qquad (18)$$

Yut et al. observed that the temperature and pressure of the air coming through the inlet affects the speed of aging (Yut & Zofka 2011). The more pressure and the more flow of air, led to lower temperature of air, and thus slower aging.

Therefore, the air flow and its temperature should be considered during the studies using such systems.

Makowska et al. (2017) studied aging in-situ as well using a different system. The ATR equipment in their set-up has the possibility to raise the temperature of the prism to 200 °C. The study was conducted at 163 °C to investigate the changes possibly observed during the RTFOT type aging. Their system was opened to laboratory air, tough.

The authors proposed to combine heated stage and chamber with air flow in further studies to better control the temperature, air flow, air purity and to allow for analysis of the gases outflowing from the chamber.

Makowska et al. observed that in fact the changes in the spectra can be pinpointed in stages, suggesting multiple types of reactions occurring one after another, which expands on the work of Yut et al. However, it was Yut et al. who tested multiple binder grades and sources in terms of recognition between them by using this type of technique first.

Some of the conclusions were that:

- The softer binder grades oxidize faster – the stiffer the binder the slower the aging rate
- New England bitumens and SHRP core bitumens expressed a different rate of oxidation

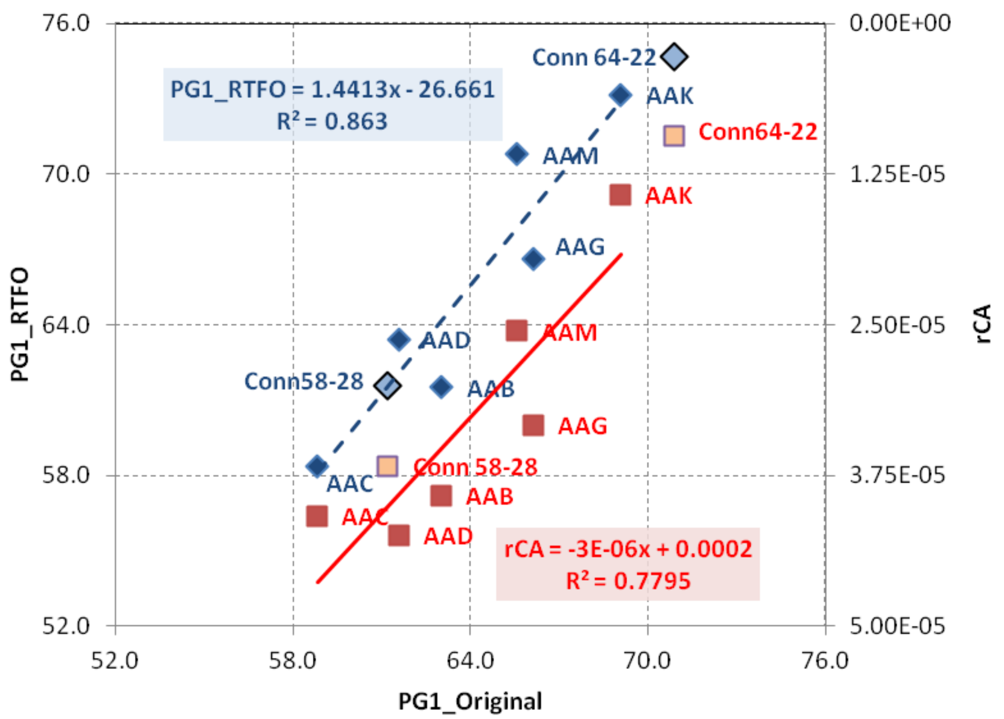


Figure 75. The comparison of oxidation rates for various binder types and grades used in the study by Yut et al. (Yut & Zofka 2011).

Based on those findings it is expected that the pattern in which the spectrum of bitumen is changing during aging can be a distinguishing parameter between crude oils or producers.

Makowska et al. observed as well that the presence of impurities in the binder, or catalysts may interfere with how the reaction proceeds. Analysis of the spectra in time resolved manner allows for following of the reaction path and determining which of the binder components may be affecting the most to the progress of oxidation (the sulphur compound oxidation, vapourization or formation of ketones) (Makowska, Hartikainen & Pellinen 2017).

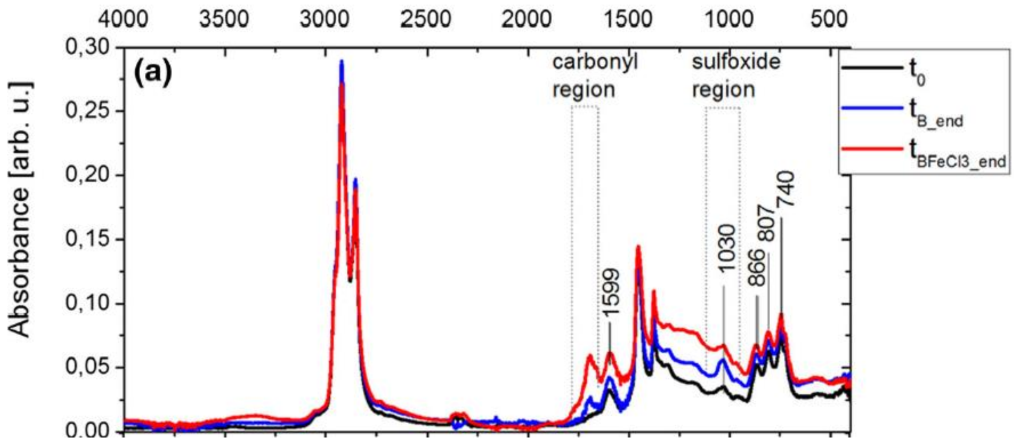


Figure 76. The example of the spectra of bitumen observed at 163 °C with (BFeCl3_end) and without (B_end) catalytic particles after 52 minutes of oxidation (Makowska, Hartikainen & Pellinen 2017).

7.3.6 Polymer type

Infrared spectroscopy conducted by means of ATR is a good qualitative tool to identify materials in the bituminous mixtures with non-overlapping signals. In Figure 77 a combined graph presents spectra of bitumen, SBS polymer, limestone filler and a mastic. It is visible that certain material specific peaks can be identified in raw materials. Looking at the mixture of those raw materials in a mastic like product, an assignment of peaks to the respective raw materials, in the spectra of the composite is possible. Similar procedure to identify components in inorganic filler was presented by Makowska et al. (2014).

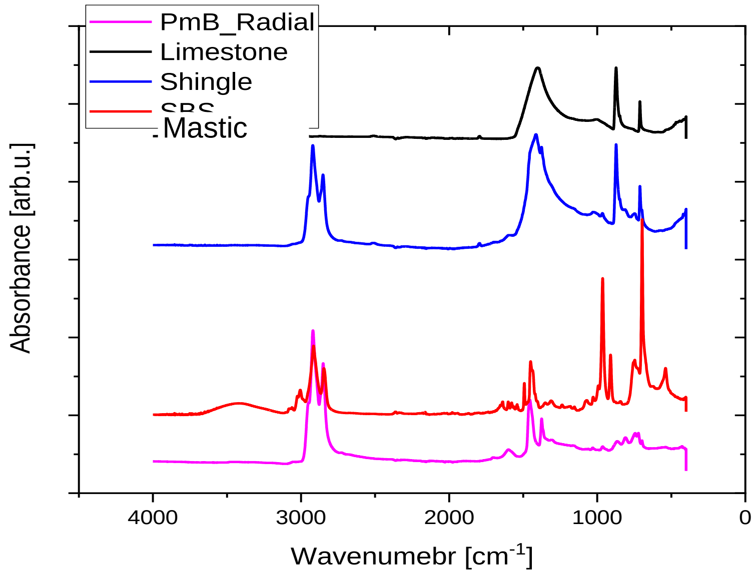


Figure 77. Demonstration of applicability of FTIR spectra in qualitative analysis of products (own data).

To the starting asphalt producer, it may seem that the polymer in bitumen is always the same type of polymer. However, a polymer additive type or origin or even simply product variation may influence the properties of final PmB but also the chemical composition of the final PmB (Polacco et al. 2015) and therefore its FTIR spectra (see Figure 78 Figure 79).

The most typically used polymer in the asphalt industry is so called SBS (styrene-butadiene-styrene block copolymer) (Polacco et al. 2015). This polymer is composed of two polymer chains that link with each other – polybutadiene (PB) and polystyrene (PS). It is therefore very important to underline that polymer products produced by one company may have a varying ratio of block copolymers e.g. 50:50 PS to PB or 70:30 PS:PB, but also how the polymers link with each other may determine the change in their properties – e.g. linear (like pearl necklace with different sizes of pearls) or radial (structure similar to colorful pom-pom on the winter hat). The fact of a range of polymer products on the market even within the name of SBS is a good thing, because there is a range of bitumen products on the market as well. To find a polymer compatible with the specific bitumen used as base bitumen, sometimes multiple types of additives need to be tested to achieve most stable and best performing PmB.

The signal in the polymer spectra will be in fact different if the PB:PS ratios are different in the material, because of the Lambert-Beer law and the fact that absorbance is related to concentration (equation 16). But the spectra may also be different because of different refractive index of the polymer, as discussed previously (equation 16 and 17). Recognizing the products qualitatively that use different polymers in the preparation of PmBs should be therefore achievable (Zofka, Chrysochoou et al. 2013), but drawing the quantitative conclusions is more challenging (Zofka & Błażejowski 2018).

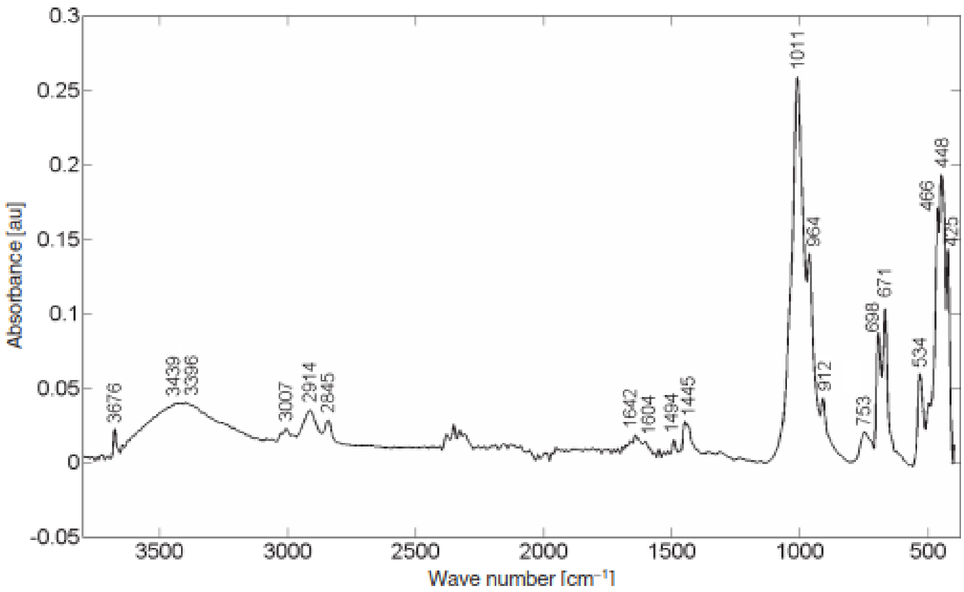


Figure 78. Baseline-corrected ATR spectra of SBS by Kraton D1101 (Zofka, Chrysochoou et al. 2013).

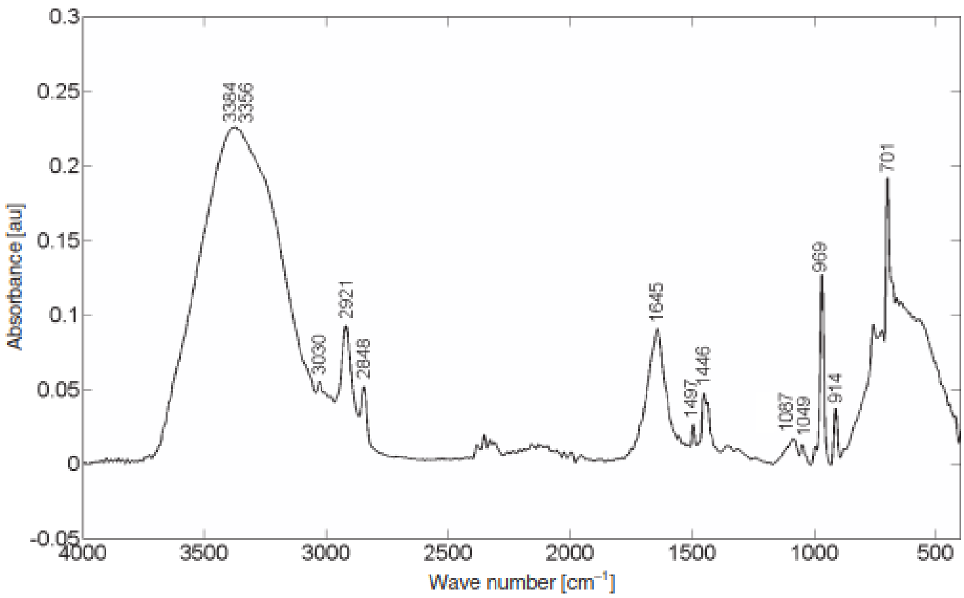


Figure 79. Baseline-corrected ATR spectra of BASF's butonal SB latex polymer (Zofka, Chrysochoou et al. 2013).

Few research efforts focused on the ability to predict the type of the bitumen and the type and concentration of modification within it. The success with distinguishing between the products was satisfactory, but the ability to state beyond the reasonable doubt what is the actual concentration of the modifier may be challenging based on the limited training data sets for the developed models. Below are reprinted spectra of investigated materials to demonstrate the differences in spectra of PmBs before and after aging procedures.

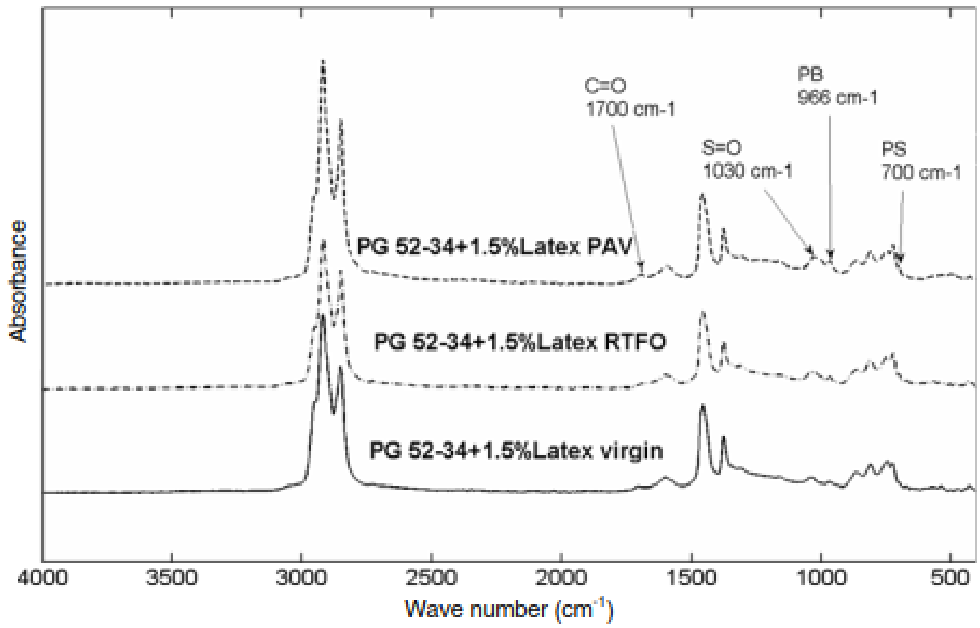


Figure 80. Comparison of the ATR spectra for virgin, RTFO-aged, and PAV-aged PG52-34 binder modified with 1.5%wt. SBR latex.

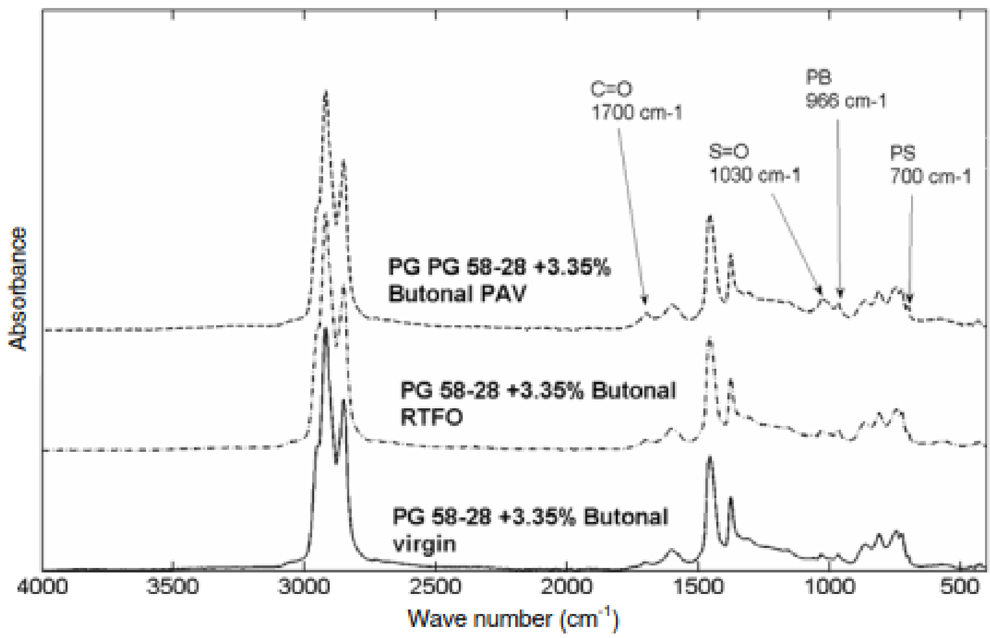


Figure 81. Comparison of the ATR spectra for virgin, RTFO-aged, and PAV-aged PG58-28 binder modified with 3.3% wt. BASF Butonal.

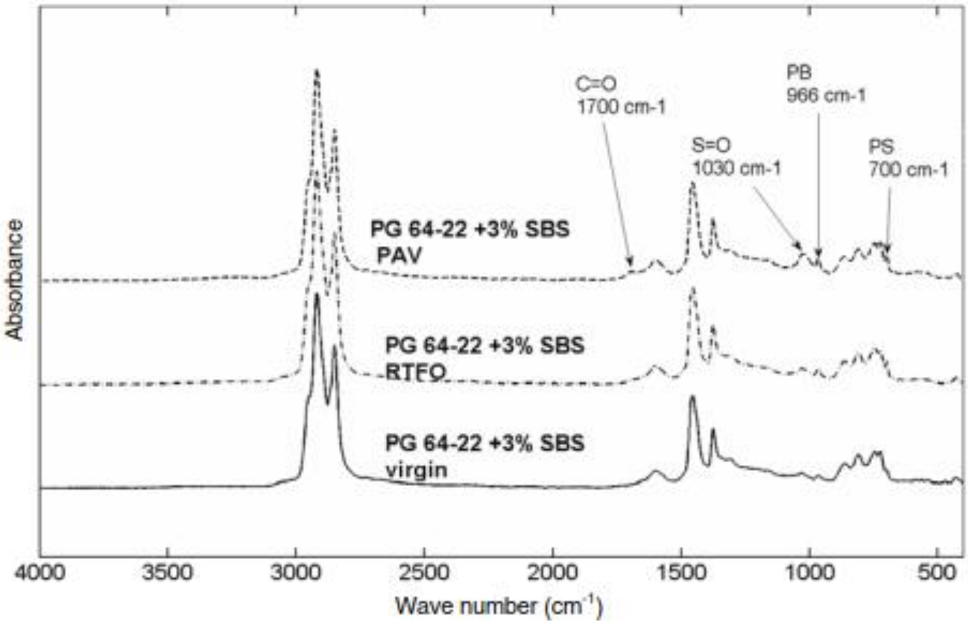


Figure 82. Comparison of the ATR spectra for virgin, RTFO-aged, and PAV-aged PG64-22 binder modified with 3% wt. Kraton SBS.

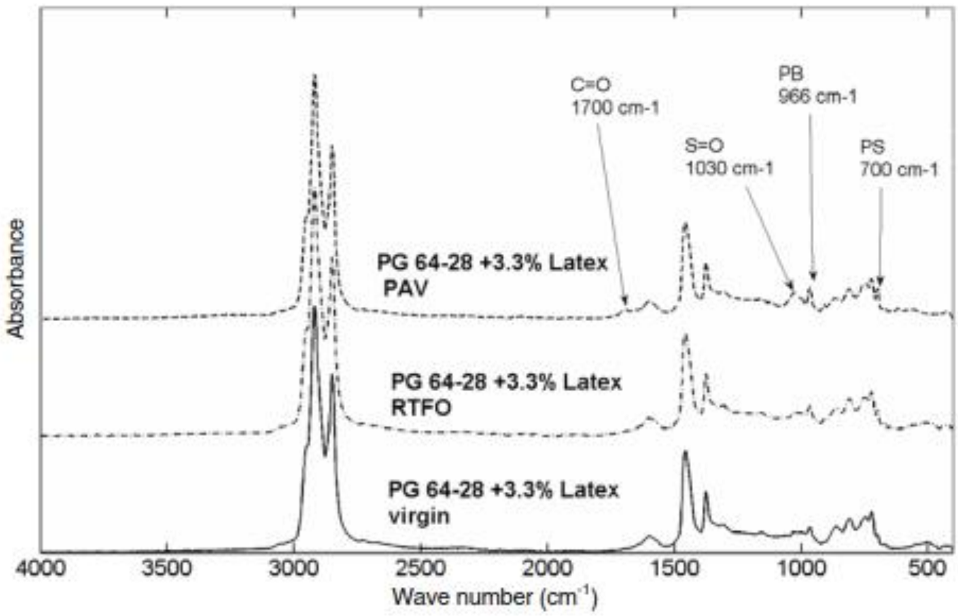


Figure 83. Comparison of the ATR spectra for virgin, RTFO-aged, and PAV-aged PG64-28 binder modified with 3.3% wt. SBR latex.

7.3.7 Polymer content

Due to the fact, that polymers used as modifiers are having a spectra differing from that of a base bitumen (Masson J-F, Pelletier & Collins 2001), it is easy to distinguish their presence in the Polymer Modified Bitumen blend using Fourier Transform infrared spectroscope with Attenuated Total Reflectance mode (see Figure 84) (Yut & Zofka 2011; Zofka, Maliszewska, Maliszewski et al. 2015). It was proposed (Zofka, Maliszewska, Maliszewski et al. 2015) that the content of bitumen could be established from calibrated data (see Figure 85). As can be seen on the reprinted figure, and as explained in previous sections, with increasing content of polymer, the polymer related peak intensity increases, but the bitumen peak intensity decreases for 2% but increases again at 4%. This is possibly due to the change in reflectance discussed in previous sections. Comparing the area under the peaks leads in such case to non-linear calibration line and inability to apply the Lambert-Beer law as visible in Figure 85.

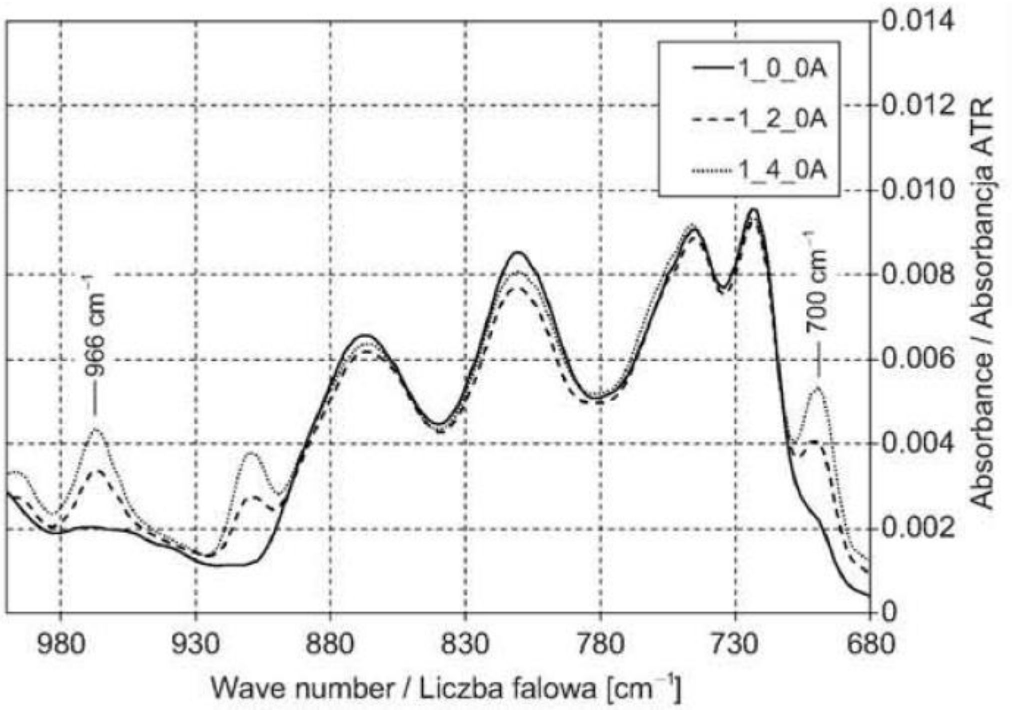


Figure 84. Spectra of base bitumen with 0%, 2% and 4% polymer content. Reprinted from Zofka, Maliszewska, Maliszewski et al. (2015).

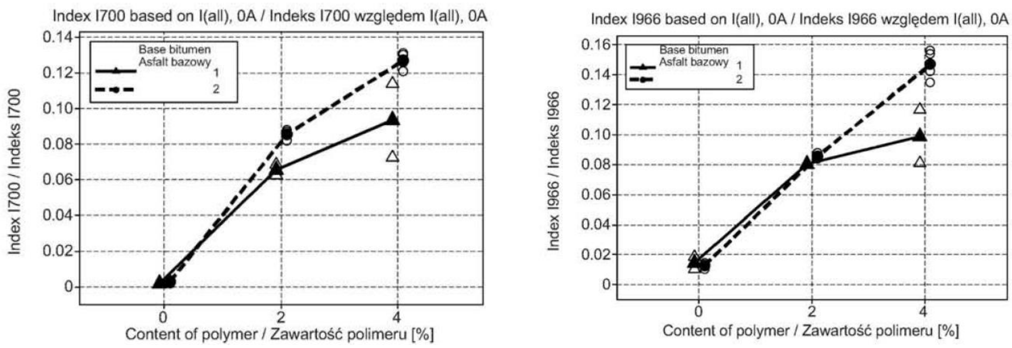


Figure 85. The relationship between I700 (polystyrene, left frame) and I966 (polybutadiene, right frame) and the content of polymer. Reprinted from Zofka, Maliszewska, Maliszewski et al. (2015).

Just recently the same group published similar study on Highly modified bitumen (6–7%) and in their conclusions they stated that the signal processed in the way described in the article is inconsistent and cannot be used to determine the content of polymer in bitumen at those concentrations (Zofka & Błażejowski 2018). The most recent conclusion by authors is to use the ATR method more to determine if the polymer is present or not than to use it for polymer content determination.

Nevertheless, they developed artificial network-based system that can be trained to determine which market product is tested. As discussed in section 7.3.3.2 and is visible in Figure 85 and Figure 86 – the effect of diffraction index may skew the intensity of absorbance and in the effect the calibration line. Despite the fact that the exact prediction on the level of 1–2% is probably not within significant doubt, the methodology can be applied to the distinguishing between products such as non-modified binder (NM), normal level polymer modified binder (up to 4%) (PM) and highly modified binders (7–8%) (HM) (Zofka & Błażejowski 2018). Therefore, a more binary approach – "no" or "yes", very much is the result that can be expected when applying the method to the quality control and material disputes.

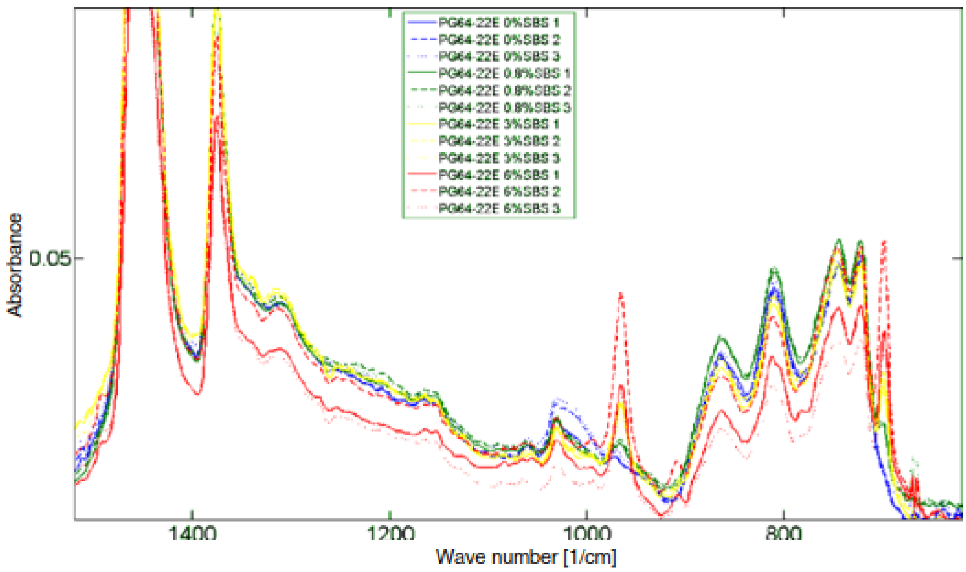


Figure 86. Comparison of the ATR FTIR spectra for PG 64-22 binders modified with 1, 3 and 6% wt. Kraton SBS (Zofka, Chrysochoou et al. 2013).

7.3.7.1 Applying Artificial Neural Network to PmB recognition

The materials used for the development of the Artificial Neural Network (ANN) (Zofka and Błażejowski, Machine Learning Technique for Interpretation of Infrared Spectra Measured on Polymer Modified Binders 2018) which allows classification of the binders into those three classes – NM, PM and HM, is presented in the Table 11, while an example spectra of those materials is presented in Figure 87.

Table 11. Binder Penetration classes and groups for development of the ANN (Zofka & Błażejowski 2018).

Penetration classes	Producer	Group
20/30 35/50 50/70 70/100	**A** **B**	**NM** (unmodified)
PMB 10/40-65 PMB 25/55-60 PMB 45/80-55 PMB 45/80-65	**A** **B**	**PM** (modified)
PMB 25/55-80 PMB 45/80-80 PMB 65/105-80	**A** **B**	**HM** (highly modified)

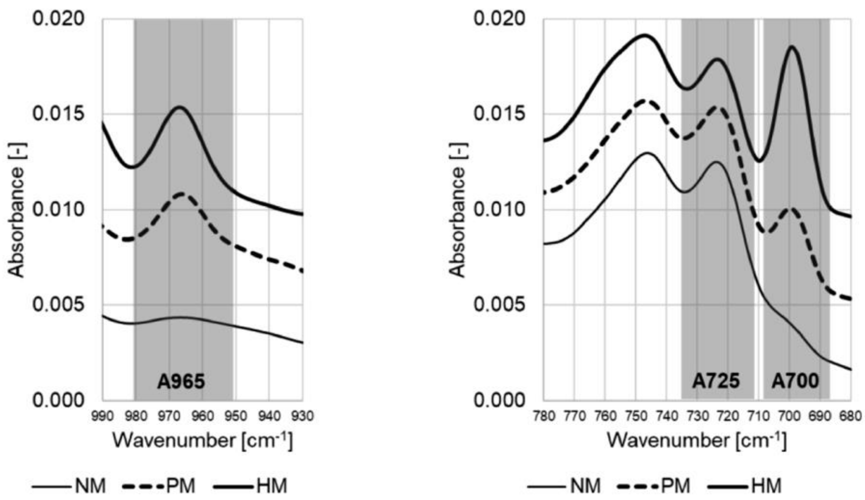


Figure 87. The response in FTIR-ATR from non-modified (NM), polymer modified (PM) and High polymer modified binders (HM) within different polymer characteristic regions after (Zofka & Błażejowski 2018).

The ANN for this problem was developed using 22 binders from two producers. For the database development 5 replicates of each binder were tested. Then the spectra were divided into 70% data for model development, 15% for validation and 15% for testing. In the testing phase it was demonstrated that the ANN developed was able to successfully distinguish between the samples and to categorize them.

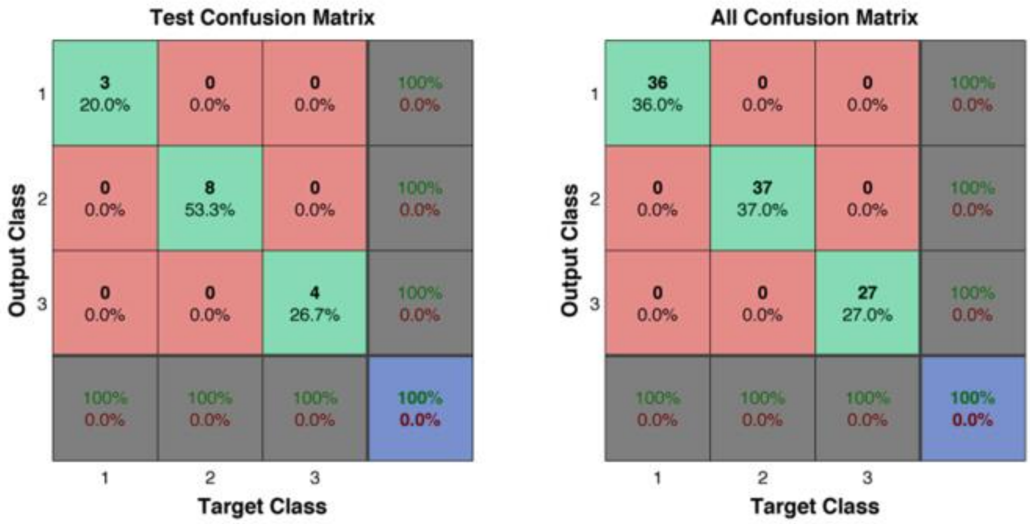


Figure 88. ANN confusion matrices: a) testing phase, b) overall; 1-NM, 2 -PM, 3 - HM (Zofka & Błażejowski 2018).

However, the bitumen source and refining process may not have as apparent influence on the spectra as the content and type of modifier. It is uncertain if ANN or Machine Learning algorithms would be able to distinguish between two products measured from raw binders as in example on the Figure 89.

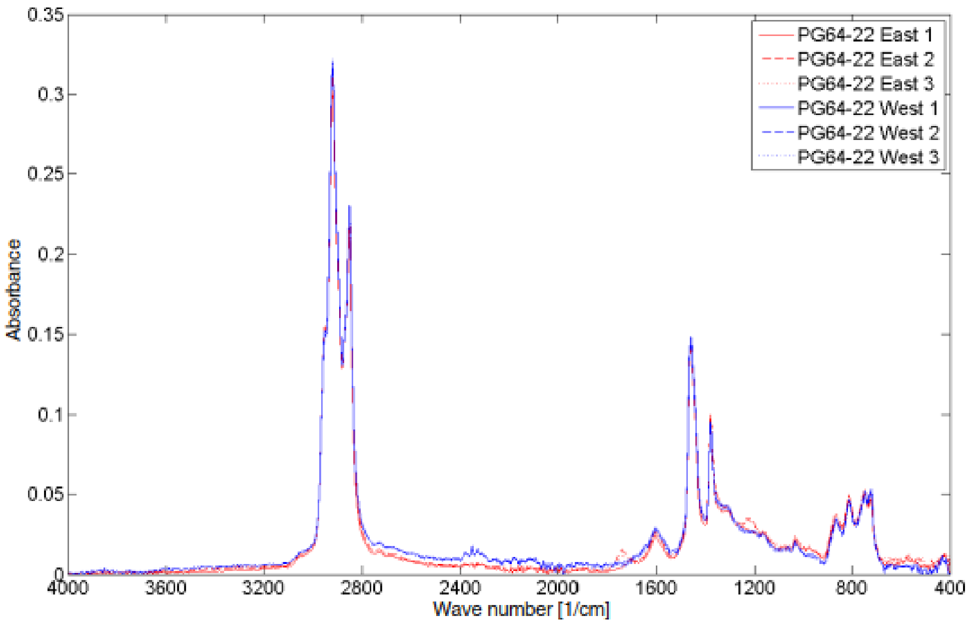


Figure 89. Comparison of ATR FTIR spectra of PG64-22 binders from different refineries (Zofka, Chrysochoou et al. 2013).

7.3.7.2 Suggestion for practitioners

The importance of the correctly build database for the development of the future artificial neural networks is underlined here. The studies reported use the limited sample sets, typically locally sourced.

The suggestion is to keep collecting the infrared data from fresh, RTFOT and PAV aged samples, as well as from those extracted from cores and RAP material before recycling – in combination with knowledge of the source of the original binder in RAP.

The role of digitalization and data storage for the future purposes is huge in this case. A data point without metadata and descriptors is useless for future developments, as will be discussed in the machine learning section of this report. Therefore, it is suggested that testing of the binders and data collection related to the materials available locally in field starts as soon as possible and is linked to the pavements, as well as their performance markers.

7.3.8 Prediction resistance to aging and rheology from infrared

One of the core parts of the Superpave grading system for binders is connected with their response during aging and not just rheology in raw form. With the possibility to identify the types of products due to their different composition, it is obvious that the next step of interest is the prediction of rheology from the recognized and assigned product type.

It was demonstrated that based on the infrared spectra of the binder an approximation can be performed in terms of recognizing the modifier and the aging susceptibility of the product (Yut & Zofka 2014). The rheological response prediction (lnG) was on the deviation level of 10% from measured values.

The parameters giving the highest predictive power of complex modulus values were due to the chemical composition of the base binder and the oxidation reaction products, such as carbonyl group and sulfoxide group, aromaticity and aliphaticity. The study found also that the biggest predictor of the phase angle was in fact the type of the polymer and its content in those PmBs (Yut & Zofka 2014). Hence, the ability to identify the modifier is of high importance (Yut & Zofka 2014). However, the most reliable data can be obtained from the binder itself and not asphalt concrete (Zofka, Chrysochoou et al. 2013).

7.3.9 Antistriping additives

The investigation related to the recognition of the antistripping additives was conducted within the SHRP2 research program as well (Zofka, Chrysochoou et al. 2013). Below are reprinted reference database spectra, obtained during the evaluation, for the two antistripping additives AD-here and Kling Beta. The qualitative distinguishing between the products was possible, when they were in their pure form, because the pure substances differ between each other.

It was observed that ATR was useful in determining the quality of the antistripping additive and to monitor its composition or purity. However, due to the fact of significant overlap between the spectra of antistripping additives and bitumen, and extremely low percentage at which the antistripping additives are added into the binder, the antistripping additives were not recognizable from asphalts with this modification (Zofka, Chrysochoou et al. 2013).

It is also advisable for practitioners to understand that not all antistripping additives will bind to the binder. Some of the additives are proposed to work as aggregate surface modifiers and as such will probably not be detectable by default from binder studies.

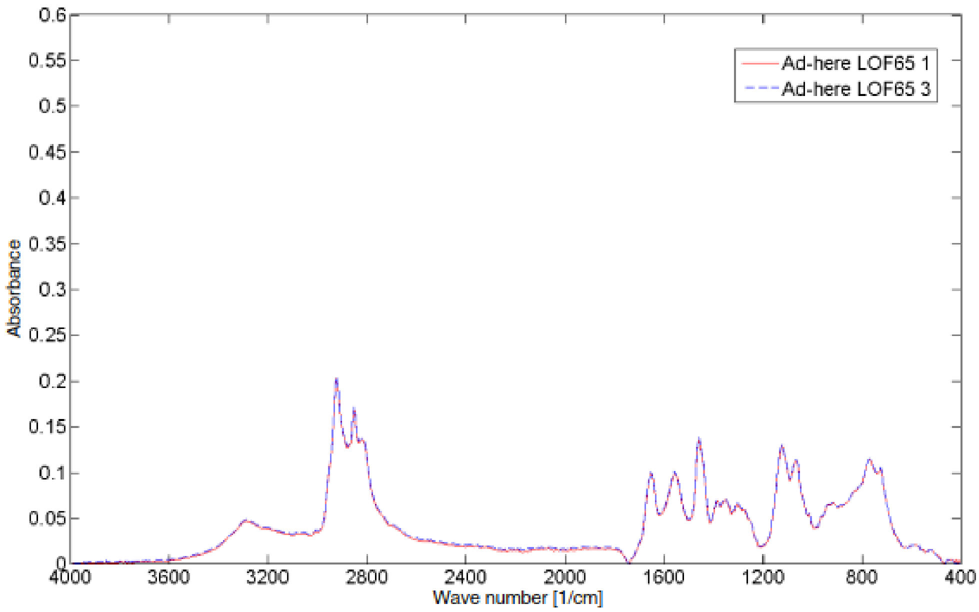


Figure 90. Baseline-corrected ATR-FTIR spectra of ArrMaz's AD-here (Zofka, Chrysochoou et al. 2013).

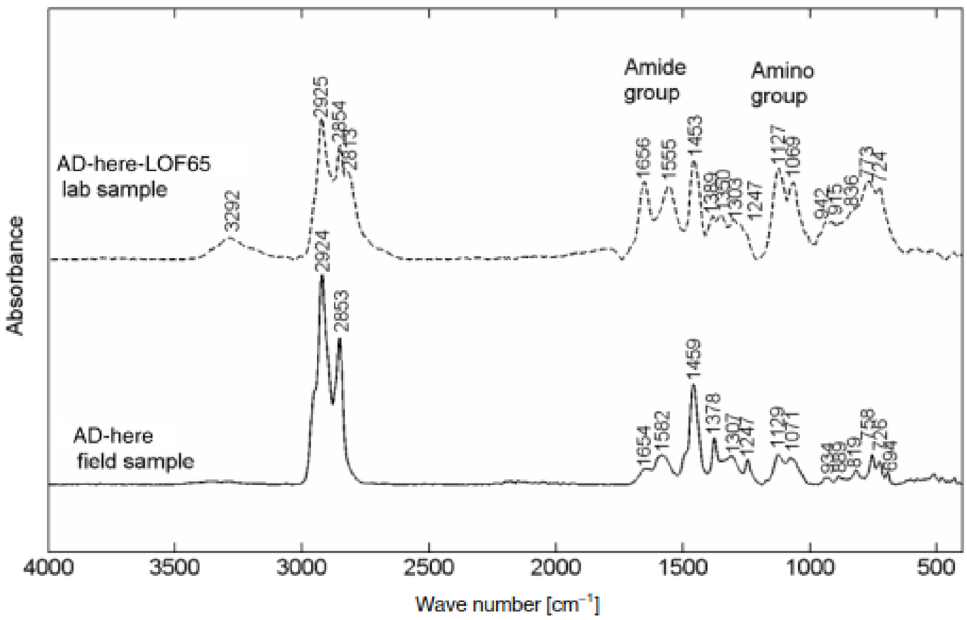


Figure 91. Comparison of the lab and field ATR FTIR spectra of ArrMaz's AD-here (Zofka, Chrysochoou et al. 2013).

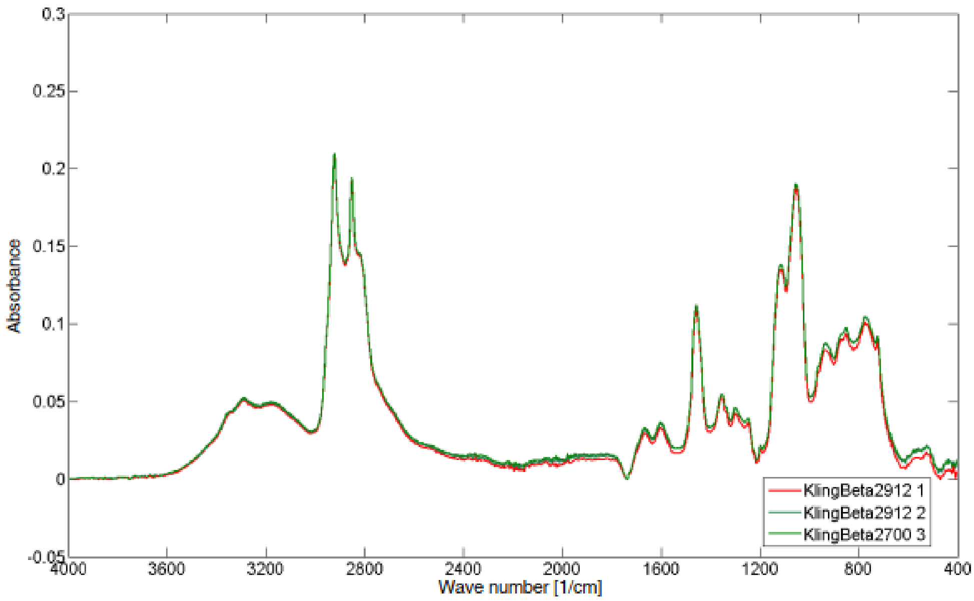


Figure 92. Baseline-corrected ATR FTIR spectra of Akzo Nobel's Kling Beta (Zofka, Chrysochoou et al. 2013).

7.3.10 Rejuvenators or extending oils

A variety of rejuvenators or extending oils can be added into the bitumen or RAP material during processing. The purpose of adding such materials is usually focusing on lowering the total viscosity of the binder in asphalt concrete in order to allow for the pavement to withstand the traffic loading during specific thermal regime, while allowing for reuse of the otherwise discarded material, namely RAP (Zaumanis et al. 2014). However, it should be expected in future that novel materials would start emerging in which blends of crude derived bitumen and bio-derived components are mixed, similar as in the case of ethanol as bio-derived extender of gasoline in Europe. In such case the properties of the additive and its content, may influence the resistance to aging (Wang et al. 2021; Blomberg, Makowska & Pellinen 2016; Elkashef et al. 2018). Luckily, it is worth to note that some bio-based rejuvenators have very distinctive signal in FT-IR spectra which can help in their identification in blends (Makowska & Pellinen 2020).

The ATR IR spectroscopy was used in the past to study the kinetics of aged and fresh binder rejuvenation and as such can be helpful to determine optimal rejuvenator content, time and temperature of mixing of rejuvenator and RAP (Karlsson R. 2003; Karlsson & Isacsson 2011; Makowska 2017).

7.3.11 Impurities during extraction and mastic composition

In the work on contamination of the bitumen with mineral particles of filler during extraction from asphalt concrete samples, it was reported that analysis of bitumen with suspended mineral matter is practically also plausible. In the article (Makowska & Pellinen 2016) author use three mineral fillers, KF (limestone), KK (granite) and FA (fly ash) (Figure 93).

The characteristic peaks for fillers were noticeable in the recovered bitumen spectra. Limestone (KF) expressed most visible change at 875 cm^{-1}, granite and granodiorite at 588 cm^{-1} and fly ashes contributed to the increase of signal at 400–500 cm^{-1}, and those bands were used in qualitative calibration. For the FA the band at 464 cm^{-1} was used, which is seemingly located on the shoulder of a peak with the maximum expressed in the far-IR region (Makowska & Pellinen 2016).

In order to determine how the filler contributes to the rest of the spectra as well as to determine the content of the impurities, a calibration was demonstrated for two fillers named FA and KK. A set of mastic was prepared for FA and KK, where 0, 1, 5, 10 and 15 % concentrations were used. In case of KF blends of 0, 0.25, 0.5, 0.75, 3, 15 and 27 % were used. For FA and KK the intensity of marker peaks increased linearly with increasing concentration. In case of KF, the dependence was linear until 15% but not above. The interpolation between points 15–27% was conducted for the higher concentrations. Example of the calibration between FA concentration and absorbance intensity of the marker peak is provided in Figure 94, where the principle of Lambert-Beer law is seemingly applicable (Makowska & Pellinen 2016).

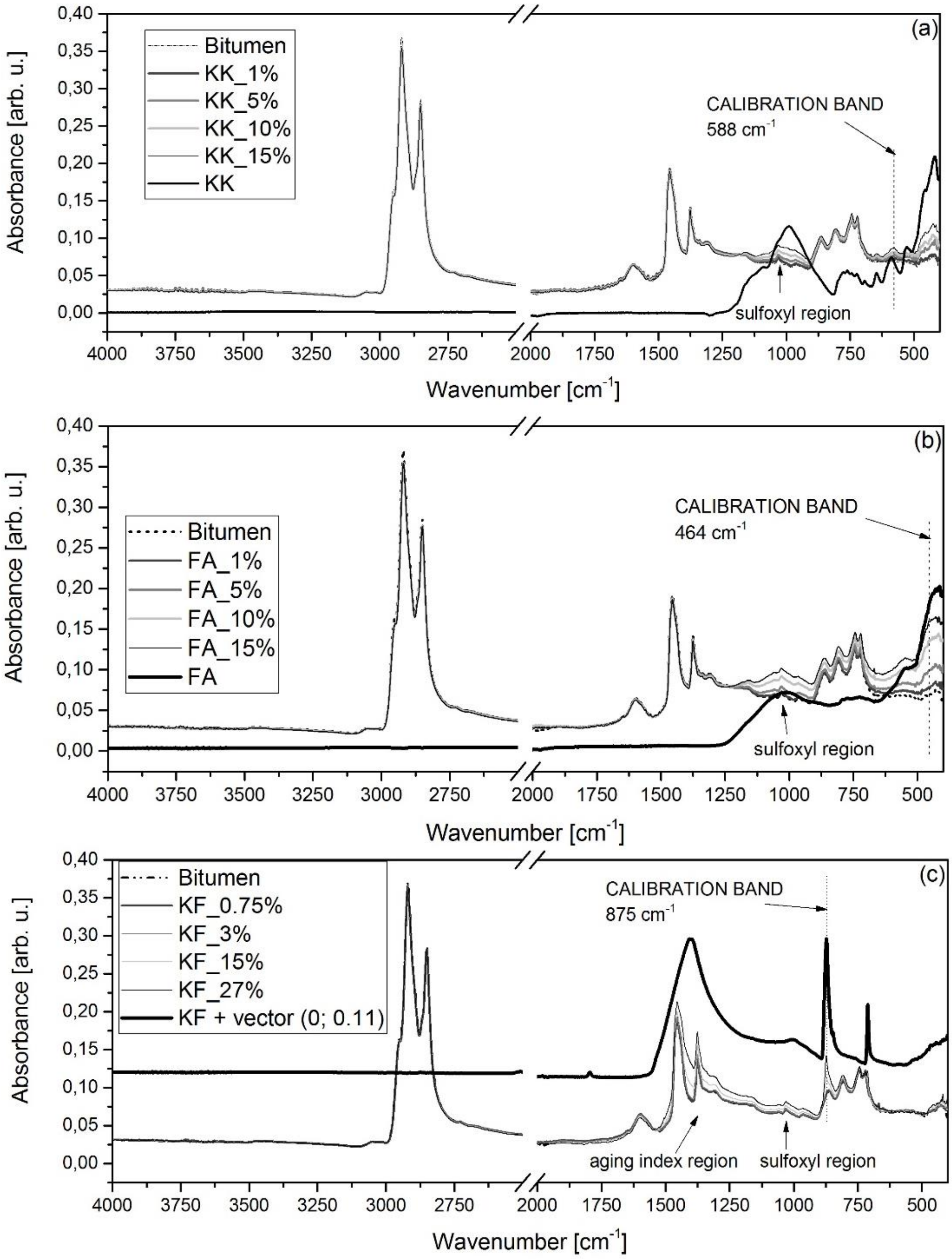


Figure 93. An example spectra used for the qualitative calibration of mastics containing a) granite, b) fly ash and c) limestone at various mass concentrations [wt.%]. The region typically used in the analysis of bitumen is marked with arrows, and so are the calibration bands used for different material combinations.

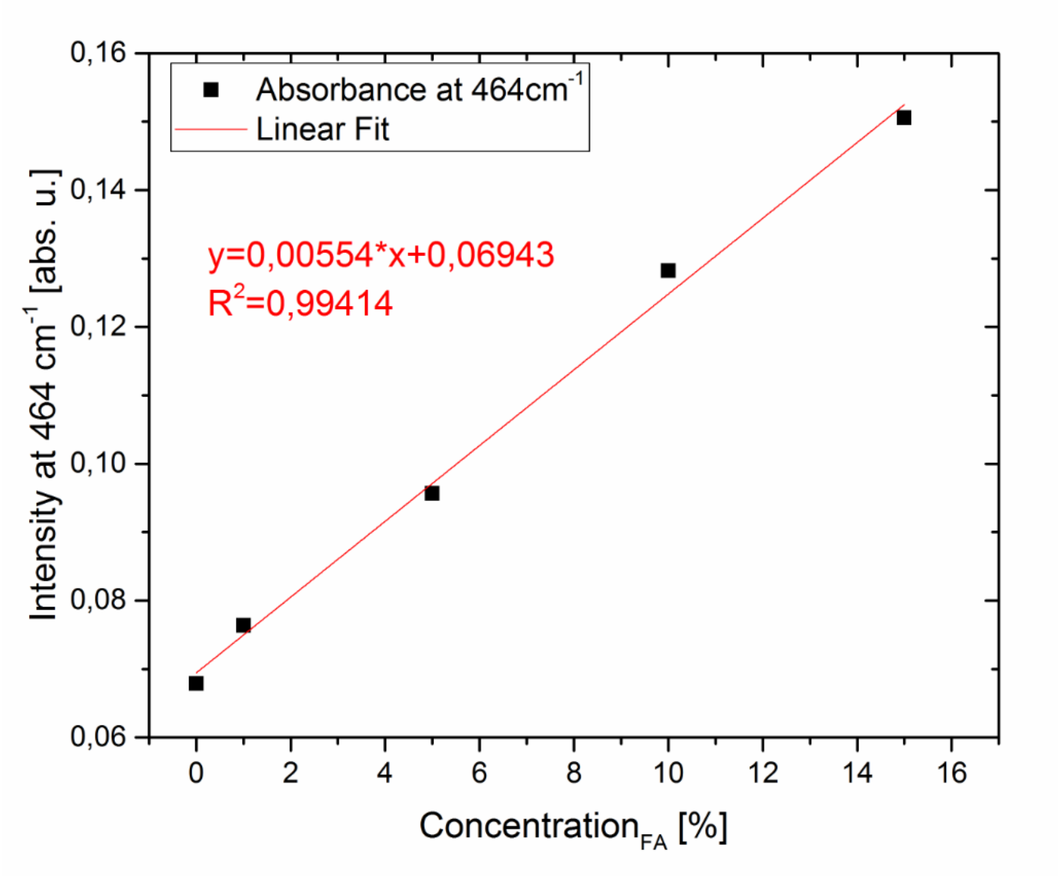


Figure 94. Example of how the absorbance spectra from ATR measurements can still follow the linearity defined in Lambert-Beer law (eq. 1).

7.3.12 Application of ATR to RAP

Some of the FTIR ATR equipment are designed to be extremely robust and readily available for portable laboratories (Figure 95). For this reason, within SHRP2 it was investigated if the samples obtained in the robust form from the field would be measurable. Additionally, the test was applied to stockpiles of RAP in order to measure their oxidation and increase in potential hardening when in storage (Chrysochoou, Johnston & Yut 2014).

Figure 95. The ATR equipment can be portable and thus available for studies of roads in field, where a material is drilled into the form of powder and measured (Chrysochoou, Johnston & Yut 2014).

As is visible in Figure 96 One of the interesting applications of ATR FTIR spectroscopy is quality control of prepared pavements with high contents of RAP. Based on the analysis of ready pavements, performed from such small sample drill out it was possible to determine if the material didn't contain RAP or contained it at higher concentrations (Zofka, Chrysochoou et al. 2013). In fact, such quality control of problematic areas on newly paved surfaces could help us understand if the locally higher concentration of RAP may be contributing to the distresses in the future etc., but also to simply conduct quality control of final product and evaluate pavement production line quality.

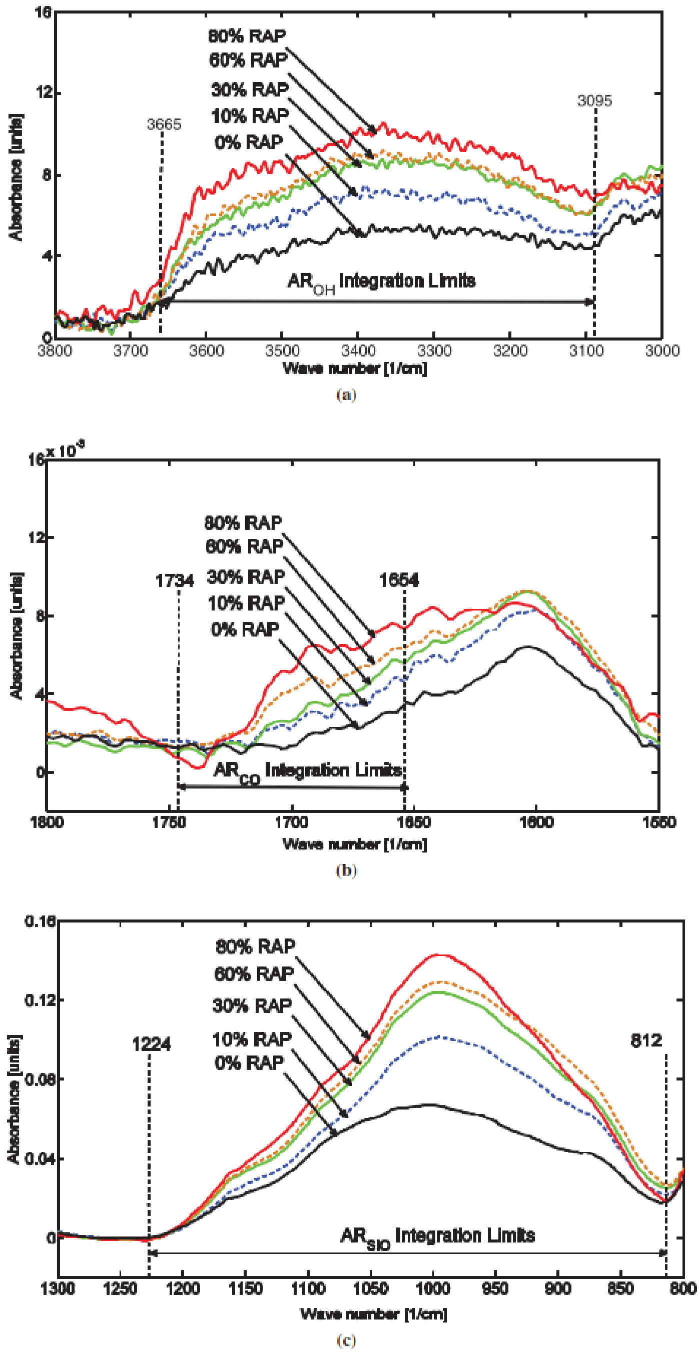


Figure 96. Integration limits lduring analysis of the ATR FTIR spectra for a) hydroxyl (AR_{OH}), b) carbonyl (AR_{CO}) and c) silicate/sulfoxide (AR_{SiO}) absorption bands. (Zofka, Chrysochoou et al. 2013).

Yet another interesting application for the FTIR technique in asphalt industry is the study of RAP piles and their oxidation during storage. The RAP piles may be waiting for use after milling for a longer time, or even prior to crushing and milling. One of the aspects investigated during the SHRP2 program was the analysis of the piles after 1–2 years in storage and 5–6 years in storage. The conclusion was that the FTIR could be a good tool to monitor not only oxidation, but also moisture content in RAP. A slight correlation was also observed between the latitude of the location of stockpile to the carbonyl index recorded from RAP. (Chrysochoou, Johnston & Yut 2014)

Such observations stay in line with assumption that exposure to sunshine and temperature plays the role in the hardening of the stockpiles, just like asphalt concrete's geographical location affects its ageing in field (Mirza & Witczak 1995).

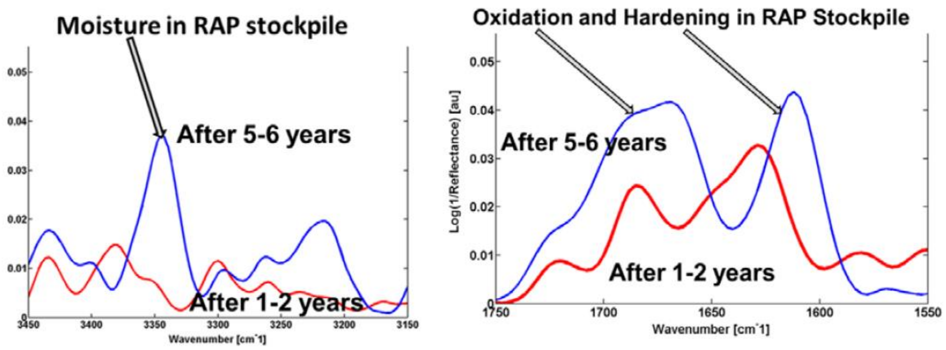


Figure 97. Comparison of moisture (left) and oxidation and hardening (right) in RAP stockpiles after 1 to 6 years of storage as measured by DRIFTS (Chrysochoou, Johnston & Yut 2014).

7.3.13 Application of FT-IR to other construction materials

During the SHRP2 program researchers evaluated the use of FTIR techniques not only for binder but for multiple applications related to road construction, which included cement concrete analysis, paints and markings quality control, etc. Based on the results of analysis it was determined that the method is most useful for the recognition of the polymer modified bitumens, but interestingly can be of significant use for the analysis of road markings. Below figure defines the average score for each application and the rank of the best applicability as decided by the researchers involved in the project based on the data obtained.

Material Category	Objective	Average Score	Rank
Structural coatings and pavement markings	Verification of chemical composition	3.05	2
	Verification of presence of solvents/ diluents	2.73	3
Epoxy adhesives	Verification of chemical composition	2.68	3
PCC	Verification of presence of admixture in fresh/cured PCC mix	2.65	3
Curing compounds for PCC	Verification of chemical composition/ degree of cure (water content)	2.76	3
Polymer-modified asphalt Binders, emulsions, and mixtures	Verification of type/class of polymer modifier	3.45	1
	Determination of polymer content	3.52	1
Antistripping agents	Verification of presence/type	3.55	1
RAP	Verification of RAP presence in mixture	2.30	4
	Determination of RAP content in HMA	3.05	2

Figure 98. The evaluation of usefulness of FTIR technique for the quality control analysis of the road construction related materials (Zofka, Chrysochoou et al. 2013).

7.3.14 Other spectroscopic techniques applied to road construction materials in comparison to IR

The SHRP2 project investigated the use of variable portable spectroscopy methods: FTIR ATR, DRIFTS, Raman, XRF and XRD for use in field for the purpose of material identification and quality control. Additionally, two stationary methods (the samples needed to be brough to the laboratory and processed there), namely Gel Permeation Chromatography (GPC) and Nuclear Magnetic Resonance (NMR). The conclusions are summarized in Figure 99 (Zofka, Chrysochoou et al. 2013).

Material Category	Objective	Portable Methods				Stationary Methods	
		FTIR	Raman	XRF	XRD	GPC	NMR
Epoxy coatings, paints, and adhesives	Presence of solvents	Yes	na	na	na	No	Yes
Waterborne paints	Presence of water	Yes	na	na	na	na	na
PCC	Identification of admixture in PCC mix	Yes[a]	na	No	na	na	na
	Quantification of content	No	na	No	Yes[b]	na	na
Curing Compounds for PCC	Identification of curing membrane on PCC surface	Yes	na	na	na	Yes	na
Polymer-modified asphalt binders, emulsions, and mixtures	Identification of polymer and water in product	Yes[b]	No[c]	na	na	Yes	Yes
	Quantification of content	Yes	na	na	na	Yes	na
HMA concrete	Detection of contaminants (e.g., motor oil, diesel fuel)	No	No[c]	na	na	Yes	Yes
Antistripping agents in binders and mixtures	Identification of antistripping in product	No	No	na	na	No	Yes
	Quantification of content	No	No[c]	na	na	No	na
Oxidation in RAP	Verification of presence in mixture	Yes	No[c]	na	na	Yes	Yes
	Quantification of content	Yes[c]	na	na	na	Yes[c]	na

Note: na = not applicable.
[a] For concentrations greater than 0.4%.
[b] High variability in results is expected.
[c] Not applicable for solids and fluorescing constituents.

Figure 99. Summary of Success in Spectroscopic Identification and Quantification of Additives and Contaminants during SHRP2 project (Zofka, Chrysochoou et al. 2013).

The Raman spectroscopy did not provide reliable source of data and therefore will not be discussed further in the context of binder. X-Ray Fluorescence was used for the studies of cement concrete and the finding was that the technique is of no particular use. However, in a different study it was pointed out that XRF can be a useful technique during the determination of composition of inorganic mater composition, such as fillers (M. Makowska 2017). The XRF is expected to be of more use when a strong non-common metal signal is expected – e.g. waste engine oil modification, pigments, paint, stability control of PmBs, studies of waste soil stabilization with bitumen.

The GPC and NMR were found as good techniques to distinguish between products (Zofka, Chrysochoou et al. 2013), and further tests indicate that indeed a separation of bitumen into components and analyzing their ratios may in fact be promising way to identify the product in hand (Planche et al. 2019). The suggestion would be to collaborate with specialists in the field of those respective techniques to evaluate how likely would those techniques be for uptake in the asphalt industry.

According to the knowledge of the author of hereby report, the techniques require a specialist for execution and analysis of data which would require the company to keep a staff devoted to the measurement in house. Nevertheless, the techniques may be providing more detailed analysis of bitumen samples if a disagreement on the product quality is encountered. Such measurements can be then outsourced to the specialized laboratories. It is advised to remember to include the reference samples when doing so, because of the lack of the status quo type database of the materials currently on the market.

7.4 Differential Scanning Calorimether (DSC)

Differential Scanning Calorimeter has been demonstrated mostly for the purpose of studying the behavior of waxy bitumen and glass transition temperature of bitumen types. The technique measures the amount of energy required to increase or decrease sample's temperature by 1 °C. The result is plotted in relation to the temperature scale used. The phase transition of mater from solid to liquid and liquid to gas typically involves additional portion of energy, so-called enthalpy of fusion. The enthalpy of fusion describes how much energy needs to be submitted to 1 mol of solid substance to melt it into a liquid. Therefore, the concentration of the solid particles in a liquid bitumen will affect intensity of energy consumption at melting point.

Essentially from the measurement in DSC we can determine:

- if the solid particles capable of melting (e.g. waxes) are present in binder
- at which temperature the transition occurs
- and how much energy is consumed on this transition (i.e. what is the concentration of the solid particles).

The technique is very useful to confirm the presence of waxes or compounds otherwise known as crystalized organic solids (Soenen, Besamusca et al. 2013; Makowska, Soenen et al. 2020 (moved to 2021)).

7.5 Techniques related to PmBs

7.5.1 Ductility

Determination of the elastic recovery of modified bitumen can be obtained using the test specified in the Standard SFS-EN 13589. The test is like the DTT test discussed previously. The test uses a specimen of bitumen molded in the form of a dog bone and attached to two holders. The two ends are then pulled apart with fixed speed and nominal force F of the device, up to the length of 200 mm. Afterwards the force is removed and a sample is allowed to shrink naturally, which is referred to as elastic recovery. The viscous element in bitumen causes permanent deformation, and it is the elastic element which acts as a spring element in typical viscoelastic models.

The measurement can be repeated at various temperatures, and as per Finnish specifications the test shall be performed at 10 °C. In some occasions the samples may break before the sample is stretched to 200 mm, due to the insufficient viscous component. In such case the length of the sample at breakage is measured and after a while the length of the half specimens is rerecorded and used in the calculations as value after relaxation. The result is then displayed in form of percentage of recovery of shape.

The test allows to differentiate between materials in terms of resistance to cracking. The MSCR test described previously (7.1.6.1.3) is in principle able to provide similar type of information, that is percent recovery.

7.5.2 Determination of deformation energy (SFS-EN 13703)

The test uses the measurement described in 7.5.1 , that is Ductility but performed at 5 °C. However, the result is not expressed in form of percent, but in the form of energy required to obtain a certain displacement within the specimen. The calculation method is described in the standard.

7.5.3 Fluorescent microscopy

Polymer Modified Bitumen (PmB) consists of the bitumen and polymer, which at certain concentration of polymer may create homogenous solutions. However, upon increase of concentration, prolonged time of storage at higher temperatures or due to the properties of polymers, the coalescence of polymer particles occurs (Zhu, Birgisson & Kringos 2014). Because polymer and bitumen have different densities, the gravitational segregation during hot storage occurs (Lu, Isacsson & Ekblad 1999). At different depths in the PmB tank, different densities, softening points and concentrations of polymer are observed (Polacco et al. 2015; Masson et al. 2003).

Because upon excitation with UV light the polymer and bitumen fluoresce differently, it is possible to distinguish the phases under the Fluorescent microscope. Seoenen et al. (Soenen, Lu & Redelius 2008) review preparation method for the samples investigated with FM. The suggestion is to use the freeze-fracture method to study the structure of PmB. The influence of sample preparation, especially cooling rate after homogenization, affects to the coalescence of polymer rich phase, hence the suggestion is to limit the time during which sample remains in hot conditions. The influence of the hot storage of the sample on the rheological data in that work is more in line with filler modified bitumen than PmB (discussed in section 7.1.6.2.2), which the FM pictures in that work confirm.

The Styrene Butadiene Styrene is composed of two blocks, namely polybutadiene (PB) and polystyrene (PS) block. The simplified chemical composition of SBS is visible in Figure 100. The order of the groups and their location in the final SBS is random or dependant on the manufacturing technology. However, in principle the aromatic rings are coming from PS, the alkane (aliphatic) chains are coming from coupling between PB and PS, while the double bond (C=C) in aliphatic chain comes from the PB. The aromatic edges (or end blocks) in the polymer were suggested to act as the network stabilizers due to the pi-pi stacking that can happen between one chain of polymer and another one. The double bonds in the aliphatic chain are what adds rigidity to the structure and increases the free volume between the molecules. In SEBS, which is a hydrogenated version of SBS, the double bonds are opened and a structure is more flexible, meaning that the free volume between the molecules is reduced.

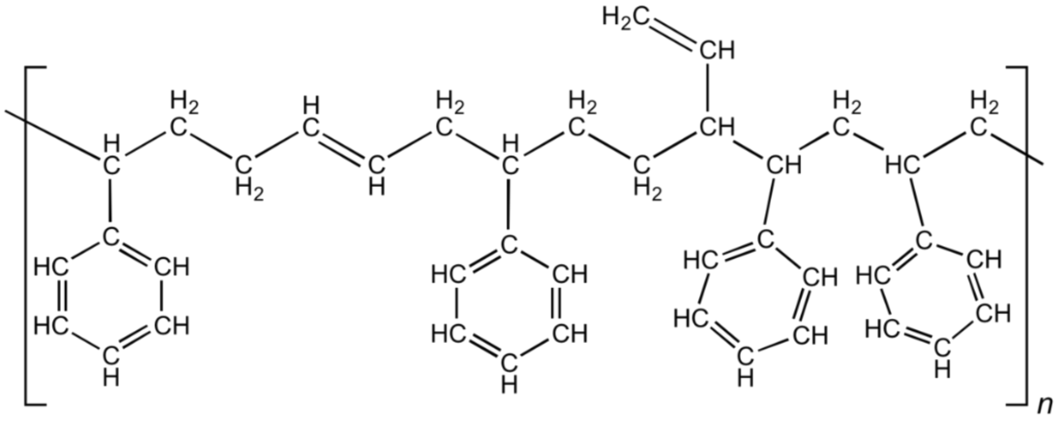


Figure 100. Chemical structure of the block of SBS (picture reprinted from Wikipedia) where aromatic rings of styrene are visible along the alkane characteristic bonds (C-C), and alkene characteristic bonds (C=C).

Below is a variety of the structures observed for stable and unstable samples of polymer dispersed in the bitumen (see Figure 101 and Figure 102) from the literature (Schaur, Unterberger & Lackne 2017). According to this recent study, the PS content is a key parameter to choose in linear polymer. Increasing PS block content leads to more coalescence and segregation. For radial polymer, the PS content was not as important, as it was proposed that the increased amount of end blocks of PS forms the polymer network (Schaur, Unterberger & Lackne 2017). The pi-pi stacking works like little magnets – the linear long chain can fold and create a particle rather than a network because the magnets in the molecule stick to itself (also referred to as self-assembly). In radial arrangement, the folding is less likely due to the geometric reasons, therefore the pulling action of the pi-pi interaction is directed at aromatic rings from other polymer molecule – and the network is formed.

The PB block was found to swell more in the saturates, but the radial polymer of similar PB content was absorbing more saturates (Schaur, Unterberger & Lackne 2017). Perhaps upon self-assembly, some of the PB chains are closed inside of the structure of the PS. The radial SBS was found to lead to higher coalescence and development of the polymer network due to the increased number of PS end blocks available (Schaur, Unterberger & Lackne 2017).

Therefore, according to their results, the good network should be the one with particles suspended, or sponge like, rather than the homogenous dispersion. Unfortunately, their DSR analysis has been conducted only at frequency of 10 Hz, and as was discussed previously, lower frequencies are more representative for large molecule studies in DSR.

The suspended particle type of network is risk prone for PmB, due to the precipitation of polymer particles in a gravitational movement and segregation of polymer and bitumen. Full dispersion is risk prone as no network is formed and material is overly soft at high temperatures, the benefit of polymer addition is not realized. Sponge like structure should be the most ideal to hold the load and to withstand segregation and drain down.

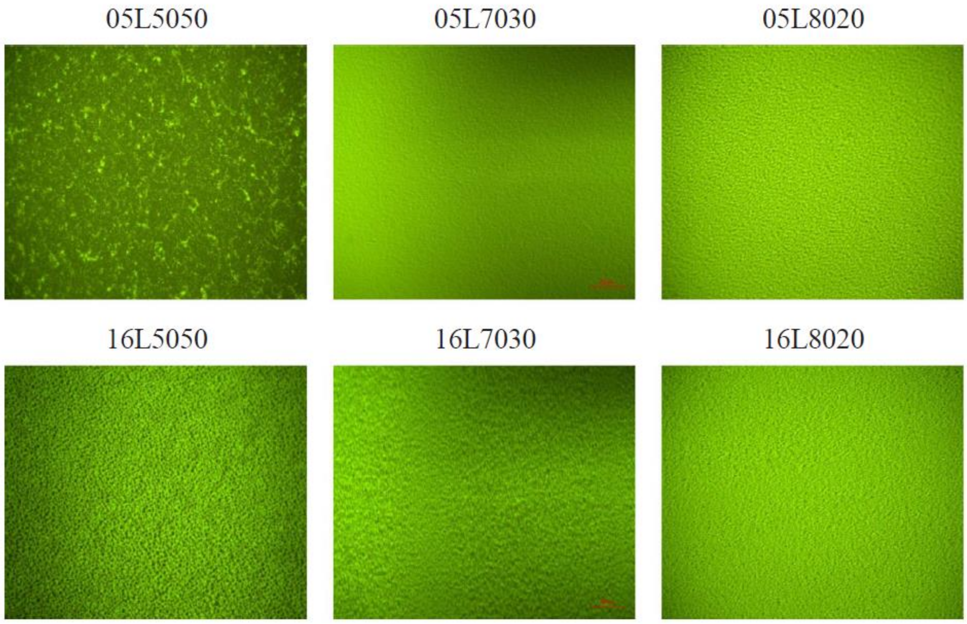


Figure 101. The effect of addition of 5 and 16% of linear (L) polymer into the bitumen 70/100 when the butadiene:styrene content varied 50:50, 70:30 and 80:20, magnification 400 x. Reprinted from Schaur, Unterberger & Lackne (2017).

The definition of the phases in the PmB connects with the composition of the phases, thus they are called bitumen and polymer rich phase. According to phase equilibrium rules of mixtures, some polymer will be present in the bitumen rich phase and some bitumen will be in the polymer rich phase, provided that both are miscible to some level with each other. The concentration of each component in the other phase will depend on the solubility of one component in another. Thus, the composition of the bitumen itself in the bitumen rich phase was not determined beyond reasonable doubt (Polacco et al. 2015). The stability of the system depends on the concentration of the polymer in the PmB, but also on the type of polymer, its molecular weight and shape. Additionally, the composition of the bitumen within the blend was not confirmed beyond the doubt to be composed of an increased amount of asphaltenes (Polacco et al. 2015)

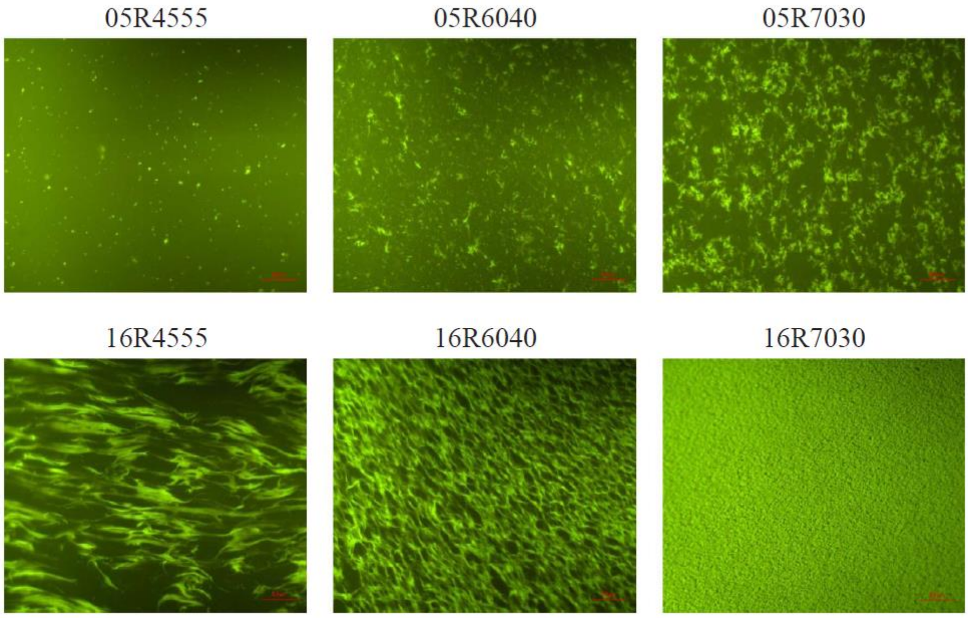


Figure 102. The effect of addition of 5 and 16% of radial (R) polymer into the bitumen 70/100 when the butadiene:styrene content varied 45:55, 60:40 and 70:30, magnification 400x. Reprinted from Schaur, Unterberger & Lackne (2017).

7.5.4 Stability

7.5.4.1 Determination of storage stability of modified bitumen – tube test

Due to the fact of bitumen and polymer having different densities, if the blend is unstable, the gravitational forces will lead to the separation of the materials. As a result, the top section would contain the majority of polymer (lower density) and bottom section would have little or no polymer content.

The test (SFS-EN 13399:2017) is performed by filling the aluminum tubes of certain shape with homogenous mixture of PmB, and storing them at 180 °C, or at a temperature specified by the producer, for 3 days. After such storage the sample is cooled down and cut into three sections – top, middle and bottom. Such prepared samples can then be further analyzed by various methods, e.g. DSR, FM, softening point or simply density measurement.

7.5.4.2 Determination of staining tendency of bitumen (Filter test)

Due to the fact, that the PmB blends are usually prepared using relatively soft binder and polymer, in case of mixture instability and perhaps separation of the liquids, what can be observed is drain-down of the softer component of the mixture. To test if the stability is achieved one can also test for staining tendency of PmB blend (SFS-EN 13301 Determination of staining tendency of bitumen). It is one of the simple and affordable ways to determine possible problems with prepared blends, however, relatively time consuming.

During the test, a retaining ring (similar as in Softening Point measurement), filled with molten bitumen on a sheet of filter paper and supported on a flat horizontal plate is heated in an oven at (80 ± 1) °C for a (120 ± 1) h. The width of the resulting stained circle around the bitumen is calculated to determine the staining tendency of the bitumen. The staining tendency has the dimension of length and results are rounded to nearest 0,5 mm.

7.6 Gel Permeation Chromatography (GPC)

Gel Permeation Chromatography is a technique in which a sample of bitumen, diluted with solvent, is injected into a chromatographic column. The column is a solid or gel of a known porosity. The smaller the molecules of compound dispersed in the solvent, the longer they will retain inside of the column before being eluted, because of the sorption and desorption actions on the surface of the chromatographic column (Spangenberg, Poole & Weins 2011). Larger molecules interact less with the column and thus are being eluted very fast from the column. The proper analogy in larger scale would be sieving of the aggregates: the large particles separate easily and flow through the sieves, but the fine dust often retains on the walls of the sieve column due to the Van der Waals forces. Changing the time of elution according to derived correlations into the average molecular weight of the molecule is plausible.

This technique has been used for various reasons, but mostly for the purpose of studies of the effect of aging (Perez-Martinez et al. 2017) and rejuvenation (Blomberg, Makowska & Pellinen 2016; Gawel, Czechowski & Kosno 2016).

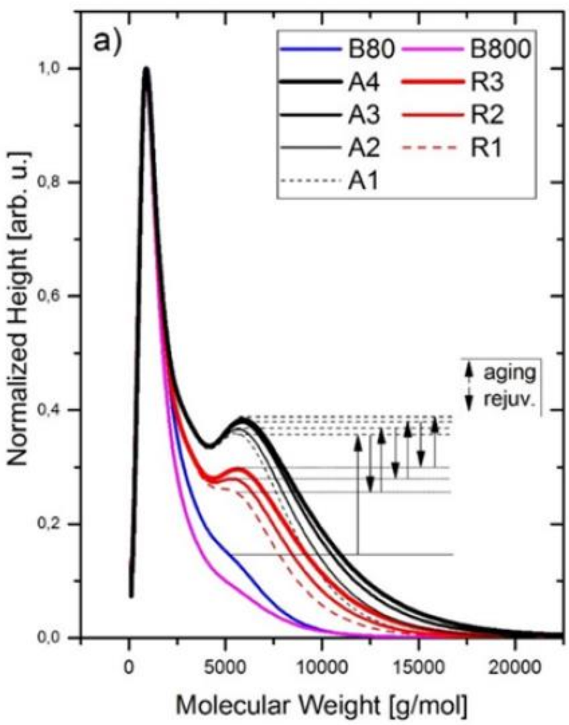


Figure 103. Example of the GPC spectra of bitumen 70/100 (B80) during repetitive Aging (A) and rejuvenation (R) with bitumen 650/900 (B800) (Blomberg, Makowska & Pellinen 2016).

It is worth to underline, that the effect of polarity on the speed of elution is substantial and considering that maltenes and asphaltenes, especially oxidized asphaltenes, can be characterized by different polarity, it is expected that the elution is not only driven by the size of the particles but also by chemical interaction with column's material. Similar principle is after all used as a basis for Thin Layer Chromatography separation (Spangenberg, Poole & Weins 2011) of bitumen known as SARA. In fact, it was recently determined that the carbon number in asphaltenes and maltenes is very similar in bitumen, and that the differences between those fractions should be searched for in the heteroatom content and aromaticity rather than size of molecules (McKenna, Marshall & Rodgers 2013). Nevertheless, with one injection and one solvent used as eluent, a separation of bitumen into various components can be achieved using the GPC and a series of interesting observations can be made that allow for distinguishing of the products.

7.7 Bitumen Fractionation (SARA)

The term SARA is often used in relation to separation of bitumen into fractions which are referred to as Saturates, Aromatics, Resins and Asphaltenes. The fractions are characterized by a different solubility in certain solutions. This fact is used to separate the bitumen by eluting it with portions of different solvents to gradually remove more polar components one at a time. (The analogy here would be removing the sugar from mixture of sugar and sand by dissolving the mixture in water, resulting in the water with sugar and the solid sand leftover.)

However, recently the understanding that the methodology derived originally for this separation into fractions has evolved over the decades is lost within the industry. Original separation was performed by means of technique described in standard ASTM D2007, the asphaltenes were separated by means of IP 143. Afterwards when the Thin Layer Chromatography appeared on the marked, it became of high interest for the quality control of the bitumen production, due to the fact of simplicity, short time and lower consumption of chemicals. The methodology described in IP 469 was derived and correlated to the previously used separation techniques. However, the methodologies are correlated and not equal. Therefore, comparing the results from one technique to the other is misleading, as well as even talking about some fractions as asphaltenes or saturates and that was demonstrated recently on the example of waxes (Makowska, Soenen et al. 2020 (moved to 2021)).

Below is a literature review related to evolution of Thin Layer Chromatography technique and how it was correlated to the previously used standard separation methods.

7.7.1 Different methods of fractionation

There are multiple standard methods for separating SARA fractions or at least asphaltenes from liquids containing asphaltenes. The TLC is compared to many of them explained in further sections in order to bring understanding about the differences between them:

- IP469 (IP 469 2006)
- IP143 (IP 143 2001)
- ASTM D 2007 (ASTM International 1998)
- ASTM D 4124 (ASTM International 2018)

- API-60 (Hirsch, ym. 1972) (the double column method).

7.7.1.1 IP143 method

The IP143, ASTM D2007 and D4124 both seem to be based on the clay-gel bed in the column separation.

The IP 143 measures mostly asphaltene content but could also say something about wax content as some waxes are insoluble in cold heptane (Makowska, Soenen et al. 2020 (moved to 2021)).

The method proceeds as follows:

a. The bitumen is boiled in **heptane** → waxes are dissolved, while asphaltenes and inorganics precipitate. **Liquid removed comprises maltenes.**
b. The material is cooled down → waxes precipitate.
c. The solid on filter is → **wax, asphaltenes, inorganic.**
d. The solid is washed with refluxed heptane → assumed removal of **waxes, but the temperature of the refluxed stream will affect here.**
e. The solid is treated with toluene → the **asphaltenes** are redissolved and **inorganic** stays on filter.
f. Then the toluene is evaporated → asphaltenes as solids are left, defined as the wax free solids.

Remark: The volume of n-heptane is scaled to the sample portion, but there seems to be somewhat a freedom to the time of refluxing with heptane in the standard ("not less than 60 minutes or until few drops of n-heptane leave no residue on evaporation").

7.7.1.2 API-60

The procedure of API-60 (Hirsch et al. 1972) proceeds as follows:

a. Two columns – clay and silica, 1 inch x 8 feet
b. N-pentane – for **saturates 2500 ml (clay column)**
c. 5:95 benzene:pentane – for <u>monoaromatics</u> 3000 ml
d. 15:85 benzene:heptane – <u>for diaromatics</u> 3000 ml
e. 60:20 methanol: diethyl ether followed by 100% methanol – for <u>polyaromatic-polar</u> compounds, 500 + 1000 ml
f. The liquids are gathered every 200 ml and evaluated by UV for content
g. They gather the fractions so that only the main peak is considered, but there is always some bleeding from the column, so-called valley. The amount of bleeding depends on crude oil. Therefore there is some material lost in between the fractions
h. The D2007 – is collecting only some liquid and discards the rest that would comprise bleeding from column

Remark: The different solvent is used to precipitate the asphaltenes (pentane instead of heptane), which will lead to different mass and composition of precipitate. As can be seen, the fractions have different names – saturates, monoaromatics, diaromatics, polyaromatic-polar, and not resins, aromatics and saturates.

7.7.1.3 D2007

The D2007 procedure is as follows:

a. **Saturates – n-pentane soluble (clay column)**
b. **Polar** – toluene:acetone 50:50 elution (clay column)
c. **Aromatics** – what stays in the column
d. **Asphaltenes** – separated in a procedure similar to IP143, by filtering the **n-pentane** solution before it is fed onto the column. The division into other fractions is done from a solution that has less than 0.1% asphaltenes.'

Remark: The solvent for precipitation of asphaltenes is also different from IP 143 procedure.

7.7.1.4 IP 469

The IP 469 procedure is based on Thin Layer Chromatography equipment. However, the separation of the fractions is using different solvent combinations than the previously mentioned methods:

a. **Saturates** – n-heptan separated on silica gel, the main peak
b. **Aromatics** – material eluted with n-heptane above the retention factor (Rf) above below 0,6 but also those eluted in second bath using toluene:heptane 80:20 mixture (silica gel)
c. **Polars I** – material that is not soluble in the above, but was eluted with DCM:methanol 95:5
d. **Polars II** – materials not eluted on the silica gel with any of the 3 above mentioned solvents.

Again, it is underlined in here that the naming of the fractions is not Asphaltenes and Resins, but Polar I and Polar II. The reason behind it is explained in the further sections.

7.7.2 Correlations of fractionation methods in literature

The introduction of the TLC-FID method resulted in a series of studies which lead to the procedure having the form and understanding as it is used currently. In order to understand the results obtained nowadays better, the review of the observations in those early years is provided in following sections.

7.7.2.1 Wiberly, Siegfriodt and DiPaola (1951)

The article (Wiberly;Siegfriedt ja DiPaola 1951) is about lubricating oils and testing for the content of pentane and benzene insoluble in them. The method they used was refluxing oils in the solvents, but then centrifuging the solution and obtaining the solids after drying. They observed that part of the "asphaltenes" stayed in the solution and made it "dirty". They advocated the use of coagulants to precipitate the solids.

In a lot of articles or methods, the coagulations step is present. Even the API-60 method (Hirsch et al. 1972) had three stages of purifying the solution before feeding onto the column. They mentioned that using, or not using, those steps affects the results.

7.7.2.2 Ray et al. (1981)

It is the work on some 40 materials, bitumen feedstocks, lubricating oils etc. and the first Round Robin of Iatroskan based TLC-FIC technique conducted by British Petroleum. Authors used TLC-FID but they compared it with D2007 and IP143 methodology, as well as with the below described column separation.

Column separation

- Slica gel 218 g, 2 cm x 110 cm

1. n-heptane – 350 ml
2. toluene – 600 ml
3. ethyl acetate – 250 ml
4. chloroform:methanol 9:1 – 350 ml

TLC-FID procedure

- n-heptane – saturates, 11cm
- toluene – aromatics, 5,5 cm
- chloroform:methanol 90:10 – Resins A, 2 cm
- Remaining – Resins B
- They use N2 for drying between the immersions.
- Solvent for dispersion is **chloroform, not toluene**

Authors already observed that heptane displaces saturates as sharp band at around 7 cm height from origin (origin defined as the spot onto which the sample is applied), but spreads aromatics along the rod. In their explanation – toluene improves separation of the peaks and the combination of the chloroform to methanol separates **Resins A from Resins B**. Similar has been observed recently from bitumen when performing staged elution (Makowska & Pellinen 2020).

What are Resins A and B? Authors define the region around origin of the rod (the place where the sample is added) as Resins B and the one at around 2 cm from origin as resins A. They specifically stay away from calling anything asphaltenes, because those two peaks are present as well in deasphalted solutions in their research.

The authors provide the comparison between the D2007, and saturate, aromatic and Polar fraction for basestock and extracts. The two peaks at origin and at 2,5 cm, in summary are related by numbers to Polar fraction of D2007. They are supposedly not asphaltenes according to the authors (Ray, Oliver & Wainwright 1981). However, there is no separation of asphaltenes before applying them onto the rods, as would happen in ASTM D2007.

One of the experiments performed in this study (Ray, Oliver & Wainwright 1981) was separation of asphaltenes and maltenes with IP143. Then both the maltene and asphaltene fraction were analyzed in TLC-FID. **It turned out that asphaltenes from IP143 split into two peaks of Resin A and Resin B in TLC-analysis.** But also the liquid that is obtained after the separation of the asphaltenes (i.e. dissolved maltenes) when analyzed with TLC-FID has a signal in Resin A and B region (Figure 104).

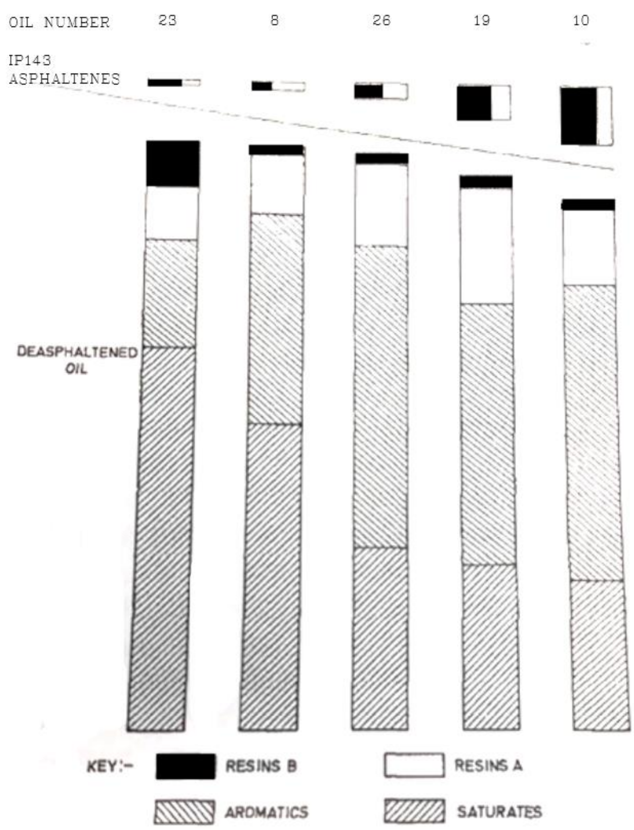


Figure 104. The TLC-FID analysis in terms of ratios of peaks recorded for the asphaltenes and deasphalted oil (both obtained using the IP 143 method), for five atmospheric residues adapted from (Ray, Oliver & Wainwright 1981).

The conclusion from the authors is that TLC-FID is a good approximation, but it does not reflect the results in line with IP143! Another aspect touched upon in the article is the overlap of saturates and aromatics in the extract from the large-scale column separation. For lubricating oil basestock, they recorded that only the first 150 ml extract contained peak assigned as saturates in 100%, with increasing time of the elution and volume of solvent, the content of aromatics increased to 100% in last portion eluted from column (but the mass eluted in those portions was really small). Authors explain that in basestock for this product the presence of alkylbenzenes, which have alkyl chains of more than 30 carbons in a chain, is common and those materials are relatively non-polar. They propose that those are the ones, which elute soon after saturates.

Already in this paper Ray et al. suggest at least for production support purposes, that the n-heptane is used to elute the fraction to the height of 10 cm, then the **partial pyrolysis scan** should be applied to remove the saturate peak. This allows for further development and separation of aromatic fractions into two. Suggested is then second procedure of separation:

- Toluene:heptane 15:85 – to 10 cm
- Chloroform – to 6 cm
- Chloroform:ethanol 95:5 – to 2 cm

The article also discusses how there is a lack of linearity of the mass to signal response. Separated by column, pure saturates are mixed with other components and analyzed in TLC-FID. The recorded value versus blended in value are not linear. The relationship is sigmoidal – underestimated at low concentrations and overestimated at high concentrations. About 40% saturates is optimal. Therefor the TLC-FID should not be understood as definite weight percentage of fractions, because there is deviation from the mass to area linear correlation.

Authors also analyze their data and suggest that the method works best for materials where the fractions are distributed almost equally. In the case when some fraction becomes dominant, there are deviations. For example, if aromatics to saturates would be mixed at 75:25 ratio, the higher concentration of aromatics may be overestimated and lower concentration of saturates would be underestimated in such case with TLC-FID and the result would be estimated as 90:10.

Table 12. Elemental analysis, GPC and TLC-FID results from separated portions of an atmospheric residue, adapted from (Ray, Oliver & Wainwright 1981).

Sample	Approx. % of atm residue	Iatroscan TLC				GPC	Av. mol wt.	Elemental analysis					H/C ratio by atoms
		Sa	Ar	Re A	Re B	Approx. mol wt range		C	H	N	O	S	
Atmosferic residue						350–5000	560	86.2	11.2	0.2	0.7	1.4	1.60
Saturates	33	100				350–2500	440	85.3	14.4	<0.1	<0.1	<0.1	2.01
Aromatic	48		99	<1		350–4000	520	86.6	10.8	0.2	0.8	2.0	1.48
Polars	6			100		350–5000	920	84.4	9.5	1.4	2.0	2.2	1.34
Asphaltenes[1]	8		6	24	70	350–6000	3490	85.8	7.7	1.2	1.3	2.9	1.07

1 Asphaltenes separated with IP143 method

The last section of the paper focused on analyzing the fractions separated from atmospheric residue with column and various processes, to the point when the material that they obtained was giving only a 100% signal in each place on the rod when analyzed with TLC-FID. They performed the elemental analysis, FT-IR and HNMR also on pure fractions. As is visible in Table 12, the sulphur content increases from saturates to Polar fractions from below 0.1% to 2.9% in their example. The more asphaltenes, the more sulphur will be present overall.

One of the conclusions from the article is that the TLC-FID should be used as semi-quantitative method, that is for production line quality control, but not used as a reflection of absolute values, especially for asphaltene content evaluation.

7.7.2.3 Poirier and George (1983)

This article investigates separation of ten bitumen and heavy oil samples from various sources and various distillation processes (Poirier & George 1983). The comparison is made to the API-60 method.

In here the authors follow the method suggested by Ray et al. (Ray, Oliver & Wainwright 1981) with the partial pyrolysis scan (PPS).

- Saturates – n-hexane to 10 cm, **then PPS 10 to 6 cm from origin**
- Second elution – benzene: n-hexane 10:90 to 4 cm
- Third elution – ethyl acetate: benzene 5:95 to 2.5 cm
- They dry in the oven **with air at 120 °C**
- The material analyzed is deasphalted by precipitation of asphaltenes in 20x the volume of **n-pentane.** The maltenes are then dissolved in benzene.

The peaks are assigned to fractions of saturates, mono- and diaromatic, polynuclear aromatics and polar material. Similar as in API-60.

The results of their fractionation by TLC-FID in comparison to API-60 give constantly similar results for saturates and aromatics, but a different split between the other two fractions. TLC seems to underestimate the polar material by 50%, and API seems to underestimate the polyaromatic ring material. This could be partially explained by observation from Ray et al. that for example a fraction understood as polar material (Resin B) can still split in TLC-FID into two peaks, e.g. 70% stays in the most polar region.

7.7.2.4 Yoshida et al. (1986)

The articles by this group are about coal liquefaction products, thus not exactly the bitumen reference. However, they use the separation technique almost exactly as in TLC-FID by IP 469, but with different initial solvent (i.e. hexane):

- **n-hexane** – saturates
- n-hexane:toluene 20:80 – aromatics was investigated as a possible – but then they decided to use just toluene
- DCM:methanol 95:5
- But they analyse only n-hexane solubles of their liquid, so “asphaltenes” are precipitated before.
- the **toluene** is used as dispersant

The interesting part is in the analysis of the reference samples (Figure 105). They use eicosane (C20 paraffin) as a reference compound for saturates. In our opinion it already also contributes partially to other fractions (Polar II), not only the peak assigned to saturates (The peak is marked with blue arrow by us in the figure). This indicates that the waxes or longer chain hydrocarbons can be also contributing to something that recently has been referred to as asphaltenes (Makowska, Soenen et al. 2020 (moved to 2021)).

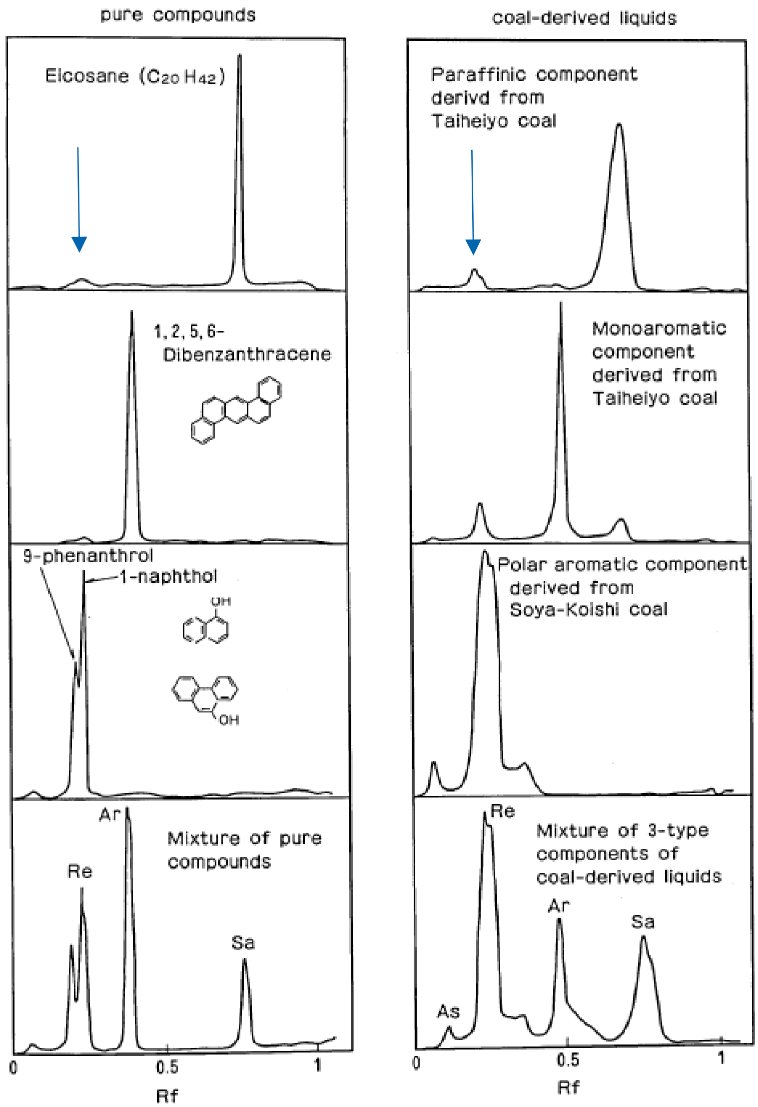


Figure 105. TLC/FID chromatograms of coal derived liquids and pure compounds as reference samples (Yoshida et al. 1986) with arrow pointing the region of Resins.

7.7.3 Lehto (Master thesis, 1988)

The research was sponsored by Neste Oy, and it was supervised by Siv Schüller (Neste Oy) and prof. Erkki Pulkkinen. Conclusion from the literature review was that there was many unknowns in terms of parameters and how they affect the final result. The method was at this point semiquantitative as per previously reported papers. The author used BIT250 and air-blown bitumen B95/35 as her study material.

Chosen combination of solvents for TLC-FID was:

- n-heptane (11 cm)
- n-heptane: toluene (80:20) (5,5 cm)
- DCM:methanol 95:5 (2,5 cm)
- 30-35 sec/rod was deemed best

The author evaluated which solvent would be best to achieve good dispersion of sample, optimal sample size, optimal hydrogen content, speed of scanning, as well as the effect of different concentrations between solvents in bath 2 and 3.

The results were compared to asphaltenes (**asfalteenit**) determined with IP 143 (5,9% and 26,9% for respective bitumens) and other fractions. The deasphalted oil was fed on the aluminium oxidide (207 g) column and divided into fractions as follows:

- Saturates (**öljyt, oils**) – n-heptan 2000 ml
- Soft resins (**pehmeä hartsi**) – toluene 1500 ml
- Hard resins (**kova hartsi**) – chloroform 1300 ml

Lehto repeated the column chromatography two times, and had repeatability within 1% for each fraction (see Figure 106). The samples were taken into GPC analysis and it was established that saturates pass through the column the slowest thus were assumed to have the lowest molecular weight, asphaltenes passed through column the fastest, and other fractions positioned in between. More information can be found in the source (Lehto 1988). The column chromatography was used prior to the referred study in the company and lasted about a week to get a result. Therefore, the TLC-FID was presented as more economical analytical method in terms of laboratory time.

	BIT 250			BIP 95/35		
Fraction	1 (p%)	2 (p%)	ka.(p%)	1 (p%)	2(p%)	ka.(p%)
öljyt	25.95	24.70	25.3	29.60	30.19	29.9
pehmeät hartsit	47.30	48.07	47.7	29.47	30.44	30.0
kovat hartsit	22.68	23.66	23.2	11.88	11.19	11.6
asfalteenit	5.84	5.86	5.9	29.62	29.48	29.6
total	103.8	102.3		100.6	101.3	

Figure 106. Composition by weight percent of bitumens analyzed with the liquid chromatography + IP143 (Lehto 1988).

Afterwards the fractions were taken one by one and analyzed by TLC-FID according to the procedure provided above. As is visible in the Figure 107, each of the separated fractions from liquid chromatography procedure, contributed to more than one region on the rod after the TLC-FID procedure, similar as reported by (Ray, Oliver & Wainwright 1981).

BIT 250	Peak area in TLC–FID Saturate	Aromatic	Polar I	Polar II
Fraction	%Sa	%Ar	%Re	%As
öljyt 1	31.05	64.41	4.54	
öljyt 2	32.89	63.11	4.00	
pehmeät hartsit 1		77.52	20.67	1.80
pehmeät hartsit 2		79.12	18.95	1.93
kovat hartsit 1		31.55	58.64	9.81
kovat hartsit 2		33.96	56.98	9.06
asfalteenit 1			22.32	77.68
asfalteenit 2			24.07	75.93

BIP 95/35	Peak area in TLC–FID Saturate	Aromatic	Polar I	Polar II
Fraction	%Sa	%Ar	%Re	%As
öljyt 1	56.36	40.54	3.10	
öljyt 2	55.16	41.44	4.41	
pehmeät hartsit 1		84.73	14.15	1.22
pehmeät hartsit 2		83.02	15.79	1.19
kovat hartsit 1		16.64	78.68	4.68
kovat hartsit 2		16.85	77.77	5.38
asfalteenit 1			20.85	79.15
asfalteenit 2			19.76	80.24

Figure 107. The column separated fractions from both repetitions, studied by the peak area ratio obtained from TLC-FID chromatograms where Re stands for Resin (Polar I in IP 469) and As for Asphaltenes (Polar II in IP 469), while Sa and Ar, for Saturates and Aromatics, respectively – adapted from (Lehto 1988).

Author was optimizing the parameters such as hydrogen flow (e.g. Figure 108), air flow, sample size (e.g. Figure 109), speed of scanning, etc. so that the result from TLC-FID chromatogram would be correlated best to the result from LC and IP143, but the comparison is not explained or rather the decision making process is not documented. However, it was advised that they are not equivalent fractions and are just correlated. The choices of some parameters are done mainly on the basis of lower standard deviation and better repeatability.

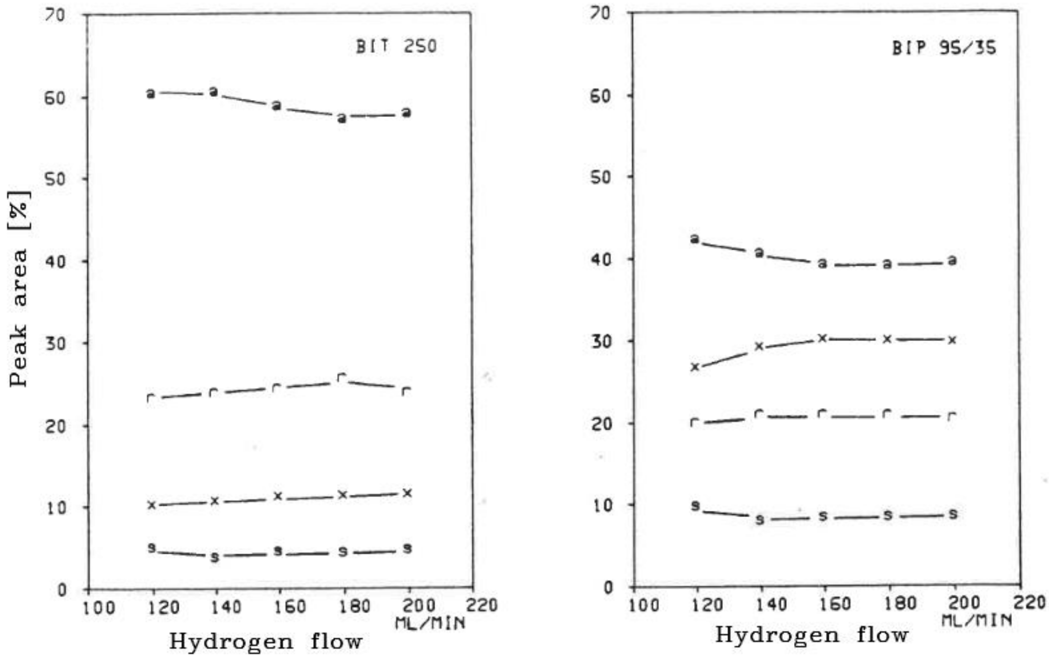


Figure 108. The results of optimization of the fractions due to the flow of hydrogen where s –saturate peak, x – polar II peak, r –polar I peak and a – aromatic peak, adopted from (Lehto 1988).

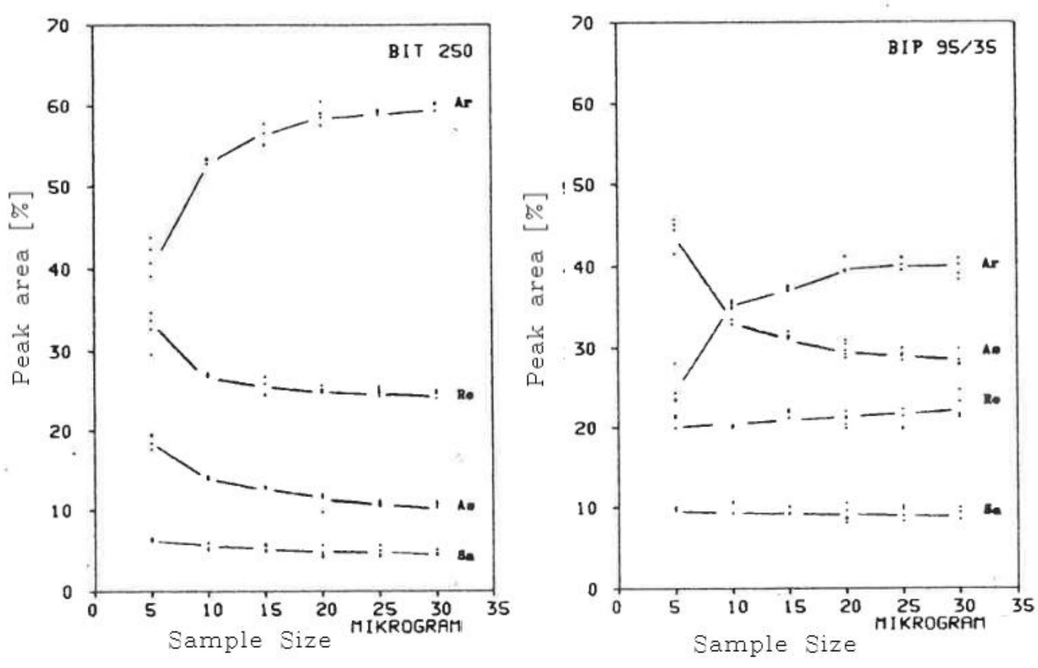


Figure 109. The effect of applied sample size on the resulting ratio between peak areas for respective peaks, where As – Polar II, Re – Polar I, Ar – Aromatics, Sa – Saturates.

As can be witnessed based on the above, every single parameter can influence the value. According to the experience of hereby author, the repeatability within the laboratory for one operator may be in fact extremely good. However, differences between laboratories, due to different equipment, sample application tools and skills in doing so, may affect to the result. In fact, the repeatability and reproducibility levels in IP 469 are extremely large for this technique (IP 469 2006), making it almost impossible to compare small changes in the product without knowing the repeatability between two laboratories specifically.

7.8 Stability by titration in solvents

7.8.1 The context for the bitumen stability evaluation

The crude oil stability depends on the ratio between asphaltenes and paraffinic oils. In order to pump the crude oil, or to reduce its viscosity some low molecular solvents may be added into crude oil. However, this sudden dilution causes aggregation of asphaltenes and precipitation of black solids, which tend to clog the pipelines or lower the yields in the refineries. The Figure 110 demonstrates the effect of dilution of crude oil with n-heptane.

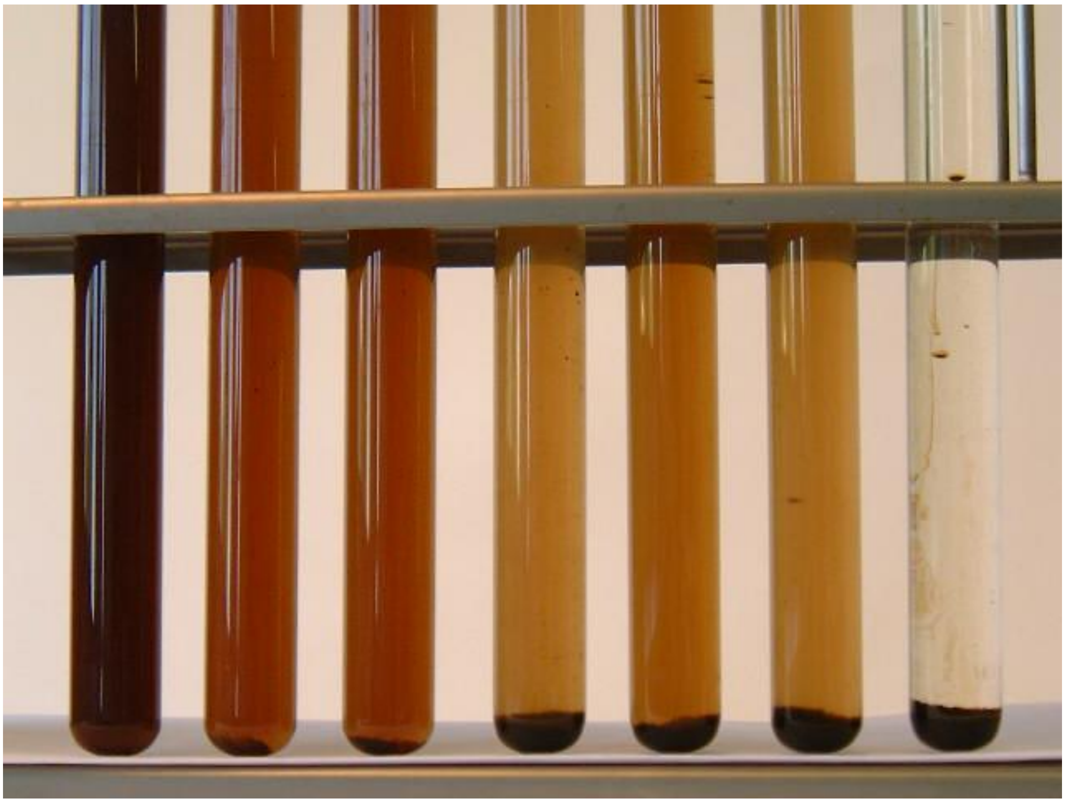


Figure 110. The solution of crude oil diluted with increasing volumes of n-heptane (from left) (Dakin 2006).

As was discussed previously, the crude upgrading processes are utilizing this fact. Deasphalting is using a diluting solvent to precipitate the asphaltenes and to recycle maltenes as a feedstock to the refinery. Visbreaking is causing a shift in the content of constituents in the atmospheric residue (AR). An example of the change in SARA fractions of the AR (otherwise understood as feedstock for the Visbreaker) and a visbroken residue (VBR) is shown in Table 13. As is visible, alongside the reduction of the viscosity, the content of saturates and asphaltenes increases, and content of aromatics decreases. Resins remain relatively on a similar level.

Table 13. The example of SARA composition of AR and VBR adapted from (Stratiev, Kirilov et al. 2008).

Fraction	Vacuum residue			Visbroken Residue			
	Sample 1	Sample 2	Sample 3	Feedstock 1		Feedstock 2	
Saturates	23,7	21,9	19,3	22,8	26,5	27,1	30,0
Aromatics	62	63,4	65,3	56,2	52,4	51,4	47,6
Resins	10,1	10,5	11,1	10,6	13,0	11,8	10,1
Asphaltenes	4,2	4,2	4,3	10,3	8,1	9,8	12,4

The stability of the crude oil, but also bitumen, can be expressed using tertiary graphs of stability (El-Sabagh et al. 2015; ABB Lummus Global B.V. 1996). In the mixture of three components multiple phases are plausible.

Let's consider an analogy of water, sugar and fruit juice concentrate. At some concentration of juice and sugar in water, the mixture is homogenous (referred to as single-phase region). When the sugar content is close to 100% by weight, the precipitate of solid sugar and a liquid of water and fruit juice are co-existing in the system. This is referred to as two-phase region (solid and liquid). All of this can be expressed on the so-called ternary phase diagrams, and if the axis lengths are equal the plot is referred to as Gibbs triangle (see Figure 111).

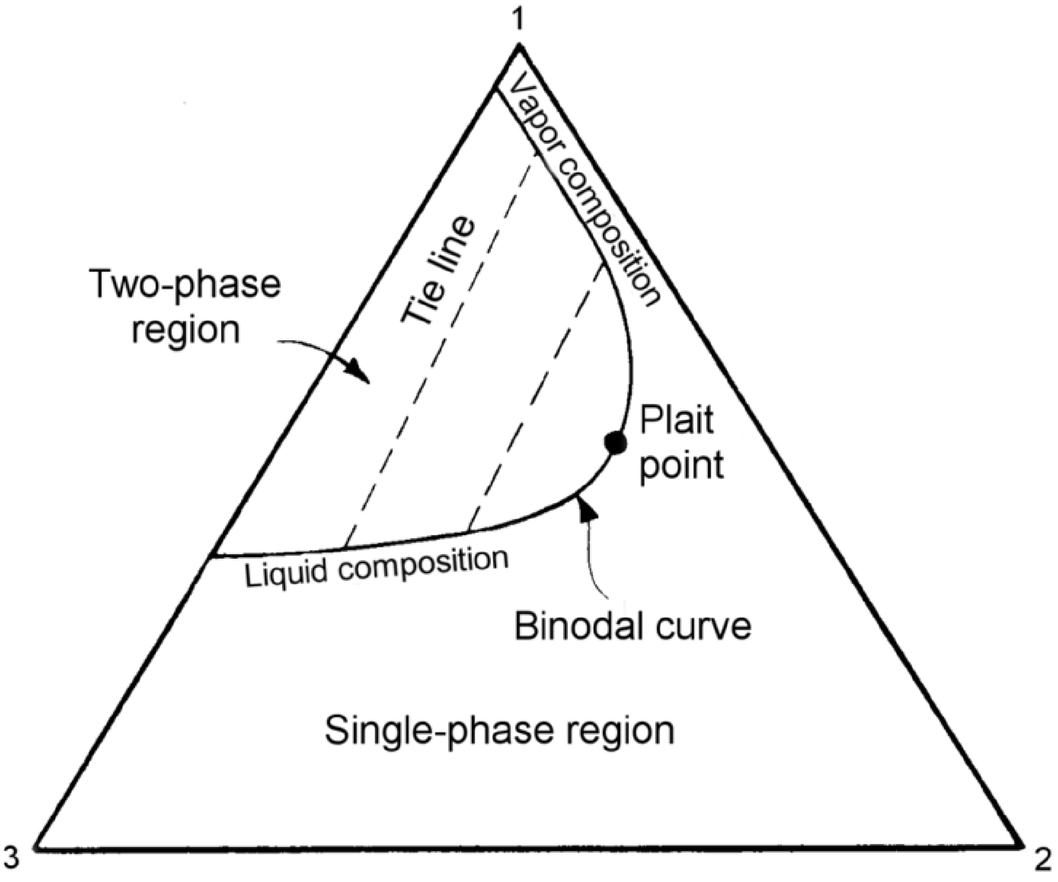


Figure 111. A schematic of a ternary phase diagram (Smith 2013).

The boarder between homogenous and two-phase region mixture of three components is marked on Gibbs triangle as a binodal curve. If the mixture has a composition inside of the region marked by the binodal curve, one should expect instability, separation and/or precipitation of solids.

All of this is also temperature and pressure dependent. Using the analogy of the water, sugar and juice, reducing the temperature below 0 °C can cause freezing of the water and its separation as a white solid (ice) in red thick liquid. Similarly, adding large amount of sugar to water with juice, may create solid in liquid phase system at room temperature, which creates a homogenous mixture when the temperature is increased.

The same principle can be applied to crude oil, AR, VBR or bitumen, as all of those mixtures contain aromatics, saturates (parrafins) and asphaltenes. There is a concentration dependent region of mutual solubility between those parameters. The visbreaking is moving the AR towards the region of instability (two-phase region) due to the change of composition. For this reason the visbreaking process lasts in the refineries as long as the mixture does not reach the instability. The main point of performing Visbreaking is to increase the yield of lighter fractions from crude oil, but to prevent the precipitation of solids which could decrease the energy efficiency of the installation as an insulation layer inside of the reactor would be formed by precipitated particles (Joshi et al. 2008). After the process, the VBR would be distilled again and the lighter, paraffinic fractions would be removed from the mixture, further increasing the content of asphaltenes in the final product (as compared to AR).

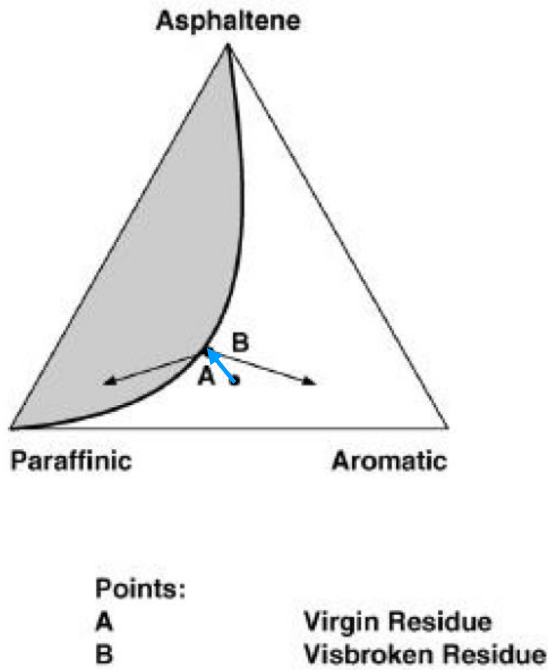


Figure 112. The example of the Gibbs diagram adapted from ABB Lummus Global for crude oil in which the orange arrow indicates the change in composition between AR and VBR. (ABB Lummus Global B.V. 1996).

After the visbreaker, the material is then distilled to remove the lightweight hydrocarbon obtained during the thermal breaking process. It is assumed that the yield from visbroker is approximately 7%. Assuming, that this 7% would be further removed during distillation, and that those 7% is in the form of saturates, we can estimate the SARA fractions of the material equivalent to bottom of the barrel – visbroken distillation residue.

All three of those products will have a different composition, defined by the general fractions. For one feedstock, the Gibbs diagram and the instability region may be constant. However, the visbreaking is altering also the chemical composition of the fractions by increasing for example the sulphur content in the VBR as was discussed in BITU2020 – Part 1 section. It is expected that the AR and VBR would have a different phase diagram. It is plausible that the concentration of the fractions assuring the homogenous structure for AR, when achieved using the fractions of VBR would result in separation of the phases.

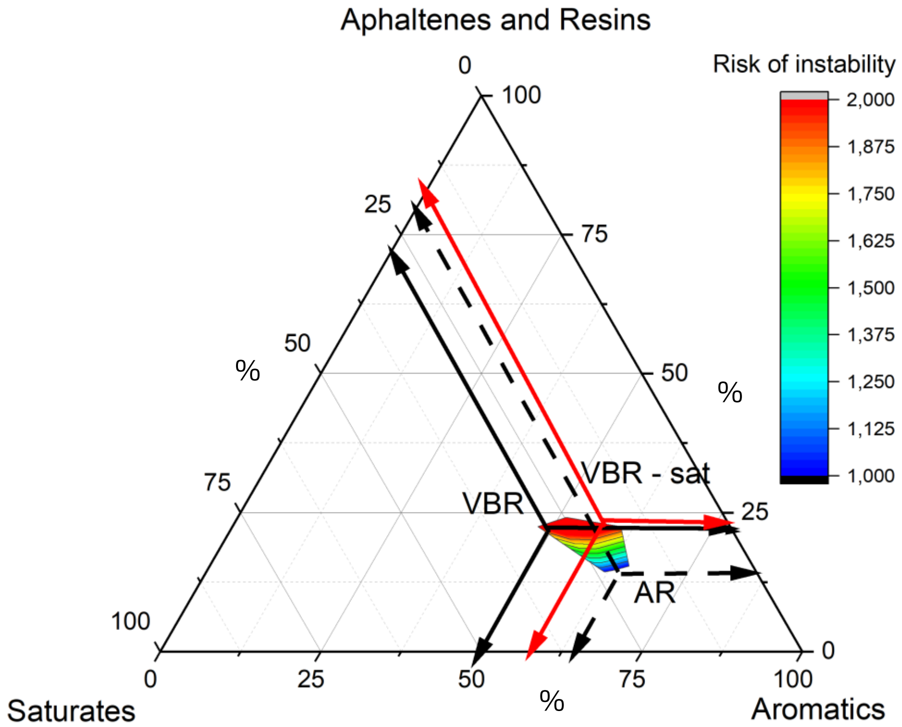


Figure 113. Using the values from the table the AR (dashed arrows) and VBR (full black arrows) are compared in Gibbs diagram. For the theoretical consideration and assumption of venting 7% of saturates from VBR during second distillation is considered as a final product of the process.

The oxidation of AR (if sold as bitumen) or oxidation of VBR (if sold as bitumen) could follow a different path towards the instability region. Instability regions for both of those materials could be still within different compositional space.

Additional problems may occur when rejuvenators enter into contact with the aged AR or VBR. The success of rejuvenation may be dependent on the original composition of the binder and chemical composition of the rejuvenator during recycling. Similar aspects relate to blending polymers and other additives with the binders containing VBR.

Therefore, it should be important to determine:

- The rheological properties of the fresh binder
- The stability of the fresh binder
- The resistance to aging (rheological)
- The resistance to aging (chemical, fraction composition)
- The resistance to aging (stability).

7.8.2 Heithaus titration

The first reports of the determination of the asphalt peptization from solution were reported by Heithaus (Heithaus 1962). In the original study, Heithaus used the bitumen sample dispersed in the toluene and conducted the titration using the solvent n-heptane. The onset of instability was depending on the initial concentration of the binder in the sample as well as the dilution factor. Heithaus demonstrated this by conducting the titration (volume of titrant, V_T) of multiple solutions of bitumen with the differing original weight of bitumen (W_a) in the volume of toluene (volume of solvent, V_s), see Figure 114. As V_T increases, the sample dilutes, and an instability in the solution is observed. At a certain ratio of bitumen to solution the precipitation of asphaltenes is observed.

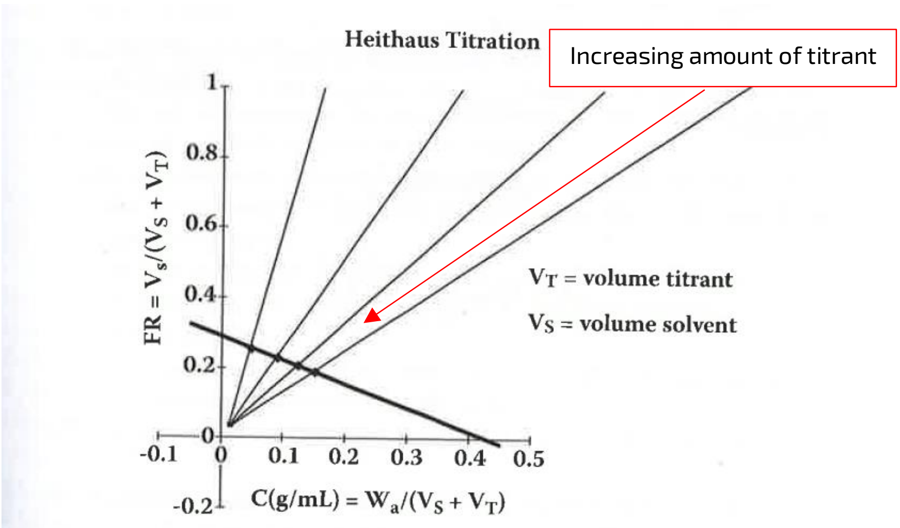


Figure 114. The principle of the Heithaus titration procedure (adapted from C. Hansen 2007).

The instability can be observed under the microscope or via spotting on the filter paper or via the spectroscopic techniques (Pauli 1996). Each of the techniques of determining the onset of the peptization will influence the repeatability.

The time between addition of the solvent and recording of the onset of peptization also plays a role in the determination of the end point of titration. Visual evaluation may be misleading. The change in the transmittance of the solution has been used for automatization of the procedure (Pauli 1996; C. Hansen 2007). Reports emerged on the use of quartz crystal resonator sensors during the procedure to detect the end of titration or peptization onset (Daridon et al. 2013).

One of the parameters of stability is unfortunately also time Figure 115. A solution diluted to the particular level may still destabilize given sufficient amount of time. For bitumen mixtures obtained by means of blending with different modifiers, the long term process we typically call physical hardening, can be affected in the case of badly matched modifiers.

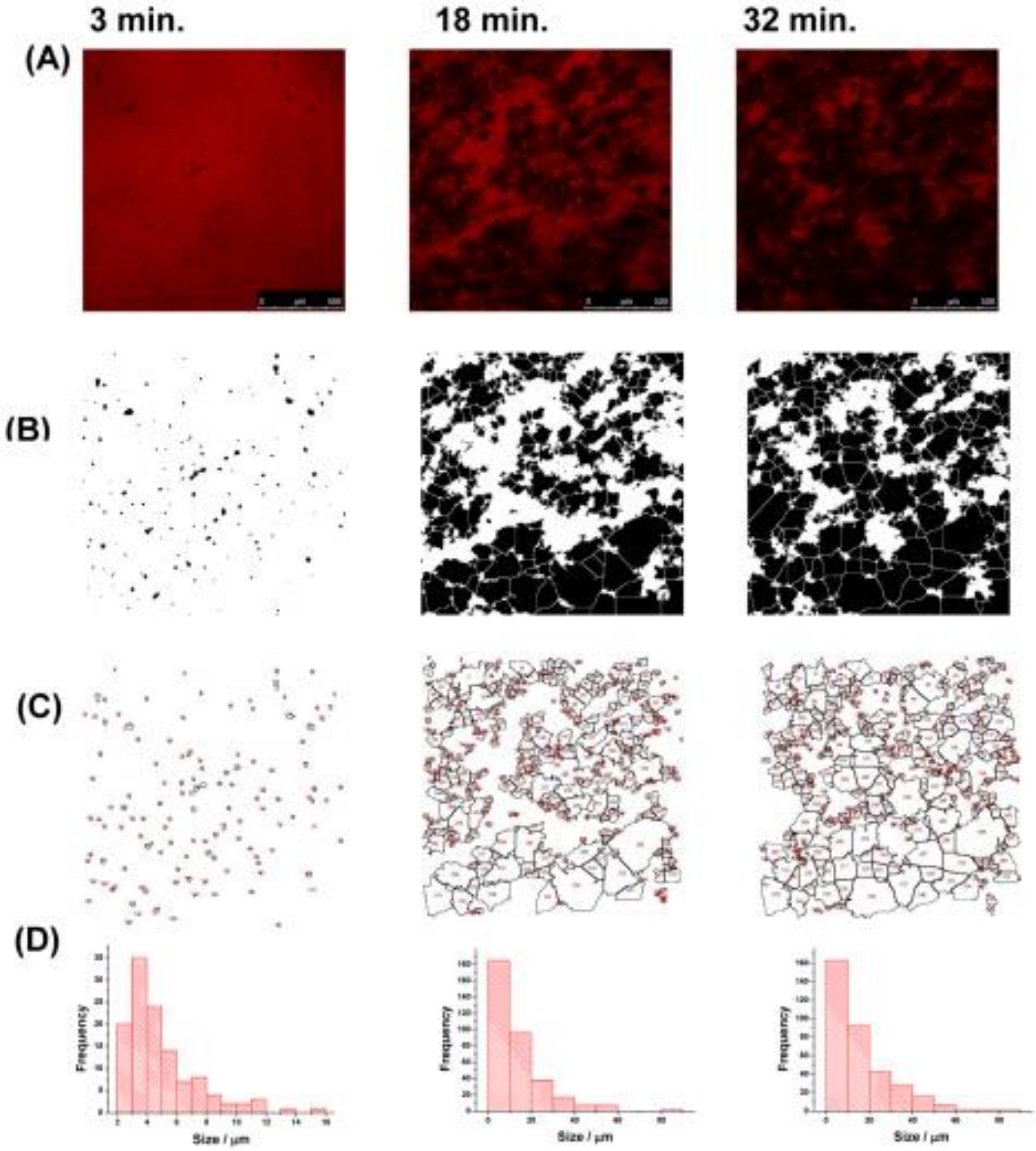


Figure 115. The effect of the time on the flocculation of asphaltenes observed using fluorescent microscopy (Franco et al. 2013).

7.8.3 Bitumen solubility parameters – BISOM titration

In this case three bad solvents of bitumen are used as titrants of the bitumen dissolved in good solvent toluene, namely 2-ethyl-1-hexanol, MEK or iso-octane (P. Redelius 2000; C. Hansen 2007).

The technique was developed based on the principles of Hansen Solubility Parameters, using solvents, which identify solubility of bitumen in solvents of various polarity (C. Hansen 2007). This assures that the focus is a bit deflected from only asphaltenes stability and some attention is given to the maltene fractions, as well as to the modifiers (such as polymers). The original Heithaus titration was focusing on the precipitation of asphaltenes from crude oil, to help with the problem of precipitation of the solids in the pipelines used for transporting of the crude. The BISOM titration is more focused on internal stability of bitumen, providing more complete picture about the material (C. Hansen 2007).

The method was not developed further from the publications in 2007. It was suggested, that including more than three solvents and including the concentrations of the bitumen in solvents, would increase the understanding of the bitumen stability and composition.

The HSP of the mixture of solvents can be similar as a HSP of a different solvent (Abbott, Hansen & Yamamoto 2015). This is a basis for development of cheap solvents for the paint industry (C. Hansen 2007). The Figure 116 presents a calculation of Hansen Solubility Parameters calculated for the blends of two solvents, example toluene-acetone and toluene-heptane. In the straightforward original approach, a set of 42 solvents (some of them rather toxic) were used to conduct the whole HSP evaluation. The availability and the upkeep of such chemical cabinet is expensive for a laboratory. In order to save space, money and to increase safety in the laboratory, the use of 3–4 solvents blended at differing ratios can be used as a surrogate.

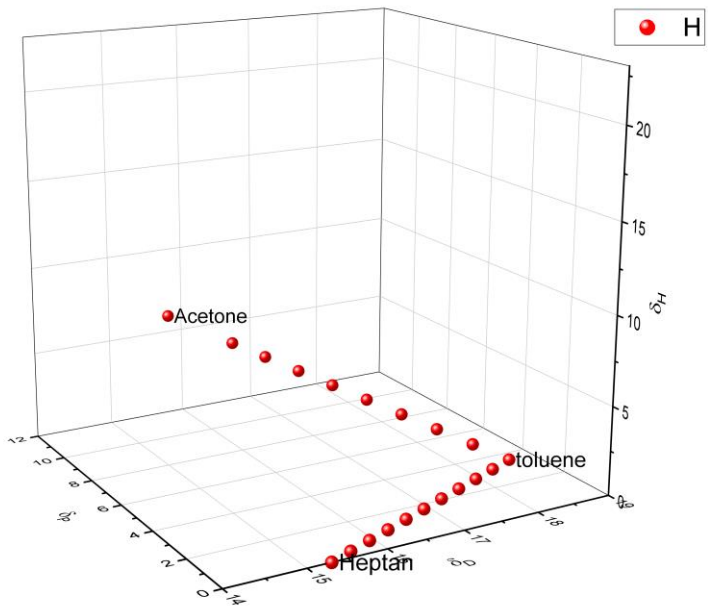


Figure 116. The explanation of the Hansen Solubility Parameter for a blend of two solvents, demonstration for acetone-toluene and heptane-toluene blends at a concentration difference every 10vol%.

The method to calculate the HSP for the blend of solvents (C. Hansen 2007), or so-called designed solvent, is described by the below equations:

$$\delta_{P\_blend} = \delta_{P\_sol1} * \frac{V_{sol1}}{V_{sol1}+V_{sol2}} + \delta_{P\_sol2} * \frac{V_{sol2}}{V_{sol1}+V_{sol2}} \quad (19)$$

$$\delta_{D\_blend} = \delta_{D\_sol1} * \frac{V_{sol1}}{V_{sol1}+V_{sol2}} + \delta_{D\_sol2} * \frac{V_{sol2}}{V_{sol1}+V_{sol2}} \quad (20)$$

$$\delta_{H\_blend} = \delta_{H\_sol1} * \frac{V_{sol1}}{V_{sol1}+V_{sol2}} + \delta_{H\_sol2} * \frac{V_{sol2}}{V_{sol1}+V_{sol2}} \quad (21)$$

where

V_{sol1} is the volume of solvent 1
V_{sol2} is the volume of solvent 2
δ_{P_blend}, δ_{P_sol1}, δ_{P_sol2} are polar cohesion (solubility) parameter of blend, solvent 1 and solvent 2, respectively
δ_{D_blend}, δ_{D_sol1}, δ_{D_sol2} are dispersion cohesion (solubility) parameter of blend, solvent 1 and solvent 2, respectively
δ_{H_blend}, δ_{H_sol1}, δ_{H_sol2} are hydrogen bonding cohesion (solubility) parameter of blend, solvent 1 and solvent 2, respectively.

Following this idea, the addition of "bad" solvent of bitumen which creates a homogenous mixture with toluene, allows one to test the solubility sphere contour for the binder (Figure 117).

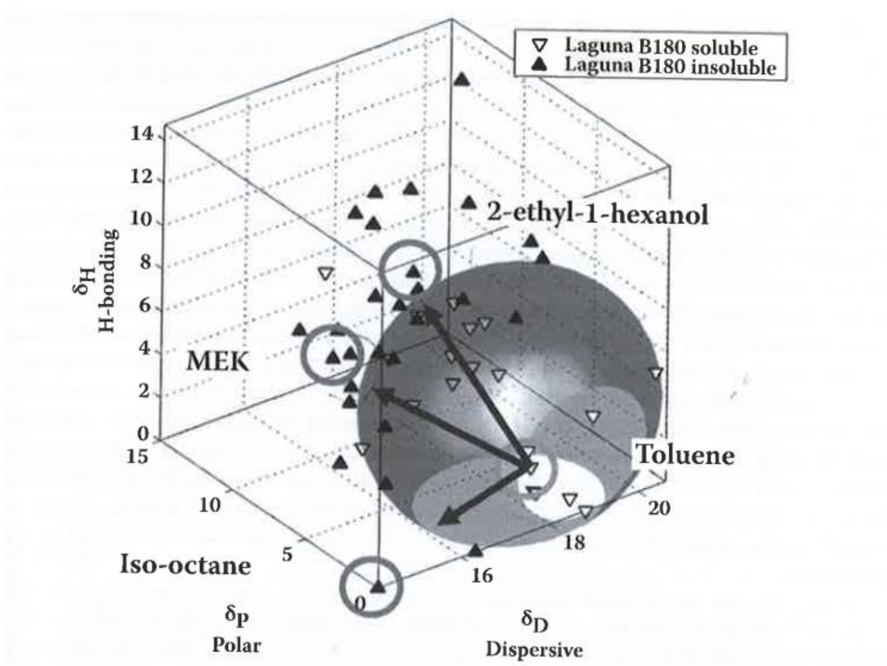


Figure 117. The idea behind establishing contour of the solubility sphere using BISOM titration (C. Hansen 2007).

The only downside of the approach for stability of the binder is the concentration of bitumen in the blend of solvents changing with the addition of the titrant (see Figure 114 in which precipitation of asphaltenes depends on the concentration of bitumen in solvent blend). Combining the automatic Heithaus (bitumen at multiple concentrations) titration with the BISOM concept of using multiple solvents to titrate the bitumen sample would be the next logical step in the development of the method.

Performing BISOM titration on 2–3 concentrations of sample could provide a better information about the bitumen. However, if one is interested in just distinguishing between the different binders, finding the right concentration which assures good differentiating possibility between the materials should be sufficient. Knowing which direction in solubility parameter is more interesting (see

Figure 118) – the one related to presence of polymer or the one related to presence of aromatics/maltenes, one extra solvent could provide enough of the information.

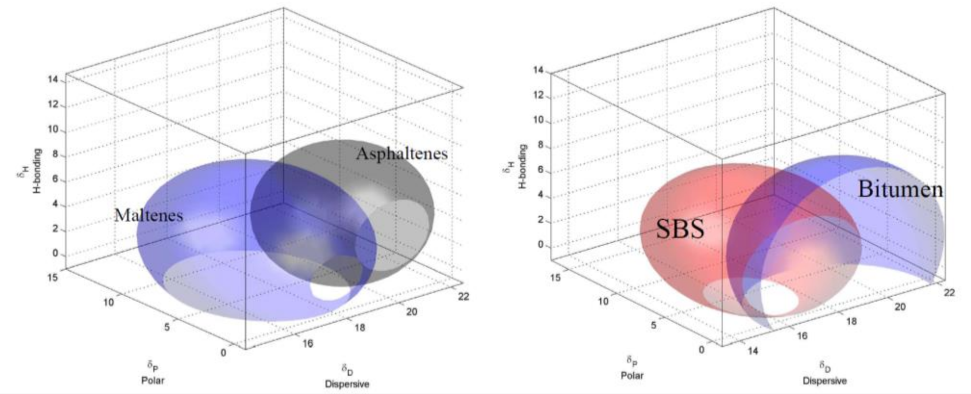


Figure 118. Solubility spheres for binder components such as maltenes, asphaltenes and SBS polymers (P. Redelius 2009).

The onset of instability can be measured also with the ATR probe, or fluorescence probe or UV-probe submerged into the titrated solution. Alternatively, the titrated solution can be pumped through separately standing spectrophotometer such as proposed for automated Heithaus titration by Pauli (Pauli 1996). The least technologically advanced analytical approach, but easiest to incorporate in any laboratory, is staining the filter and observing if the rings form on the stain – as this indicates instability and precipitation of particles from solvent.

Additionally, the effect of temperature (at least in the range between 5–40 °C observed in field) could be an interesting addition to understand the reasons of failure and loss of stability in some of the bituminous products (Zubair, Ramasamy & Hilmi 2015).

7.8.4 Content of Visbroken residue – titration in iso-octane

7.8.4.1 The test method

The altered chemical makeup of bitumen can be determined by so-called Heithaus titration using a specific solvent, in this case iso-octane (Dasek et al. 2017). It has been established that during the visbreaking the content of the asphaltenes increases and sample becomes more sensitive to the interaction with saturates.

Because visbroken residue is less stable in alkane solvents than atmospheric residue, the one component titration is providing enough information. A titration with isooctane of the bitumen sample dispersed in toluene was demonstrated as sufficient in evaluation of the potential presence of the visbroken residue (Dasek et al. 2017). A caution is advised in case some other additives are present, as the method was developed only on non-modified binders.

The method uses 1 g of bitumen sample, dispersed in 10 ml of toluene. The isooctane is added in portions of 1 ml addition every 30 s. After 30 s, the droplet of the solution is stained on the filter paper. If the series of rings appears – this indicates instability (precipitation of particles). The volume of isooctane at which the rings are observed is recorded. The volume of isooctane needed until the instability is observed divided per initial volume (10 ml) is defined as Floculation in Toluene Index (FTI). Therefore, the lower the FTI, the less of the alkane was required to destabilize the binder, thus more likely the sample contains visbroken residue.

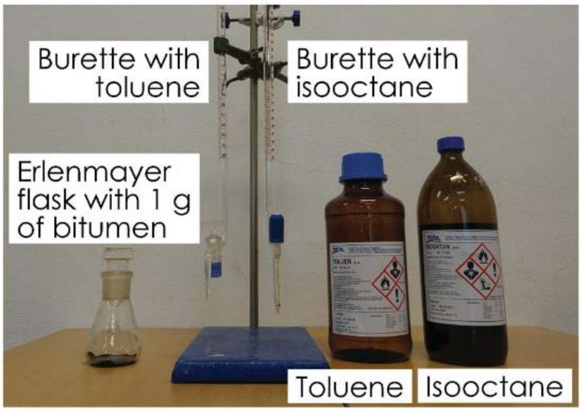


Figure 119. Aparatus used for the single point precipitation titration (left) and the example of test results (right) (Dasek et al. 2017).

The apparatus necessary for the test comprises of basic laboratory glassware. The handling of the toluene and isooctane may be the more challenging aspect, requiring the station with good ventilation.

7.8.4.2 Evaluation of FTI using samples

The set of references was build based on the two bitumen grades: 50/70 and 70/100, into which Vacuum Flashed Visbroken Residue (VFCR) was added at concentrations of 0, 10, 20, 30 and 40%. Against this reference the set of 15 paving bitumens available on the Czech market were tested using the same method (Dasek et al. 2017).

Samples were aged using the 3xRTFOT aging procedure to achieve similar rheological changes in binder as after the PAV treatments (Siddiqui & Ali 1999). Samples before and after aging were characterizes using the FTI, DSR and BBR to evaluate the susceptibility of the binders to thermo-oxidative aging.

The higher the visbroken residue content in the binder, the lower the FTI was recorded. The changes between the 30 and 40% of added VFCR to the reference binder were below the analytical range of the methodology (it is assumed at 2 ml of solvent addition). Nevertheless, the differences between reference and binder modified by more than 20% of VFCR are evident and distinguishable.

One of the evaluation criteria used in the analysis based on FTI was increase of the Softening Point ($\Delta R\&B_{3xRTFOT}$) after 3xRTFOT (similar to RTFOT+PAV procedure). The procedure adapted by authors assigned the well performing and stable bitumen samples to be characterized by $\Delta R\&B_{3xRTFOT}$ below 15 °C. Clearly, binders with significant amount of VFCR added, as well as some commercial binders with the very low FTI, increased in $\Delta R\&B_{3xRTFOT}$ above 15 °C, with the highest ones above 18 °C.

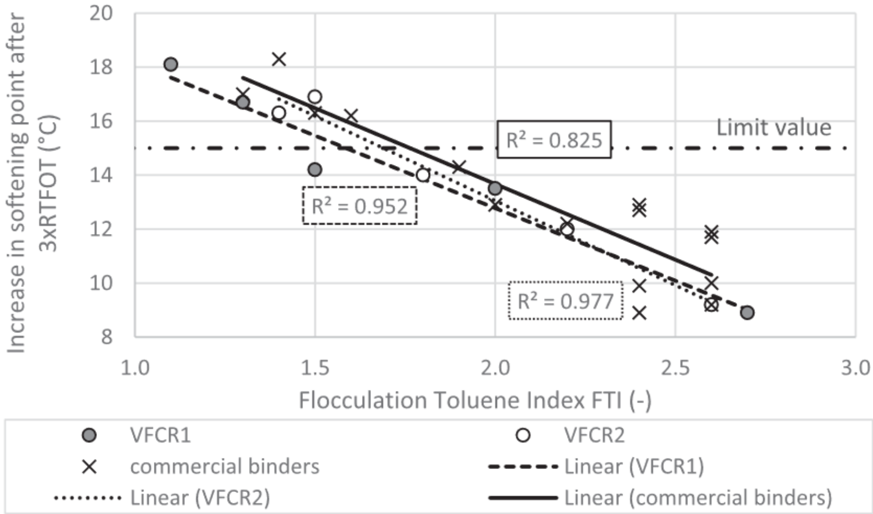


Figure 120. Commercial bituminous binders and VFCR concentration series – correlation between increase in softening point values after 3xRTFOT ageing and FTI values (Dasek et al. 2017).

Interestingly, the samples containing VFCR up to 10% were not significantly affected in terms of their rheology or their susceptibility to thermo-oxidative aging at all. The differences between reference and VFCR modified binders were not apparent after the first RTFOT. The differences between binders become observable after prolonged aging for both references and tested commercial samples.

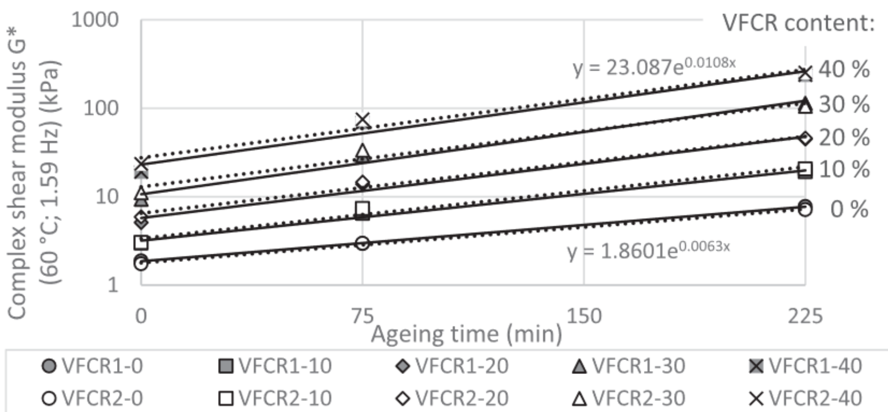


Figure 121. VFCR concentration series – relation between the exposition time of ageing and complex shear modulus at 60 °C of bituminous binders (Dasek et al. 2017).

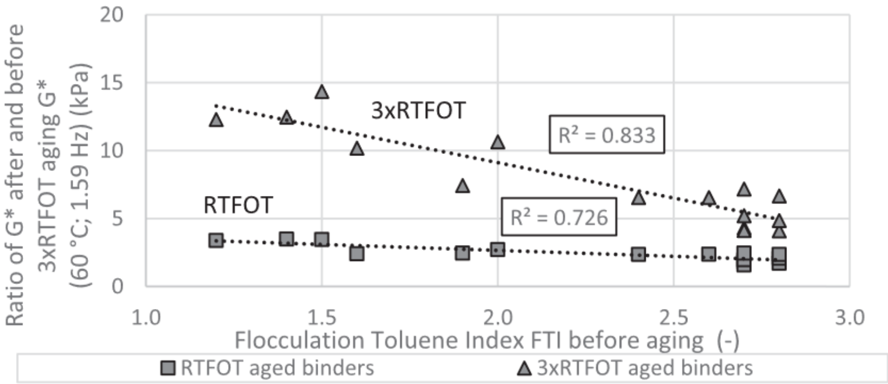


Figure 122. Commercial bituminous binders – relation between the ratio of complex shear modulus G values after ageing and before, and FTI value of binders in various stages of the ageing process (Dasek et al. 2017)*

One of the observations of this research indicated that small addition of the VFCR may in fact be positively affecting the low temperature resistance, as investigated with BBR.

The effect of the high VBR content was implied as negative from field observations, but the explanation of the mechanism of failure was not attempted by the authors. It is also important to note that only samples of one grade of bitumen were tested, and there is a missing information about softer grades used typically in Finland, as well as in respect of comparison between grades. Such is suggested to be performed on old and newer binders entering the market.

7.9 Acid and Base number

During the ASTO research program, three of the tested bitumen sources were investigated using so-called base and acid number. The results of the test and the meaning of the findings are described in BITU2020 – Part 4 report file. In summary, there was a link between moisture susceptibility of the asphalt concrete and the acidity and basicity of the binder, when in contact with acidic aggregates. Some of the adhesion promotors were enhancing basicity and some acidity of the binders. It is postulated that knowing this characteristic of the binder at hand should be an extremely useful tool during the first choice of adhesion promotor type.

The ASTO report indicates that the standard ASTM D664-81 was used (Pylkkänen & Kuula-Väisänen 1990). Currently the ASTM D664-07 ("Acid Number of Petroleum Products by Potentiometric Titration") covers only the acid number, while ASTM D4739-08 ("Standard Test Method for Base Number Determination by Potentiometric Hydrochloric Acid Titration") is an alternative to the part of the ASTM D664-81 discussing basicity number. The European standard corresponding to the current version of ASTM D664-07 is SFS EN 12634 ("Petroleum products and lubricants. Determination of acid number. Non-aqueous potentiometric titration method"), but a different solvent mixture is used in it (toluene, isopropanol and dimethylsulfoxide).

7.9.1 Principle

Nevertheless, the principle of the methods is similar. The sample of 1 g dissolved in a mixture of toluene and isopropanol (and dimethylsulfoxide in case of SFS EN 12634). The titration with known solution of potassium hydroxide (KOH) is conducted and evaluated potentiometrically (using the calibrated pH meter). Typically, the end of acid number titration happens when the value of pH = 13.5 is recorded or when the titration curve bends towards almost vertical shape.

In the petroleum – both strong and weak acids can be present, thus the titration can result in curve with multiple bend points. In such case the reported value can be either a total acid number, or then separately strong and weak acid number. The concept is explained on the example provided in the D664-07 (Figure 123). The curve A is taken from the reference in which there is no dissolved sample. In the case of sample B, in which no clear sharp inflection in the curve is visible the end of titration is defined as the pH obtained with the used reference buffer aqueous solution (in this case pH = 11.5). The sample C, containing weak acids is titrated until the sharp inflection is observed, and the acid number is read from the curve region where the line is almost vertical to the axis. The total acid number is calculated as a difference in the amount of the KOH solution used for a sample, and for the reference.

In the sample D in which both weak and strong acids are present the titration is performed in a different way. The solution is titrated with KOH solution until the pH = 4 and the turning point is recorded. Then the blank solution is titrated with 0.1 mol/l HCl until the pH = 4.

The strong acid number is calculated as volume of KOH times its concentration and volume of HCl times its concentration.

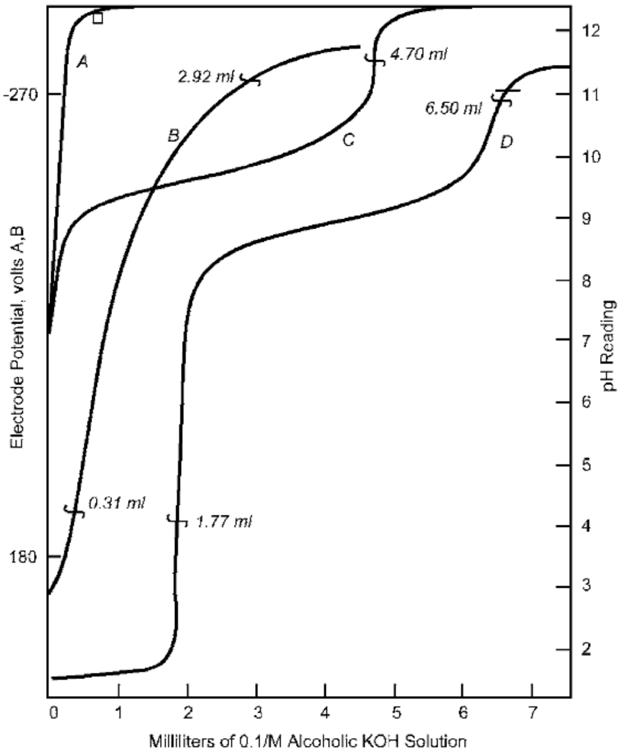


Figure 123. Example of the acid number titration curve, where A is reference liquid without sample, B is the sample without the clear sharp inflections, C is a sample containing weak acid, D is a sample containing both weak and strong acids.

7.9.2 Difference in ASTO

The description of the procedure in the ASTO report, indicates that the sample is titrated with KOH solution or with HCl solution. The end points of the titration are defined either as the clean inflection points in the curve or the pH of the reference solutions used (pH = 4 or pH = 13.5). Then the two curves are joined and the HCl amount recalculated into the KOH amount. Both basicity and acid number are given in the unit of mg/g KOH.

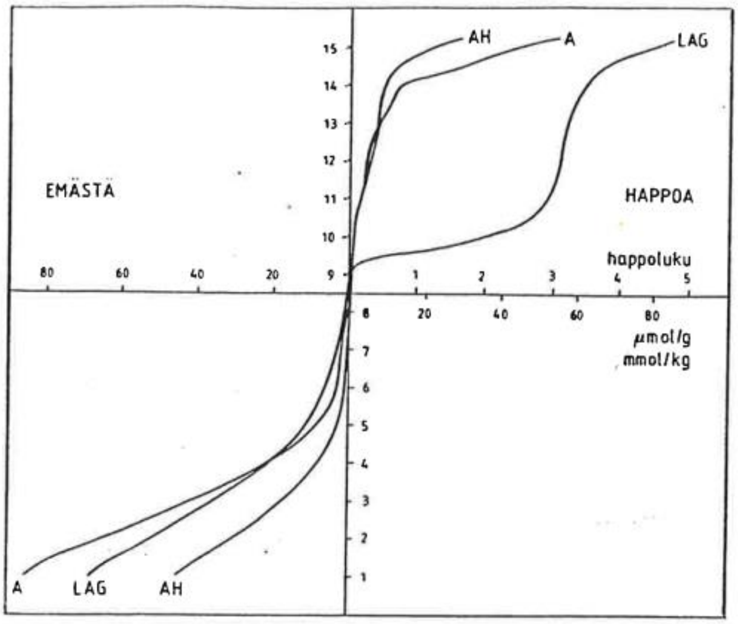


Figure 124. The titration curve of bitumen A, AH and LAG (Pylkkänen & Kuula-Väisänen 1990).

Apparently, the three tested bitumens were differing during the titration. The difference is more visible looking at the curves than on the actual number, which may be misleading due to the unit used. Interestingly, the addition of amine or tall oil derived modifiers was detectable in the acidicity and basicity curves during titration.

It is suggested hereby that this simple technique should prove interesting to determine the presence of potential modifiers in the products delivered to the asphalt plant. The first step to choose the correct adhesion promotor is a knowledge of the character of the material that will be used. As is discussed in BITU2020 – Part 4, there is a limiting value for example for the surfactant's concentration.

Additionally, it would be essential for quality control purposes to establish if the acidic and basic number change permanently in the material. To establish if the adhesion promotor was in fact used, the basicity number could be used in combination with FT-IR type analysis. Some amines express a significant peak in FT-IR, but that peak is relatively less intensive than other peaks typically observed in the bitumen. It is interesting if the basicity number would be more sensitive to the presence of amines as modifiers of the bitumen. Also, would aging affect to the acidity and basicity number.

Multiple reports on addition of adhesion promotors into the asphalt concrete focus on the fact of improving the resistance to moisture damage or reducing the temperature of production.

However, a lot of questions remain unanswered on the subject of the amine's life-cycle in the asphalt concrete production. Some of the products on the market are water soluble and some are volatile with low boiling point. The concerns from the contractors and researchers are that the material is potentially either evaporating during production or washes away during the performance. No tests for presence of adhesion promotor are made from the binders extracted from RAP. There is little knowledge on how the addition of the adhesion promotor, e.g. amine based would influence the behavior in the second cycle of RAP reuse. Until we learn how to distinguish the presence of the additive in the material in a rapid and simple way, some of the questions will remain unanswered.

Testing the basicity of the binder could prove:

1. Indicative of the need for amine based additives
2. Indicative of the presence of amine based additives
3. That material is more susceptible to stripping if its acid number changes dramatically during oxidation

Testing of the acidity of the binder could:

1. Indicate the compatibility with the certain type of aggregate
2. Indicate the need to use the basicity increasing adhesion promotors when acidic aggregates are designed for use
3. Indicate the presence of rejuvenators with high acid number in the binder extracted from RAP
4. Indicate the necessary dose of basicity increasing adhesion promotors to assure good adhesion

7.10 Volatile matter analysis

Bitumen has a distinctive smell, and often the differences in the product are recognized by professionals in field by "different smell". The olfactory recognition of products is utilized in industries such as perfumeries, wine production or tea composition. However, each one of such specialistic applications requires a specially trained person for recognition. Using the principles related to why we smell things differently, the attempt to create artificial nose for the bitumen was approached.

7.10.1 Artificial Olfactory System (AOS)

7.10.1.1 Recognizing bitumen origin/refinery

In the original study by (Autelitano, Garili et al. 2017) the researchers investigated a possibility to distinguish between binders using the Artificial Olfactory Systems (AOS), also referred to as artificial noses. In this study an artificial nose was created by combination of 12 gas sensors. Each of the gas sensors has a sensitivity to a different type of compounds and their specific sensitivity is provided in Figure 125.

The initial tests were performed on bitumen kept at 22 °C in the closed bottle. The gas emitted from the sample was then analyzed. The gas itself is then heated up by the equipment in the olfactory chamber and is analyzed at higher temperature (46 °C).

Position	Model	Application	Typical detection range	Features
SF3A	TGS813	Combustible gas detection	General combustible gases (500–10,000 ppm)	For wide range of gases
SF11A	TGS842		Methane (500–10,000 ppm)	Low sensitivity to interfering gases
SF7A	TGS826	Toxic gas detection	Ammonia (30–300 ppm)	High sensitivity to ammonia
SF6A	TGS825		Hydrogen sulphide (5–100 ppm)	High sensitivity to hydrogen sulphide
SF5A	TGS822	Solvent vapour detection	Alcohol, Toluene, Xylene, etc. (50–5000 ppm)	High sensitivity to alcohol and organic solvent
SF5A1				
SF8A	TGS830	Halocarbon gas sensor	R-113, R-22 (100–3000)	High sensitivity and selectivity to various CFCs, HCFCs
SF9A	TGS831		R-21, R-22 (100–3000 ppm)	Quick response to R-21, R-22
SF10A	TGS832		R-134a, R-22 (10–3000 ppm)	High sensitivity and selectivity to R-134a and R-22
SF2A	TGS800	Air quality control	Air contaminants (1–100 ppm)	High sensitive detection of contaminants in air
SF12A	TGS880	Cooking control	Volatile gases and water vapour from food (30–30,000 ppm)	Total gas detection in cooking process
SF13A	TGS882		Alcohol vapour from food (50–5000 ppm)	Alcohol gas detection in cooking process

Figure 125. The characteristics of the initially used sensor array after (Autelitano, Garili et al. 2017).

The researchers tested three bitumens of the penetration grade 70/100 obtained from visbreaking process. Two of the binders were coming from the same refinery, but the binder A1a and A1b were the products of refining two different batches of crude oil from the Mediterranean Sea. The bitumen A2 was a product coming from a different refinery and the crude oil source was the Arabian crude oil. More information about the binders can be found in the original articles (Autelitano, Garili et al. 2017; Autelitano & Giuliani 2018; Autelitano, Garilli & Giuliani 2019).

The researchers demonstrated that the binders can be differentiated between, especially the group A1 and A2. The differentiation between A1a and A1b was more challenging.

It is worth noting that the tests were conducted on the cold binder samples. As was discussed in previous sections, the oxidation of binders of varying origin results in a different thermo-oxidation pattern. This is visible in a differing infrared spectrum of the binder after exposure to oxygen at high temperatures, but also is proposed to result in varying concentration of the gases flowing out of the system as a byproduct of the oxidation. Depending on the crude oil origin and refining process, as well as the temperature and pressure of oxidizing gas, the concentrations will vary and give different signal response in such AOS solution.

7.10.1.2 Using commercially available e-nose solutions

As a follow up study (Autelitano, Garilli & Giuliani 2019) the researchers reported investigation of the same products using commercially available e-nose solutions, namely ISE Nose 2000 (Soatec) and CyraNose 320™. The ISE Nose operating principle is explained in detail in (Autelitano, Garili et al. 2017) and CyraNose 320 in (Autelitano & Giuliani 2018), but the latter AOS contains 32 gas sensors.

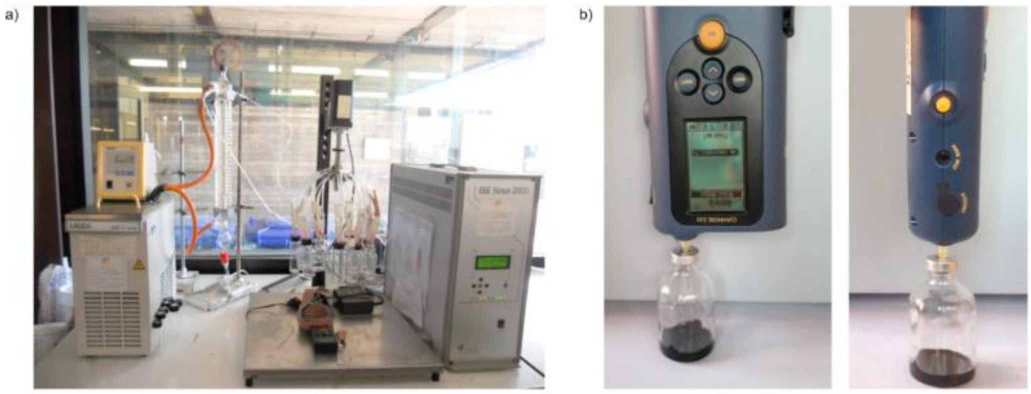


Figure 126. An example of the commercially available AOS used in the research projects after (Autelitano, Garilli & Giuliani 2019).

An ability to distinguish between bitumen A and B was confirmed. The differences between the batches of bitumen A are not as apparent when the three bitumen are plotted together. However, as researchers have argued, when both batches of A are compared with each other, the distinguishing is plausible (Autelitano, Garili et al. 2017).

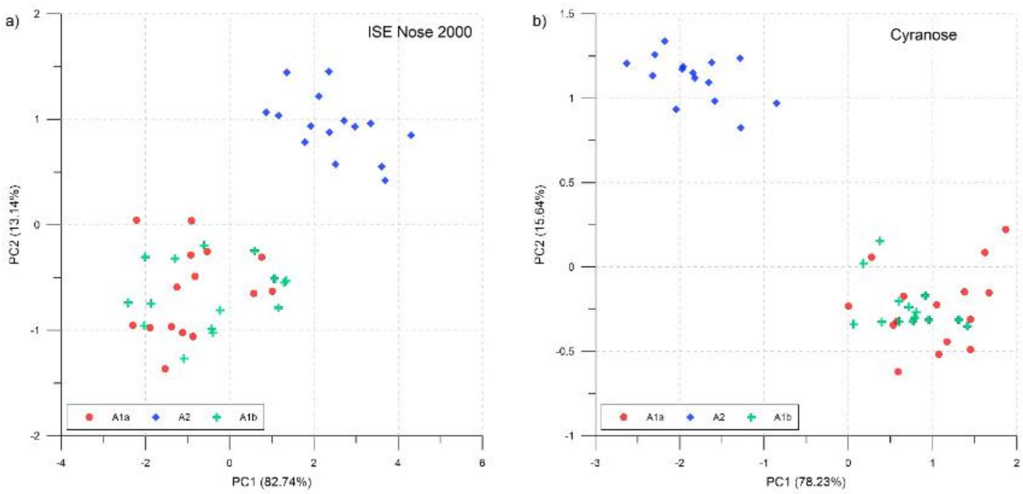


Figure 127. 2D-PCA plot elaborate on measurements performed with two e-noses, a) ISE NOSE 2000 and b) Cyranose (Autelitano, Garilli & Giuliani 2019).

However, using the experience reported, the type of the used AOS will create different Principle Component Analysis correlations. Therefore, the calibration and own database collection is necessary. It is perhaps envisioned that the technique is good for the companies internally to distinguish between the sources of the binder supplied to the asphalt plant. Most likely the response will also depend on the gas sample collecting (venting) system applied, as well to the size and type of the bitumen storage tank. The further investigations indicate that the temperature at which the gases are emitted plays a role in terms of signal recorded by the AOS (Autelitano & Giuliani 2018).

7.10.1.3 Recognizing additives at various temperatures

The same research group investigated if the e-nose type recognition could be applied to distinguish between different modifier in bitumen. For this purpose, two bitumen derived in two different plants, but each one using Arabian crude oil during refining, were used – B1 (Pen25 °C = 84 0.1 mm; R&B = 46.4 °C) and B2 (Pen25 °C = 86 0.1 mm; R&B = 46.2 °C). The additives evaluated are listed and described in Table 14.

The effect of those additives was tested using the TLC fractionation, and results are presented in Table 15. However, the allowed repeatability and reproducibility of the test itself (IP 469 2006) is not allowing the analyst to perform the differentiation of the materials beyond reasonable doubt. The researchers performed also the rheological analysis of the blends and observed various effect of the additives, which stays in line with what was previously discussed within this report.

The researchers then focused on differentiating between additives themselves at elevated temperatures (110 and 130 °C) consistent with the asphalt production, using the e-nose solution. The recorded signal of the materials from multiple used gas sensors was then fed into the Machine Learning algorithm and a relatively good separation of the products was achieved with principal component analysis (PCA), except for the SW and MW (Figure 128).

Table 14. The list of modifiers tested by the e-nose solution (Autelitano & Giuliani 2018).

Code	Type of modifier	Information provided for product	Concentration by weight of bitumen [%]
CA1	Adhesion enhancer	Ofamine derivative	0,5
CA2	Adhesion enhancer	Mixture of vegetable origin enhancer substances	0,75
OSA	Odour suppresant	Complex mixture of hydrocarbons, alcohols and essential oils	0,017
MW	wax	semi-synthetic refined Montan-type wax, derived by solvent extraction of fossil deposits of lignite-rich or brown coal vegetable substances, blended with fatty acid amide	3,0
SW	Hard wax	Synthethic hard wax, by product of Fischer-Tropsch process (FT)	3,0

Table 15. The result of IP 469 fractionation analysis of the bitumens modified with additives specified in Table 14 (Autelitano & Giuliani 2018).

Blend	Saturates [%]	Aromatics [%]	Resins [%]	Asphaltenes [%]
B1	5.2	52.6	26.6	15.6
B1-CA1	5.4	46.1	29.9	18.6
B1-CA2	5.4	50.5	27.2	17.0
B1-OSA	5.2	47.7	27.3	19.7
B1-MW	5.9	45.1	27.7	21.3
B1-SW	5.1	48.3	26.4	20.2
B2	2.3	55.3	25.5	16.9
B2-CA1	2.5	48.9	27.6	20.9
B2-CA2	2.8	47.0	28.6	21.7
B2-OSA	3.0	47.8	30.5	18.7
B2-MW	2.4	47.3	26.6	23.9
B2-SW	2.8	47.1	27.2	22.9

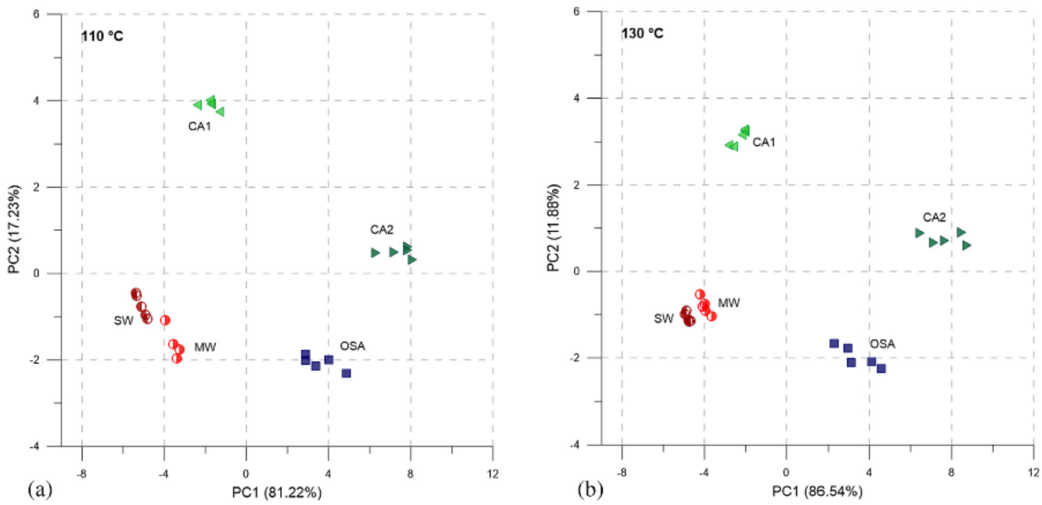


Figure 128. Two-dimensional PCA plot for each additive described in Table 14 at (a) 110 °C and (b) 130 °C (Autelitano & Giuliani 2018).

The researchers then attempted to differentiate between blends with additives at similar temperatures 110 °C and 130 °C, but it turned out that the signal is not differing significantly at those temperatures. The reason perhaps may originate in the very low concentration of the additive, indicating the signal of the additive not being extremely influential on the odor emissions. However, the researchers observed a pseudo-horizontal displacement in the signal as a result of the temperature to which the sample was exposed. The samples were also investigated at 160 °C and 200 °C. It was at the highest tested temperature that the differentiation between the e-nose recorded signals was identified more clear by the Machine Learning Algorithm (Figure 129) (Autelitano & Giuliani 2018).

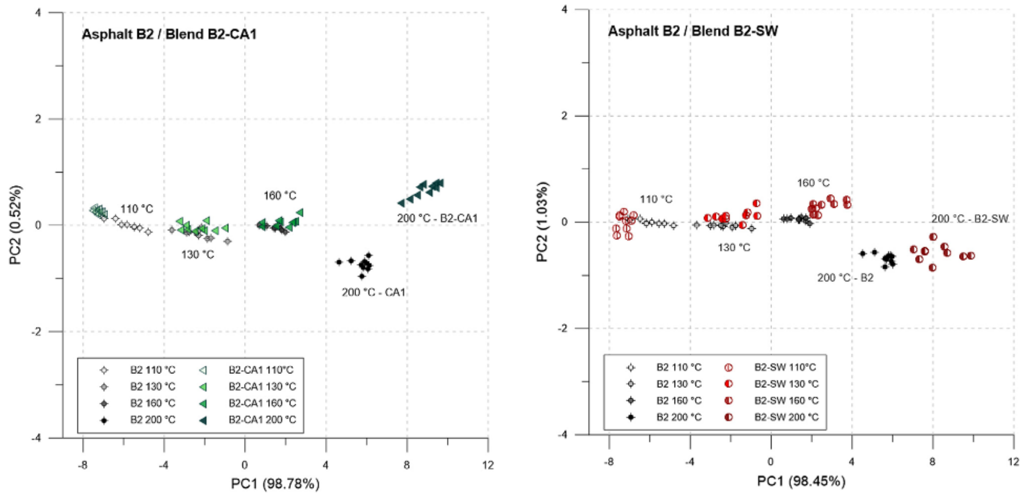


Figure 129. The effect of temperature on the two-dimensional PCA plot of Asphalt B2 with and without additive CA1 (a) and SW (b) (Autelitano & Giuliani 2018).

The temperature to which the sample is heated affects the progress of oxidation, but also the rheology of the sample and thus diffusion of oxygen into the sample and diffusion of gas particles out of the sample. Even tough, the differentiation between various used additives was successful using the commercial CyroNose 320, the differentiation between binders modified with the additives was more challenging. The clear separation in PCA signals was achieved only above 200 °C, which is hopefully not the temperature of transport or storage of the binder but can be used for testing purposes. Therefore, the recognition of the modifier presence is perhaps more reliable using other techniques mentioned before.

However, the procedure could be implemented into multilevel analytical process of binder recognition in the laboratory or in a special separate sampling/monitoring unit. The distinguishing between various sources or origins of the binder using the AOS is extremely promising approach for the problem investigated by the BITU2020 project. Collection of the binder sample from the tank or from the transporting truck are typically discouraged in the industry due to the health risks associated with hot bitumen handling.

Usually, the samples representative for the tank would be provided by the supplier. However, the mismatch between the sample and tank may occur. The purpose of the quality control in the company would be to assure that the product delivered is the product for which the traditional QA reports exists. The sudden change of the signal in AOS is envisioned to prompt the quality control in the company to retest the material and to confirm that it meets the expectations of performance.

Using the AOS, the rheological data and field performance data – the machine learning can be facilitated to create predictive models of performance for various bitumen types. At the moment, the bitumen liquid and emitted gas sampling solutions are the challenge for the quality control in the asphalt plant, but hopefully those are only engineering challenges, which can be easily overcome.

7.10.2 Gases during heating

In the previous studies on the evolution of the bitumen spectra in-situ during heating, it was observed that not only the spectra of bitumen changed with exposure to oxygen (Makowska, Hartikainen & Pellinen 2017). The spectra had varying signals coming from potential small molecular gaseous compounds such as hydrogen sulfide, carbon dioxide and water. Those compounds could be side products of the oxidation reaction, which was proposed for the analyzed bitumen based on the time resolved changes in the spectra.

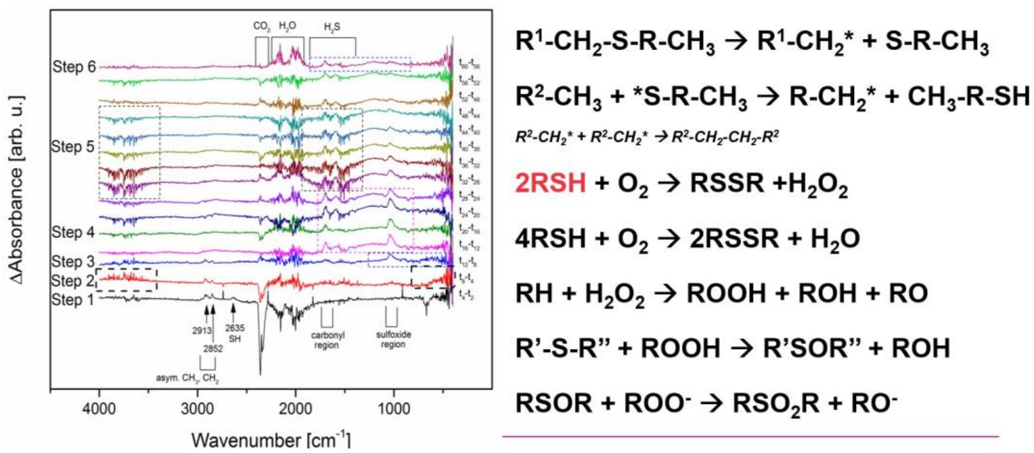


Figure 130. The time resolves changes in FTIR spectra collected every 4 minutes from bitumen heated to 163 °C in the presence of air (Makowska, Hartikainen & Pellinen 2017).

It is plausible that the initial composition of the bitumen may skew the reaction towards an alternative direction or path. In this case it is envisioned that a different time-resolved combination in the outflowing gases would be observed. Some of the plausible small molecular reaction products of oxidation and exposure to high temperatures in absence of air include water, CO, CO_2, H_2S, SO_X, NO_X, NH_3 and volatile hydrocarbons.

In the anesthesia procedures, the outflowing gases from the patient's lungs are monitored for the presence of the anesthetic (ProMed Strategies, LLC under an educational grant from PHASEIN AB, Sweden n.d.). The method is allowing for determination of the changes in theater allowing for an extremely fast reaction from the doctors. Other methods, such as GC and NMR are time consuming at the moment and delay the response.

The use of the infrared analysis of the gases outflowing from the heated bitumen could answer the question on the origin of the binder. Due to the different chemical markup – aromaticity, sulfur content, nitrogen content and varying susceptibility to oxidation and decomposition in hot storage, the emitted gases in the tank should be different for different products. Incorporating in-plant monitoring system for the tanks after refill could create a possibility to recognize the differences in the content of sulphur and oxidation resistance. This information could then be further coupled with rheological properties of the binder as well as performance in field to create the database of responses and actions required for certain types of products.

Below are the typical gas infrared spectra reprinted from the NIST database to demonstrate the regions of contribution from each of the possibly emitted gases due to oxidation and decomposition. As it is visible, there is a possibility that the gases do not overlap and as a result that there could be a possibility of distinguishing the ratios of concentrations of them in the gas.

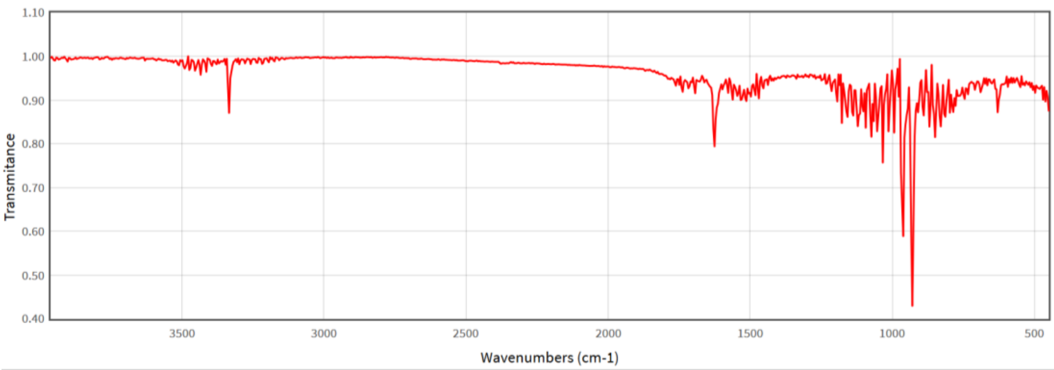


Figure 131. Example of the IR spectra of ammonia. Source: NIST.

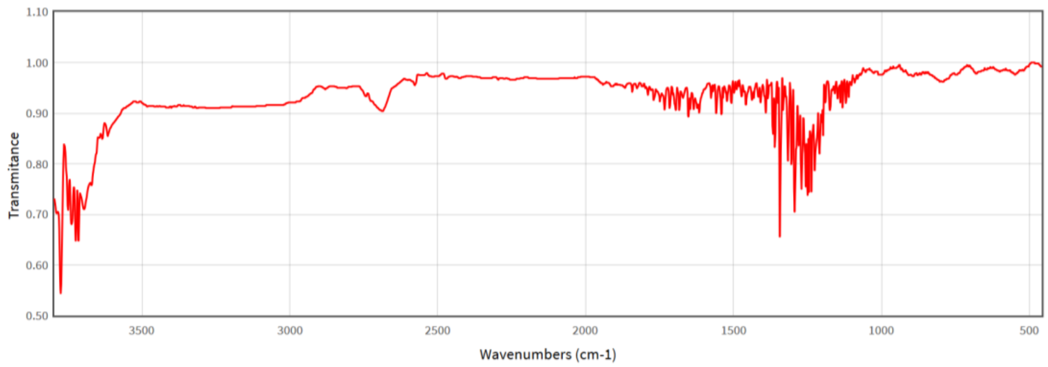


Figure 132. Example of IR spectra of hydrogen sulfide. Source: NIST.

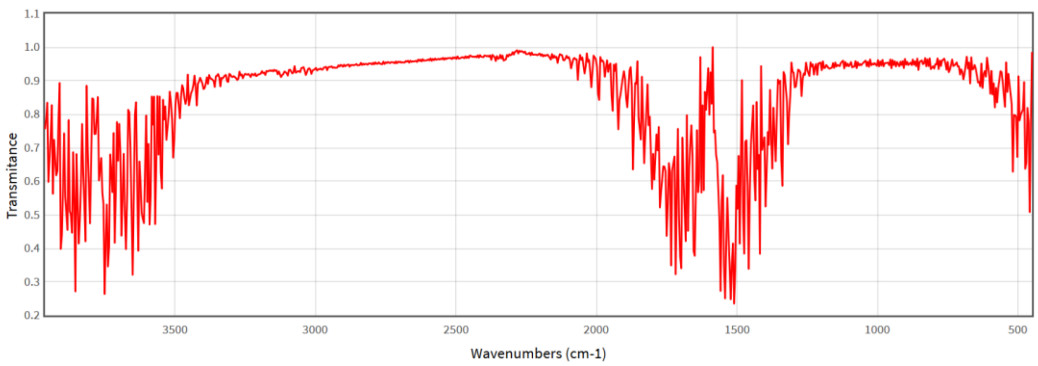


Figure 133. Example of the IR spectra of gaseous water. Source: NIST.

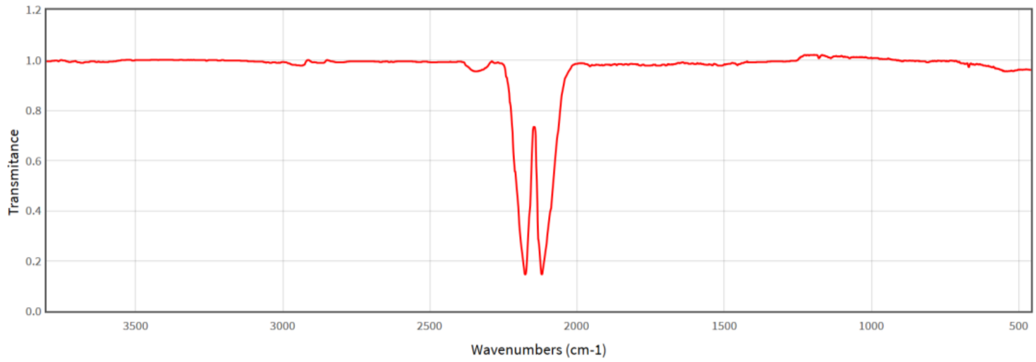


Figure 134. Example of the IR spectra of carbon monoxide. Source: NIST.

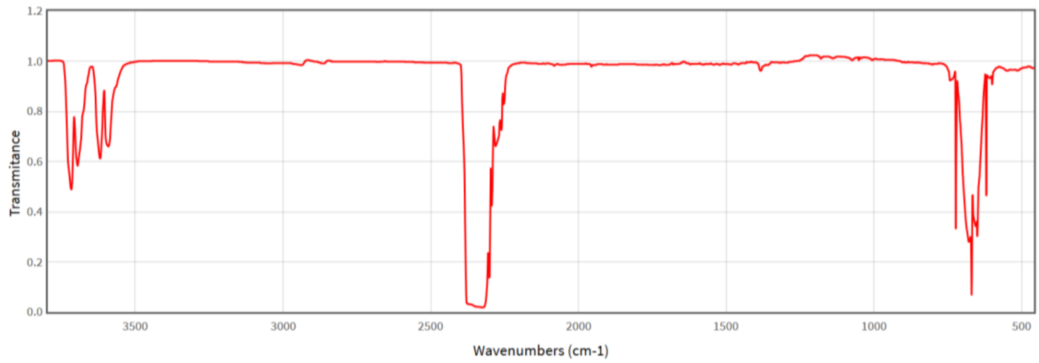


Figure 135. Example IR spectra of carbon dioxide. Source: NIST.

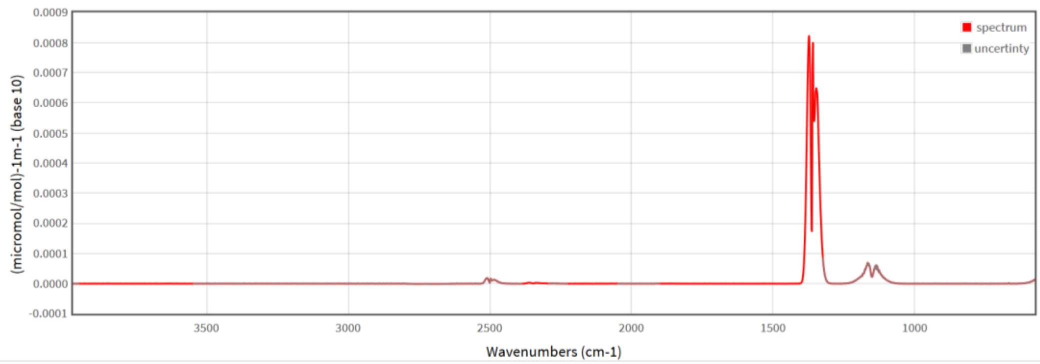


Figure 136. Example of the spectra of sulfur dioxide. Source: NIST.

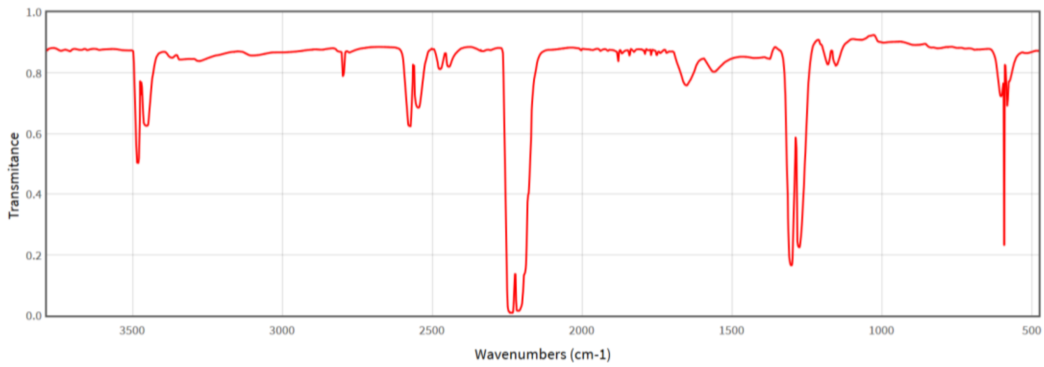


Figure 137. Example of the spectra of nitrous oxide. Source: NIST.

The envisioned system would investigate the evolution of gases in fresh bitumen during the controlled heating from 25 to 163 °C, as well as the continuous gas release at 163 °C in contact with air to investigate the mechanism of thermo-oxidative resistance, by looking at the gaseous reaction products.

The other application would be investigation of gases in the tank during the storage at set temperature in the absence of oxygen to distinguish between the products when they are stored in tanks.

7.11 Machine Learning based solutions

As was now discussed at lengths, the aging of bitumen or product recognition can be performed from spectroscopy, or rheological measurements, or many other chemical data. Coupling the large amount of information becomes a troublesome task for the operator. Providing multiple complex equations often is more of a threshold than a solution for the uptake into the industry. One of the interesting ways on how to utilize the large number of data coming from raw and aged bitumen as well as gasses eluted during aging, and linking it to rheological or physico-chemical performance is machine learning.

7.11.1 Definition of machine learning

Machine Learning (ML) is a set of computer programs that "learn" from data that is given to the programs. Learning in this context refers to adaptive improvement of performance of the algorithm even though the algorithm is not explicitly programmed to perform better. In other words, a machine learning algorithm has an initial structure, but the improvement of performance comes from the experience that the algorithm receives from the data.

The machine learning algorithms find patterns from the given data to construct models. These models are used by the algorithm to make predictions from new data. The basic process of an ML algorithm happens in four steps:

- First the algorithm is given data that it uses for learning.
- Next the algorithm produces a model from this data for making predictions.
- Third step is to test the model. The testing is made commonly on a separate testing set. The purpose of the testing is to verify that the model predicts accurately with other data than it was trained on.
- Last step is that the model can be used for predicting outcomes out of new data. (The MathWorks, Inc. (MATLAB and SIMULINK) Accessed: 14.5.2021; Mohammed, Badruddin Khan & Bashier Accessed 17.5.2021; Géron Accessed: 17.5.2021).

Machine learning algorithms (MLA) have some advantages compared to other computer algorithms:

- MLAs are used for problems that have lots of complex data;
- MLAs do not need hand-tuning or a long list of rules to function with complex data.
- MLAs can find solutions from data without human supervision.
- MLAs can adapt to new data (Géron Accessed: 17.5.2021).

7.11.2 Models that can be used – their purpose and possibilities

The MLAs can be separated into two categories: Supervised and unsupervised learning algorithms. The difference between these algorithms is that the supervised algorithms are given input data and output data. Unsupervised algorithms are given only input data.

Supervised algorithms function so that the input data is labelled with output data. Labelling means that the input attributes are paired with predetermined output attributes. The algorithms task is to find variables from the input data that predict the output. This way the algorithm makes a predictive model that can be used to predict the output for new input data (Kotsiantis 2007; Alloghani et al. 2020).

Supervised learning algorithms are divided into regression and classification algorithms. Difference in the two algorithms is the type of the output data. Classification algorithms predict discrete outcomes and regression algorithms predict continuous outcomes. Spam filter in email is an example of a classification algorithm. The algorithm classifies the mails into spam and non-spam with a model that it has constructed from previous emails that have been classified (Géron Accessed: 17.5.2021). An example of a supervised regression learning task would be predicting the age of a viewer watching a given YouTube video (Murphy 2012-08-24).

Unsupervised learning algorithms are not given any output data. The function of unsupervised learning algorithms is to find patterns, similarities and differences from the given input data without any given target values. Commonly used unsupervised learning algorithms are clustering algorithms. They are used to group the data into homogenous groups using patterns that exist in the data (Mohammed, Badruddin Khan & Bashier Accessed 17.5.2021) (Bonaccorso 2019). Unsupervised learning models can be used to pre-process data for supervised learning algorithms. Important unsupervised learning task are anomaly detection and dimensionality reduction. With anomaly detection techniques the algorithm is used to find defects or outliers from the dataset (Géron Accessed: 17.5.2021; Lynch, van Berkel & Frieboes 2017).

An application of MLAs is data mining that is also known as knowledge discovery from data. Data mining is a process of discovering patterns in large amounts of data. MLAs can find characteristics from the data that define the output variables. This information could help in finding new attributes from the data that are not apparent without the algorithm. Machine Learning algorithms can help humans learn this way from large data (Géron Accessed: 17.5.2021; Han, Pei & Kamber 2011).

7.11.3 Previous examples with bitumen

Machine Learning algorithms have been used to analyse bitumen. In 2019 Zofka & Blazejowski (Zofka and Błażejowski, Machine Learning Technique for Interpretation of Infrared Spectra Measured on Polymer Modified Binders 2018) used an artificial neural network supervised classification algorithm to classify polymer modified bitumen. The algorithm could successfully classify all 22 bitumen samples as unmodified, modified and highly modified. The bitumen samples were analysed with infrared spectroscopy. The algorithm was trained with the spectra from the infrared spectroscopy (Zofka and Błażejowski, Machine

Learning Technique for Interpretation of Infrared Spectra Measured on Polymer Modified Binders 2018). In 2017 F. Autelitano et al. (2017) used two different unsupervised algorithms to distinguish samples from three different bitumen from each other from odour fingerprints taken from the bitumen by means of up to 32 sensors. The bitumen could be distinguished with enough precision when samples from two different refineries where researched (Autelitano, Garili et al. 2017; Autelitano, Garilli & Giuliani 2019).

7.11.4 Designing Machine Learning for our study

Why use machine learning in bitumen characterization? Classification and regression algorithms could be used to identify rheological properties of bitumen from spectra analysed with infrared spectroscopy. Unsupervised learning algorithms could be used to distinguish malfunctioning, for example visbroken bitumen samples or REOB containing. These analyses would enable to analyse bitumen with considerably less time than with test conducted in laboratory. Also, a possibility of Machine Learning techniques is to find new variables from IR spectra and bitumen's chemical composition that could be linked to the performance of the bitumen from rheological data that has been determined from the bitumen samples.

7.11.5 Performance of MLAs

Designing and constructing machine learning algorithms is a process of trial and error. It is not possible to know which algorithm will perform best for a certain problem. The type of problem and the data that is used may help in deciding which algorithms to use. In the process of choosing well performing algorithm the data set is commonly divided into a training set and a testing set. The training set is used to compare the performance of different algorithms, while the test set is used to verify that the algorithm is performing for other data sets, as well as it is performing with the original training set (Géron Accessed: 17.5.2021).

Size and quality of a data set will influence the performance of the Machine Learning algorithm. The algorithm must have enough data that a working model can be computed (Géron Accessed: 17.5.2021). Amount of data needed for properly functioning models must be researched. Previous studies by Zofka & Blazejowski and F. Autelitano et al. have shown that predicting models can work with sample sets of 20–50 bitumen. However, the demonstrated differentiation was between 2–3 categories of products. It must still be further researched that how many samples is needed for models that can predict with sufficient accuracy from various bitumen in various circumstances. At some point the aspect of data redundancy starts to play a role. Redundancy of data is defined as the point beyond which any further feeding of data to the algorithm is not causing significant improvement in the algorithm.

An application that can be used in Machine Learning algorithms is online learning. Online learning means that new analysed samples will be used to as new data to improve the performance of the algorithm (Géron Accessed: 17.5.2021). Monitoring of the algorithm must be considered when training an online learning algorithm. How is it ensured that the model would perform as it should as the predicting model is changing? This must be kept in mind during trials.

7.11.6 Suggestion for research:

1. Designing the database – which spectra is expected to be indicative of what? How much data is necessary to distinguish between multiple parameters? Is adding additional parameters improving the categorization? How to collect data in the best way?
2. Developing the database collection protocol – sample naming, list of tests, procedures.
3. Performing supervised learning knowing the link between IR and rheology and performance.
4. Performing the unsupervised learning to investigate yet unapparent patterns between bitumen parameters.
5. Adding more dimensions of data to differentiate between samples not only in x-y coordinate system, but also in XYZ coordinate system – not only spectra – but development of spectra over time and possibly gases.
6. Testing the redundancy levels for bitumen applied in the field.

8 Introduction to Moisture damage mechanism – Part 3

During the year 2015–2020 an observation of moisture damage related issues in asphalt pavements in Finland started to occur more frequently, or rather brought up to public attention by publications in national news outlets, e.g. (Lehtinen 29.1.2018). The previously conducted analysis, indicates that due to the climate change Finland may find itself in a shift between a country with the main concern of failure related to studded tyres and thermal cracking, towards a country with substantial increase of temperature cycling over zero degrees Celsius with significant precipitation (Laukkanen, Halonen & Pyy 2012). In anticipation of such change, the previous research indicated the alterations to the quality control system related to water sensitivity in Finland, by inclusion of the freeze-thawing cycle into the currently used evaluation technique, namely Indirect Tensile Strength Ratio between dry and moisture conditioned asphalt samples (Laukkanen, Halonen & Pyy 2012; Finnish Pavement Technology Advisory Council 2017). Even the winter of 2019/2020 in Southern Finland could have been characterized mostly by positive temperatures and precipitation in form of rain, rather than snow. Anticipation of future failures is of current concern.

However, during the initial discussions during the preparation of BITU2020 project, a consensus was reached that a review of all of the aspects which are influencing the moisture damage, not only the specific testing method developments, should be conducted for the benefit of further development of specifications in Finland. Therefore, hereby report aims at providing the review of theory existing in the literature on aspects related to interaction between raw materials, characteristics of raw materials which affect the resistance to moisture damage (bitumen and aggregates), influence of moisture as well as the influence of air void content.

9 Moisture damage

9.1 Definition and scope

Moisture damage in asphalt pavement is defined as a deterioration of asphalt performance due to the exposure to moisture (Behiry 2013; Dore & Zubeck 2009). In intermediate and warm climate regions, the issue relates mostly to the loss of strength and durability inflicted by the presence of water in the structure, either in air voids, on the interface between aggregate-binder or within binder/mastic.

In the context of cold climate engineering, water trapped inside of the pavement has additionally an opportunity to freeze and thus expand its volume when the temperature in Celsius degrees within the pavement structure is cyclically positive and negative, so-called "cycling over zero" (Laukkanen, Halonen & Pyy 2012). The volume expansion of water, during shrinkage of asphalt inflicts expansion forces within the pavement. This can cause either micro cracks or separation between the grains. The effect of deicing chemicals on the adhesion or cohesion in asphalt concrete is different, than that of pure water.

For this reason, the moisture damage analysis in this report will not only look at the interaction between the bitumen and aggregate but tries to include the aspect of freezing into the discussion.

9.2 Exclusion

The aspect of water seepage through asphalt concrete cracks in wearing course to beneath layers, and its effect on propagation of potholes, is not the subject of this report. The effect of moisture in the granular course and subgrade is not discussed. The report focuses on the effect of the material compatibility between aggregates, bitumen, and modifiers in asphalt concrete on performance of wearing course layers. The effect of material composition on the adhesion of bitumen to the aggregate is the main subject of discussion.

The review of the testing methods, investigating water sensitivity or resistance to moisture damage is not within the scope of Part 3 in BITU2020 series. The review of the testing methods available on the market and review of international experience related to the various techniques is provided in Part 4 of the BITU2020 report.

10 Moisture damage mechanism

The mechanism of moisture damage can be divided into two processes (Caro et al. 2008):

1. Transport of moisture – in the form of liquid or vapor the water permeates into the asphalt mixture, through binder, mastic and reaches the interface between binder and aggregate
2. The response of the system – the change in the structure of the asphalt concrete influencing the deterioration of performance

The reduction of mechanical properties (response of the system) of asphalt concrete due to moisture damage can be divided in general as

- the adhesive failure – the loss of bond between aggregate and bitumen
- cohesive failure – the loss of bond between two grains of aggregate covered with bitumen, the failure occurs through bitumen/mastic

The adhesive failure, also known as stripping, is proposed to be manifestation of:

- displacement (S. Anastasio 2015; Hunter, Self & Read 2015; Dore & Zubeck 2009; Caro et al. 2008; Behiry 2013)
- detachment (S. Anastasio 2015; Hunter, Self & Read 2015; Dore & Zubeck 2009; Behiry 2013)
- film rupture (Hunter, Self & Read 2015; Behiry 2013)
- chemical disbonding (S. Anastasio 2015; Hunter, Self & Read 2015).

The loss of cohesion is proposed to be manifestation of:

- spontaneous emulsification, i.e., dispersion of water in bitumen
- mutual solubility of water and bitumen
- micro or macro cracks inflicted due to expansion forces caused by freezing water.

Types of distresses caused by loss of adhesion and cohesion (Caro et al. 2008):

- stripping – the separation of bitumen film from aggregate (Caro et al. 2008)
- raveling – dislodgement of rocks from the pavement surface (Caro et al. 2008)
- shelling – the loosening and removal of aggregates from seal coat or other surface treatment (Caro et al. 2008)
- hydraulic scouring – the pumping of the water into the asphalt concrete structure by the action of tires and pressure induced by them (Hunter, Self & Read 2015; Laukkanen, Halonen & Pyy 2012; Caro et al. 2008).

The origin of the stripping damage is postulated to be related to pore pressure issue. The damage inflicted by water pressure in the pavement to the film of bitumen or structure of mastic, introduces microcracks. The water has then easier path to travel to the interface between bitumen and aggregate. Alternatively, or simultaneously, the diffusion through the mastic causes the transfer of the water to the interface (Caro et al. 2008)

The water trapped in the pores causes distress:

- in combination with traffic – in badly compacted mixtures, the water remains in the air voids and as a result of traffic loading causes failures (water is medium with extremely low compressibility, when it fills the voids the load is transferred)
- in combination with freeze-thawing – the water trapped in air voids freezes and increases its volume, while asphalt concrete shrinks – this generates expansion forces leading to cracks,
- in combination with heating – water trapped in non-connected air voids expands at higher temperatures, i.e. during summer after rain expansion may happen before full evaporation occurs.

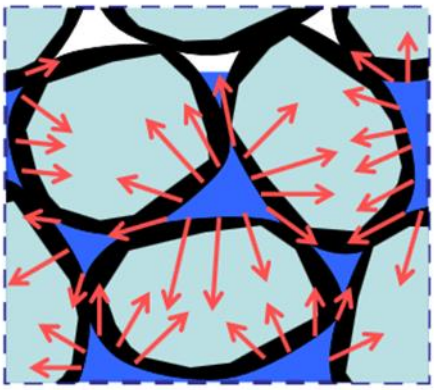


Figure 138. The forces inflicted by water expansion in air voids due to either traffic loading, freezing or heat induced thermal expansion (after N. Kringos 2007).

10.1 The properties affecting the moisture damage mechanism

The moisture damage mechanism depends on many aspects related to the material properties as well as to the external factors.

The list of all the identified parameters is provided in the Table 16, adapted from (Hunter, Self & Read 2015). Some of the parameters are in direct control of the construction team, such as design (including choice of materials), workmanship and air voids content. Other aspects related to material properties are not possible to influence, such as aggregate properties or rheology of the binder. Those parameters are measurable, and at large already required for testing in the specifications. Based on the result of the tests of raw materials a proper decision on the compatibility of materials can be achieved. A decision about the use of performance improving additives can be done at that moment.

However, the one aspect not included in the Table 16 is an actual compatibility between bitumen and aggregate. The theories focused on establishing this parameter are the main focus of this report.

Table 16. Material properties and external factors potentially affecting the bond between bitumen and aggregate (adapted from Hunter, Self and Read 2015). The properties to which an immediate effect can be inflicted during production, design and maintenance are bolded, the properties measurable are in italic, and the properties currently measurable and described in specifications and guidelines are underlined.

Aggregate properties	Bitumen Properties	Mixture properties	External Factors
Mineralogy	Rheology	Void content	Rainfall
Surface texture	Electrical polarity	Permeability	Humidity
Porosity	Constitution	Bitumen content	Water pH
Dust		Bitumen film thickness	Presence of salts
Durability		Filler type	Temperature
Surface Area		Aggregate grading	Temperature cycling
Absorption		Type of mixture	Traffic
Moisture content			Design
Shape			Workmanship
Weathering			Drainage

11 The mechanism of adhesive and cohesive failure of asphalt concrete

As mentioned above, the damage due to moisture presence in asphalt concrete is a result of either adhesive or cohesive failure (Figure 2). Each of those mechanisms can be explained by multiple theories, which are strongly related to the conditions in which the pavement is located. For example, there are different reasons behind the cohesive and adhesive failure in cold climate regions, than in the warm climate regions. However, the theories are not mutually exclusive.

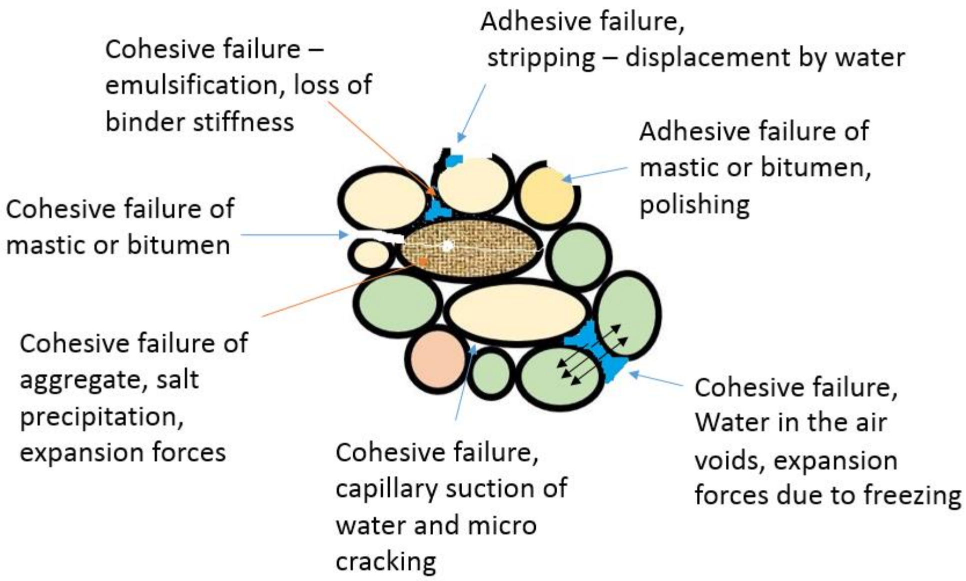


Figure 139. The non-loading related modes of and reasons behind observed moisture damage.

11.1 Adhesive failure

The adhesive failure between bitumen and aggregate can occur due to the incompatibility of those two components of asphalt concrete. In Figure 3, the visual explanation of the concepts related to some stripping theories is presented.

However, the loss of bitumen from the surface of the grain of aggregate can occur due to the polishing by the action of tires. Considering the specific conditions of Finland, the action of studded tires may inflict the immediate cracks in the bitumen film, which cause higher susceptibility to water damage. Additionally, weak boundary between the aggregate grain and bitumen contributes to failure at the interface.

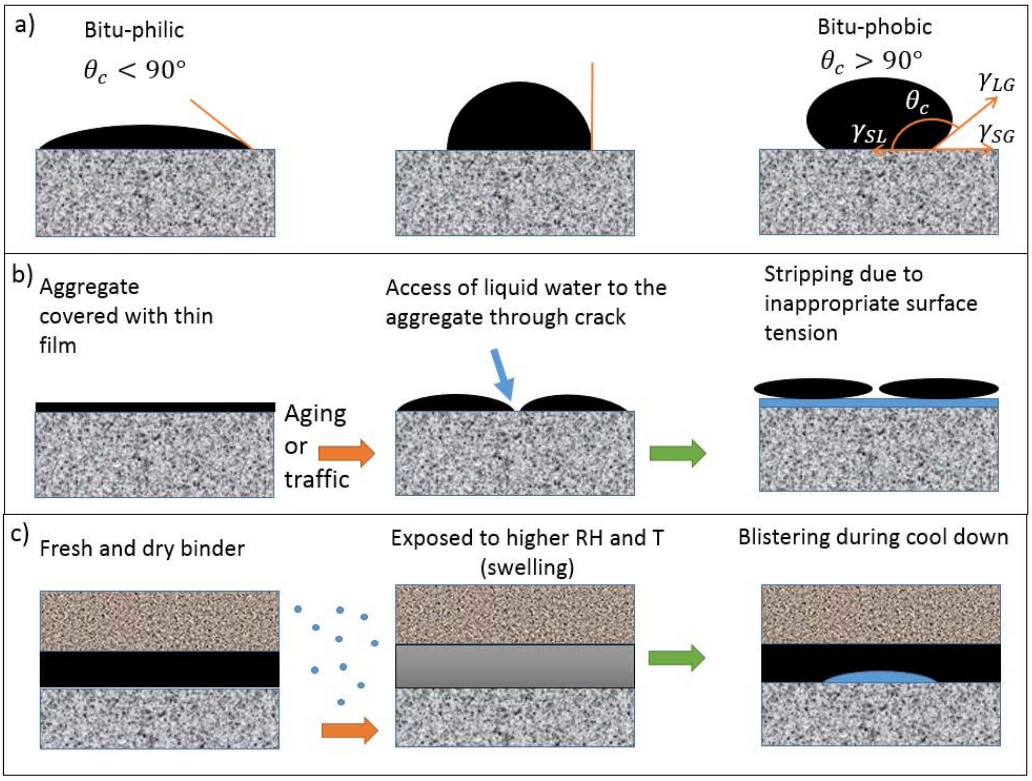


Figure 140. The explanation of the theories behind the non-traffic induced adhesive failure in asphalt concrete.

11.1.1 Weak interface region

The theory states that the failure may occur due to the weakness of the interface region between aggregate and bitumen (Caro et al. 2008). If the aggregates are covered with dust prior to application of bitumen, and especially if the bitumen content is insufficient in the mixture, the risk of bad contact between aggregate grain and bitumen is high (Caro et al. 2008).

One reason behind the lowering of the strength of the interface layer may be due to the reaction of the aggregate surface with water, e.g. dissolution of complexes (Caro et al. 2008). For pH levels below six, the dissolution of carbonates occurs, and above 8 the dissolution of silica layers is favored. The rainwater on average has a pH of 5.6 (Liljestrand 1985).

Additionally, in the case of not fully weathered slag aggregates similar effect may occur. The surface of slag grain may be changing due to exposure to moisture causing an expansion and localized change in density.

11.1.2 Electrostatic forces and zeta potential

During exposure to water a layer of ions may be formed on the surface of the aggregate. Two layers are formed – a stern layer and a diffuse layer. The stern layer is composed of layers of ions with opposite charges. The diffuse layer is a layer beyond stern one, into which the ions diffused due to thermal motion.

The measure of the electric potential at the shear plane between those two layers, known as electrokinetic potential or zeta potential, has been used to study the debonding in asphalt concrete in the presence of moisture. For the bitumen and aggregate this value depends on the pH and the material. It was postulated that if both components have for example a negative potential across the pH range, the repulsion between the materials will cause debonding (Caro et al. 2008).

11.1.3 Chemical bonding

The aggregates, which contain carbonate content (also known as basic aggregates) are easier to coat with bitumen than the aggregates with high silica content (also known as acid aggregates). The hydroxyl group of the acid aggregates has an affinity with the carboxylic group (present in binder, especially aged binder). On the other hand, in the presence of water, those bonds are prone for displacement (Caro et al. 2008).

As a result of studies on multiple aggregate types and few binder types, the conclusion indicated that the composition and rheology of the binder was not as influential to this type of the adhesive failure, as an aggregate surface composition was.

Chemical bonding related adhesive failure mode is the basis of the explanation of the positive effect of the hydrated lime addition. The reaction between calcium hydroxide and carboxylic groups in bitumen, causes the neutralization and deactivation of those groups in bitumen which otherwise when adsorbed on the aggregate would be prone for displacement in moist environment (Caro et al. 2008)

11.1.4 Surface tension and the adhesive bond

The surface free energy (SFE) of the materials is the reason behind the physico-chemical adhesion between the two materials (Caro et al. 2008). The surface free energy theory suggests that surfaces attract other substances to reduce the free energy (Zhang et al. 2018). The SFE is also understood as the amount of work necessary to create a unit of surface area in the vacuum (Caro et al. 2008).

The surface free energy (SFE) of solid can be determined using the contact angle measurements between liquid and solid surface (Figure 140) (Kłonica & Kuczmaszewski 2017). In order to conduct the calculations, results from at least two liquids of known surface tension are required.

$$\sigma = \gamma + S\frac{\partial \gamma}{\partial s} \quad (22)$$

where

σ – surface tension
γ – surface free energy
S – unit of area of a given body.

In case of liquids, the following assumption is made

$$\frac{\partial \gamma}{\partial s} = 0 \Rightarrow \sigma = \gamma, for\ S \neq 0 \quad (23)$$

As a result, the Young equation

$$\sigma_{SV} = \sigma_{SL} + \sigma_{LV} cos\theta_v \tag{24}$$

where

σ_{SV} – surface tension in the solid-gas interface, σ_{SL} – surface tension in the solid-liquid interface, σ_{LV} – surface tension in the liquid-gas interface, Θ_V – equilibrium contact angle, takes form:

$$\gamma_s^0 = \gamma_{sl} + \gamma_{LV} cos\theta, \tag{25}$$

where

γ_s^0 is a surface free energy of solid in vacuum
γ_{sl} and γ_{lv} are solid liquid interfacial tensions at equilibrium.

The SFE of liquid on a surface can be calculated from an equation, which considers the influence of the polar (p superscript) and dispersive (d superscript) component:

$$\gamma_{sl} = \gamma_{lv} + \gamma_s^0 - 2\sqrt{\gamma_s^d \gamma_{lv}^d} - 2\sqrt{\gamma_s^p \gamma_{lv}^p} \tag{26}$$

Knowing the liquid-vapor surface tension of the two liquids and combining the equation 3 and 5, the calculation of the SFE for the solid surface can be achieved (Zhang et al. 2018).

Once the SFE is obtained for the aggregate, the use of bitumen as a third liquid allows for studies on the surface tension of the bitumen. Knowing the interfacial tension between the aggregate and bitumen in air allows us to study the work of adhesion in air:

$$W_{SL} = \gamma_l (cos\theta + 1) \tag{27}$$

The same equation can be used to study the work of adhesion between aggregate and water (W_{sw}), between aggregate and bitumen (W_{sb}), and between bitumen and water (W_{wb}). When the asphalt concrete is submerged in the water the system and its adhesion work (W'_{bsw}) can be defined as:

$$W'_{bsw} = W_{bw} + W_{sw} - W_{bs} \tag{28}$$

The work of adhesion is understood as the work necessary to displace the unit of area of bond between aggregate and bitumen, to form a new bond between water and aggregate (Caro et al. 2008). For the purpose of analysis of numerical results – the lowest work of adhesion of the system indicates the least propensity for stripping. The higher the value of work the more likely the stripping occurs.

In the study of ten bitumens and three aggregate types (limestone, granite, diorite) it was determined that the lowest stripping work was observed for limestone aggregates. The average work of stripping for limestone was 165 mJ/m^2, while for Granite and Diorite the values were 197 mJ/m^2 and 198 mJ/m^2, respectively (Zhang et al. 2018). The analysis of the effect of silane modification of the aggregate surface and its effect to the work of stripping, resulted in reduction by half for two of the reported bitumens and limestone aggregate.

Studies on the difference in the contact angle between various bitumen indicate that the composition of the bitumen has an effect on the work of stripping (Hefer, Bhasin & Little 2006). Different additives affected bitumens in a different manner (improving or deteriorating the work of adhesion in the system) (Hefer, Bhasin & Little 2006).

The procedures to determine components of the work of adhesion are described and discussed in the report of BITU2020 – Part 4. However, it is worth mentioning that the work of adhesion theory is based on the assumption and thus the results are relative and values hard to compare. The assumption used is connected with the equality of the polar and dispersive components of water at 20 °C (Hefer, Bhasin & Little 2006). What is also worth bringing to attention is that stripping occurs at low temperatures. In those conditions, the relationship between the components of the work of adhesion may be different.

11.1.5 The surface tension of droplets on aggregate

On ideally flat surface the measurement of contact angle is plausible. However, the contact angle on the porous surface may change due to imbibition of liquid into the pores on the surface. During that process the diameter of the droplet may change, and so does the wetting angle (Boulangé, Bonin & Saubot 2013). If the surface is not extremely porous, the droplet spreads and angle changes smoothly. If the surface is highly porous, the change of the shape of the droplet and the wetting angle occurs in steps (Figure 141).

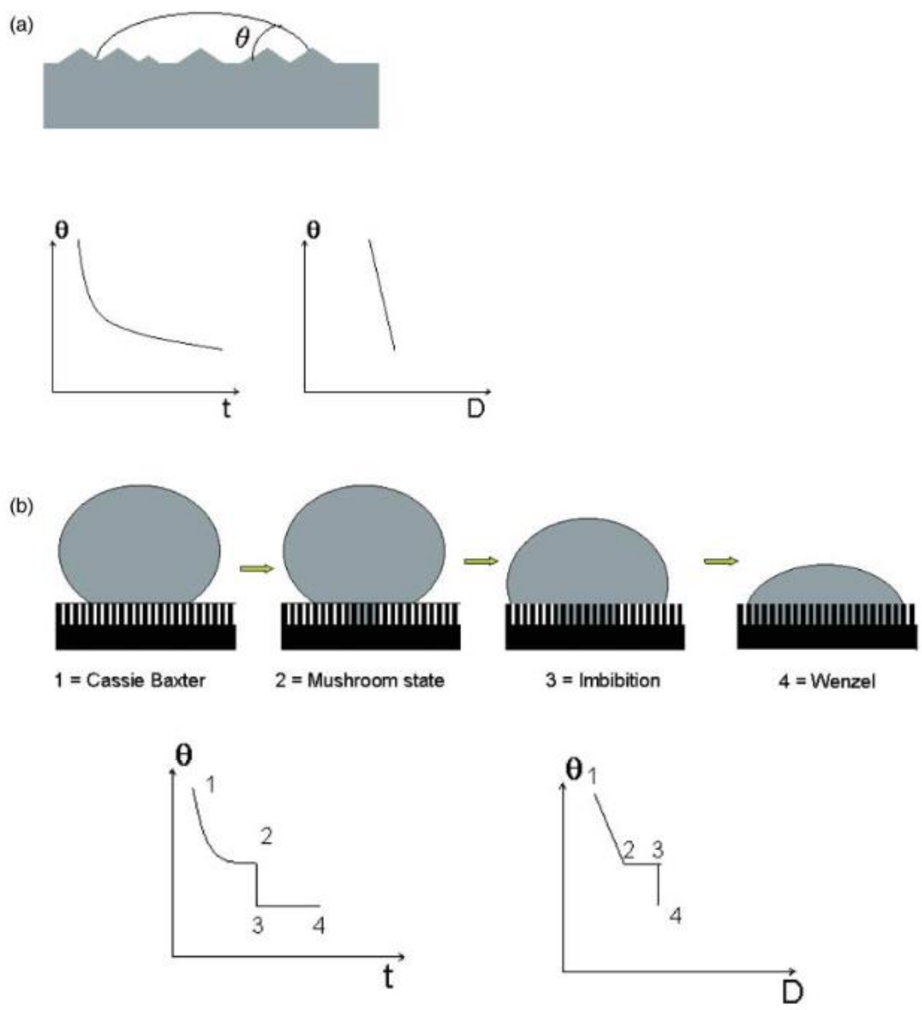


Figure 141. Wetting of textured surfaces: Wenzel model on rough surfaces and its kinetics (a), wetting on the textured surfaces and kinetics of water droplet evaporation (b). Reprinted from Boulangé, Bonin & Saubot (2013).

The addition of adhesion promoters (i.e. amidoamine, fatty acid and phosphate) was not found to significantly alter the surface tension of bitumen. However, the addition of the adhesion promoter seemed to alter the distribution of the fractions of some of the bitumen when tested using the Thin Layer Chromatography. An addition of up to 1% of various adhesion promoters could alter the "aromatic/resin" ratio significantly from 1.3 to 2.4. The binders with higher aromatic/resin ratio corresponded with higher adhesion to aggregates (Boulangé, Bonin & Saubot 2013).

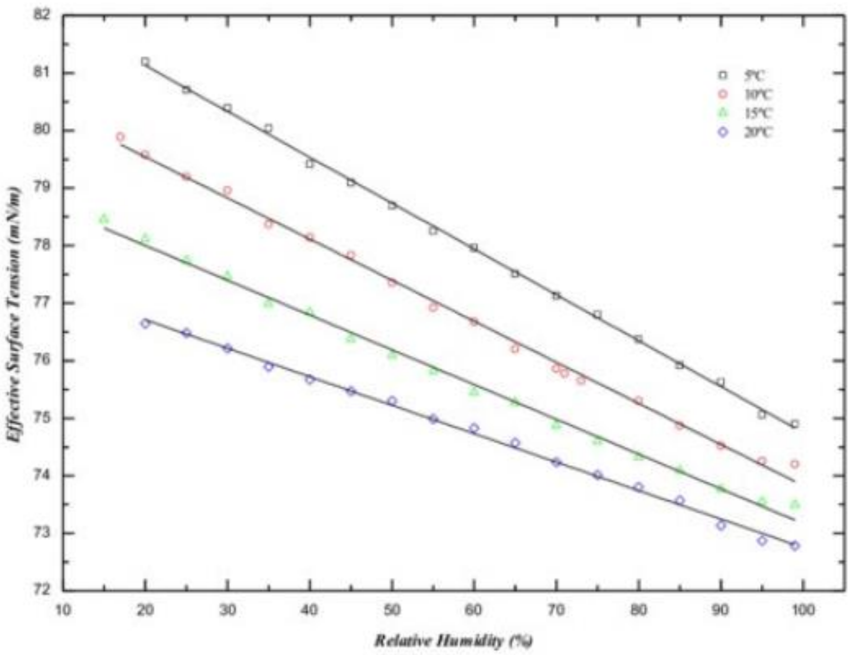


Figure 142. The effective surface tension of water in the function of relative humidity and temperature. Reprinted after Pérez-Díaz, Álvarez-Valenzuela & García-Prada (2012).

Using this as an analogy, the effect of the aromatic to resin ratio in the starting material could prove useful for evaluation of the stripping propensity. The change between VR and VBR is in fact in shift towards lower aromatic content. What will be the effect on the adhesion?

Because the surface tension of bitumen is related to the surface tension of water at certain temperature and humidity, it is worth noting that surface tension of water changes as a result of those two parameters (Figure 142) (Pérez-Díaz, Álvarez-Valenzuela & García-Prada 2012). At low temperature and low humidity, the water droplet is having the highest surface tension. Whereas at higher temperatures and higher relative humidity (RH%), the surface tension reduces. This means that aggregate surfaces can be more hydrophilic at low temperatures and low RH%, while they can be more bitu-philic at higher temperatures and higher RH%.

11.1.6 Hansen Solubility Parameter (HSP) and adhesive bond loss due to blistering

In the temperature conditions considered as normal laboratory conditions, i.e. 20–25 °C and atmospheric pressure, bitumen is not miscible with water. However, the situation is different at elevated temperatures or at high relative humidity conditions. This phenomenon can be explained by the Hansen Solubility Parameters (HSP) theory (C. Hansen 2007).

The basis of HSP theory claims that a cohesive energy of the substance (E) is a sum of energies that make it up. The meaning of the cohesive energy is in the energy required to vaporize the substance (causing it to transition from liquid to gaseous state). Those subcomponents of the cohesion energy are called:

- the energy related to dispersion interactions (E_D)
- the polar cohesive energy (E_P)
- the energy related to electron exchange parameter (E_H).

The solubility parameter (δ) is defined as the energy divided by the molar volume of the tested substance.

In the absence of the other two parameters, the dispersive cohesion energy (E_D) is always present. The alkanes, which do not have polar component or hydrogen bonding interaction, upkeep the cohesion due to the dispersion interactions.

Polar cohesive energy is due to polar interactions between molecules of the substance considered. In the systems with permanent dipole-permanent dipole interaction. Presence of dipoles and high polar cohesive energy is not equal with the water solubility.

The electron exchange parameter covers the interactions related to the bonding between molecules due to for example the hydrogen bonding. This parameter is more indicative of the mutual solubility with water.

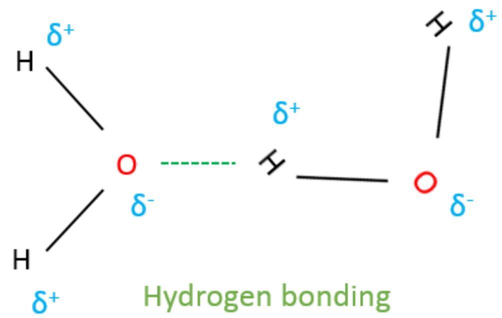


Figure 143. The hydrogen bonding and dipol-dipol interaction.

The three parameters (δ_D, δ_P, δ_H) can be plotted in three dimensional space. The pure substances, defined as a solution of one type of molecules, are typically having a well-defined point within this three-dimensional space (Table 17). Solutions of multiple substances, for which establishing molar volume is challenging, such as polymers or bitumen are defined by so-called solubility sphere. By dissolving the bitumen in for example 48 pure solvents, we can establish in which solvents the bitumen is not soluble in and which solvents are creating a stable homogenous solution with it. Mathematical fit is used to determine the sphere of solubility based on the binary response (soluble or non-soluble) (P. Redelius 2000). Such sphere is defined by the center coordinates of the sphere and the radius of the sphere (RAD) (C. Hansen 2007). An example of such sphere is provided in Figure 144.

The mutual solubility of two substances is governed by the distance between the points (two solvents), or the sphere and the point (e.g. bitumen and toluene). If the distance is large, the two substances are non-miscible. If the RAD of the sphere is larger than the distance in the solubility parameters (Ra) of sphere and the solvent, the two are fully miscible. However, in the situation when the HSP of a solvent is within the RAD of sub fraction of a mixture, e.g. heptane and maltenes the solvent may solve part of the mixture and cause precipitation of the insoluble particles (in this case asphaltenes).

Based on the chosen data provided in Table 17, we can see that methanol is not a solvent for bitumen, while toluene is. The heptane case discussed above is causing the instability and onset of asphaltene precipitation, thus creation of heterogeneous suspension.

Table 17. The HSP parameters for chosen substances (C. Hansen 2007).

Substance	Dispersion	Polarity	Hydrogen Bonding	Radius (RAD)
Methanol	15.1	12.3	22.3	
Heptane	15.3	0	0	
Toluene	18.0	1.4	2.0	
Bitumen (average)	17.9	4.6	3.23	5.45

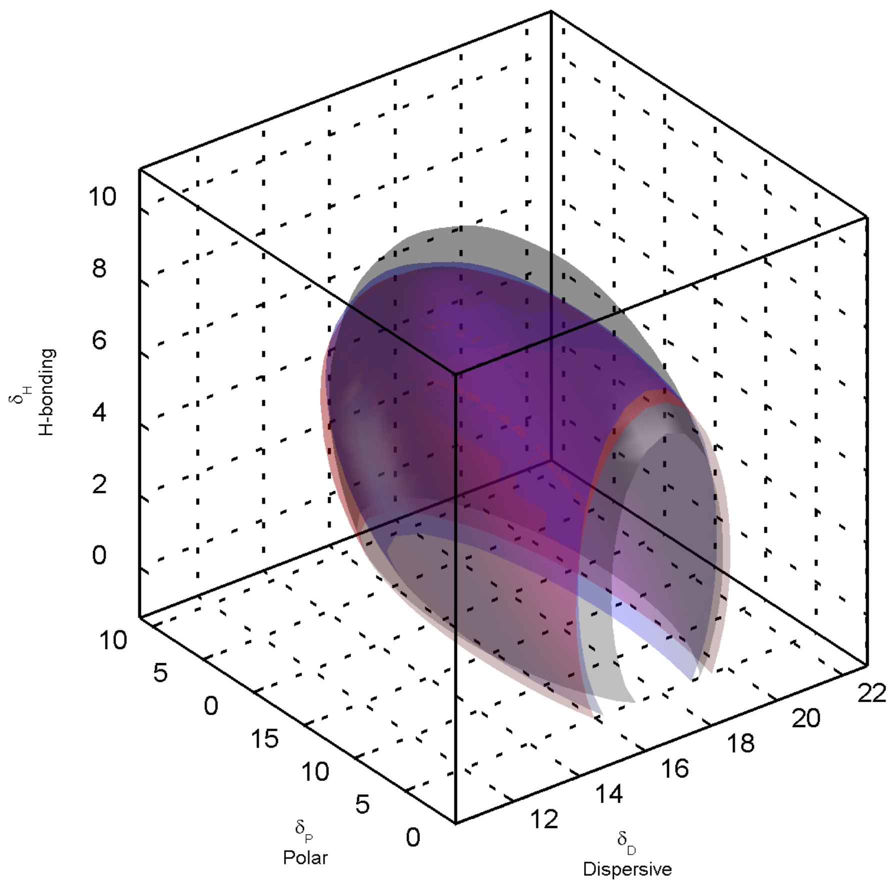


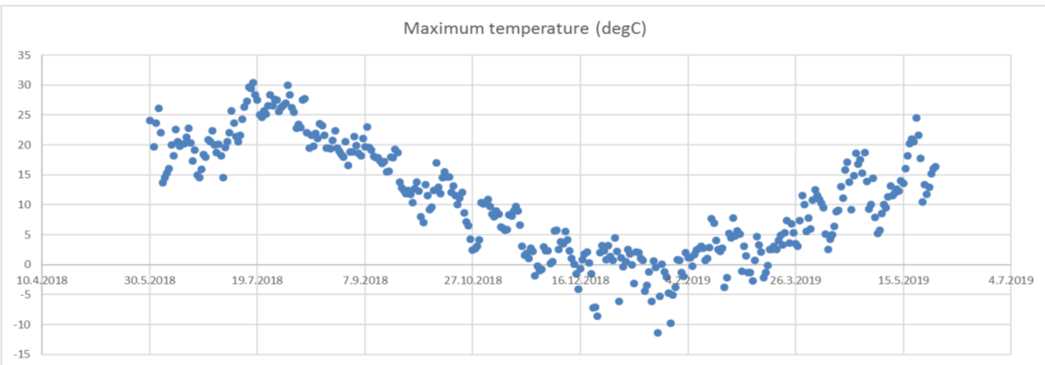
Figure 144. The results of the HSP determination for bitumen specimen prepared from the Highway Number 1 in Finland – the colors represent bitumens collected from various sites (after (M. Makowska 2017).

The reason for the discussion of HSP theory in the context of moisture damage is the peculiar case of water as a solvent. The solubility parameter of water is changing quite drastically with increase in temperature. The water in form of a liquid is having quite high polar energy and hydrogen bonding. However, with increasing distance between the water molecules upon heating, and eventually evaporation, leads to the loss of hydrogen bonding component. This simple fact is postulated to explain the blending of the steam and bitumen for reduced viscosity during the foaming of bitumen.

However, water in the air is also in the state in which hydrogen bonding is at the minimal value. The high relative humidity in the environment in which asphalt pavements are located essentially exposes the bitumen to gaseous water as a solvent. When the moisture from the air diffuses and blends to some extend with the bitumen, the bitumen may lose its cohesion and we would observe it as a cohesive failure or the reduction of the asphalt concrete strength. However, if the temperature of the mixture drops suddenly, the hydrogen bonding parameter increases and association between two water molecules becomes more pronounced. This leads to the formation of droplets of water, which may sweat out of bitumen. If the sweating happens onto the bitumen surface, this water can evaporate and leave the system. On the other hand, if the water is trapped inside of the pavement and the aggregate is highly hydrophilic, the droplet of water may precipitate on the interface between hydrophilic aggregate and bitumen, leading to blistering and stripping (Figure 140 c), leading to the adhesive failure.

In summary, the asphalt concrete may be impermeable to liquid water, and demonstrate the moisture damage due to exposure to air of high relative humidity at high air temperatures and followed by sudden drop in temperature. Such behaviour was in fact observed in regions of low precipitation (Caro et al. 2008). The relative humidity in the region of Helsinki is provided as a context for this theory in the Figure 8.

The high relative humidity levels during autumn months, at relatively lower temperatures in comparison with spring and summer months, potentially lower the surface tension of water. Typically, the moisture damage demonstrates itself in Finland in the spring, which could be connected with this phenomenon of increasing surface tension of water due to the change in RH and temperature relationship.



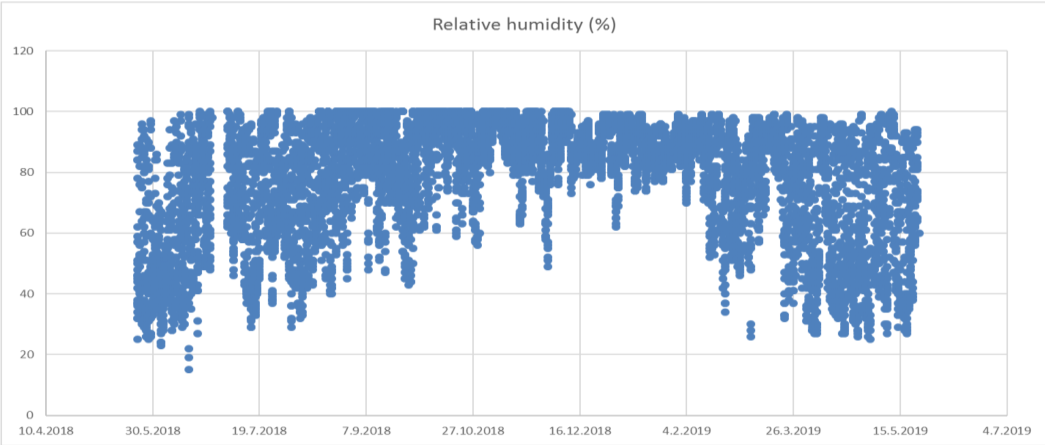


Figure 145. The maximum daily average temperature with corresponding relative humidity data for Helsinki Kaisaniemi (Source: Ilmatieteenlaitos).

One of the suggestions would be to investigate the possibility of decreasing the surface tension of water during those months by for example an application of typically used deicing chemicals, such as sodium or calcium chloride. A comparative study of varying maintenance techniques on the pavement during spring could decrease the occurrence of moisture damage.

11.2 Loss of strength due to cohesive failure

The current Finnish Asphalt Specifications (Finnish Pavement Technology Advisory Council 2017) use the measurement of Indirect Tensile Strength, and evaluate the mixtures based on the ratio between dry and wet conditioned samples. The test effectively tests cohesion of the compacted asphalt mixture. The issues related to the testing methodology and proposed methods of its improvement are a subject of the report provided as a result of BITU2020 – Part 4. This section focuses on the effect of the multiple mechanisms of loss of cohesion on the cohesion of asphalt concrete due to exposure to moisture, de-icing and low temperatures.

The loss of strength can be of course due to the lack of adhesion between the binder and aggregate. The mechanical properties of the asphalt concrete are linked to the properties of bitumen, e.g. he relationship between binder stiffness and asphalt stiffness (Bonaquist, Pellinen & Witczak 1998), resistance of asphalt concrete to low temperatures is linked to bitumen's resistance to cracking (Kanerva & Nurmi 1991). Based on the analysis using the semi-circular bending test of asphalt, the rheological characteristics of bitumen and its composition, as well as type of aggregate and air voids, affect to the fracture energy, thus cracking (Li & Marasteanu 2010). Therefore, the loss of cohesion within the binder, i.e. the change in its rheological characteristic due to exposure to moisture, is expected to affect to the strength significantly. Of course the loss of adhesion between the bitumen and aggregate will cause the lack of cohesion within the asphalt. However, within this part of the report the aspect of adhesion influence is omitted.

11.2.1 Hansen solubility parameters and cohesive failure of bitumen

The concept of organic liquid swelling in humid environment was described in the context of blistering and adhesive failure in the section 11.1.6 . The sweating out of this water may lead to blisters and adhesive damage. However, the blending with gaseous water, or swelling, may have an immediate effect on the cohesive strength of the binder. Additionally, the sudden drop of temperature from just above 0 °C to minus may additionally cause freezing of crystals and microckracking in the binder.

For polymers, the Soluble Water Exuded At lower Temperatures (SWEAT), is measured in a simple test. The polymer films of 1 or 2 mm are submerged at various temperatures in water and the increase of mass is measured over couple of days (C. Hansen 2007).

One could postulate that the water boiling bottle test of adhesion is utilizing the same principle (Hugener, Beltzung & Angst 2012). The experience of Switzerland suggests that better prediction of propensity for moisture damage is obtained by using the water at elevated temperatures, at least above 50 °C, followed by the cooling. The propensity for susceptibility to diffusion of water into the bitumen could be a noteworthy parameter to follow. The currently applied methodology from SFS-EN 12697-12 (method A) suggests conditioning in 40 °C and cool down to room temperature. Identification of the temperature differentiating the best between the bitumen samples in terms of their resistance to swelling could be of interest for improvement of the predictive power of the currently applied tests.

11.2.2 Emulsification, osmosis and the influence of sodium on cohesive failure of bitumen

The reason behind this subject is connected with the similarity of the emulsion composition and bitumen in cold climate conditions with an amine-based adhesion promoter. The emulsion of bitumen in water is typically designed by the use of amine additives, which are suspended in the acidic liquid solutions of water with an addition of calcium chloride (Hunter, Self & Read 2015; Khan 2018). In principle, using an amine promoter in the rain (average pH = 5.6) with the possibility of the calcium chloride presence, creates a possibility of unintentional emulsion formation.

This is considered a risk for the cold climate, where calcium chloride ($CaCl_2$) is used not only as a de-icing chemical during the winter, but also as a dust stabilizing chemical on the gravel roads. The emulsification would cause a decrease in viscosity of the binder, lower cohesion, potential bleeding and decrease of pavement strength.

Although bitumen is considered a water proofing material, at very thin thickness of film, it can be considered a semi-permeable membrane. This has to do with the content of electrolytes naturally present in the structure. The issue is the most significant during the preparation of emulsions of bitumen. If the concentration of the sodium chloride (NaCl) is above 300 parts per million (ppm) the osmosis causes an uptake of the water into the droplet, causing a swelling. (Hunter, Self & Read 2015) The addition of $CaCl_2$ is used to stabilize the emulsion and prevent the swelling due to osmosis. It is unclear, if bitumen with high sodium content and an added amine additive would undergo osmotic swelling in the absence of $CaCl_2$.

In the context of previously discussed legislative changes, the alterations in bitumen composition are envisioned, potentially leading to an increased content of visbroken residue. How the sodium (Na) content changes between feedstock from VR unit and VBR is not apparent from the literature (Stratiev, Kirilov et al. 2008; Stratiev, Nedelchev et al. 2015). Some mineral content increases and some decreases. This parameter should be studied more. The evaluation in the context of the susceptibility for the uptake of the water into the asphalt structure is suggested.

The electrical conductivity of the bitumen and mineral content, linked with tests on diffusion of water into the bitumen could provide additional information about susceptibility of the product to the cohesive failure due to moisture.

11.2.3 Cohesive failure of asphalt concrete due to deicing chemicals

One of the factors affecting the cohesion in asphalt concrete due to moisture is weathering of the aggregate itself. This aspect was studied extensively during the ASTO project and as a result the current Finnish Asphalt Specifications define the mineralogy of the aggregates suggested for use in the paving industry. Nevertheless, with the changing sources of aggregates it is worth reminding about the potential risks connected with water absorption of aggregate as well as with crystallization of deicing chemicals inside of the aggregate structure. The absorption of water into the structure plays an important role, as during the freezing the volume expansion inflicts forces capable of breaking the rock. The crystallization of crystals in between the layers of minerals inside the rock inflict the expansion forces causing the weathering to be more pronounced, than in just the presence of water.

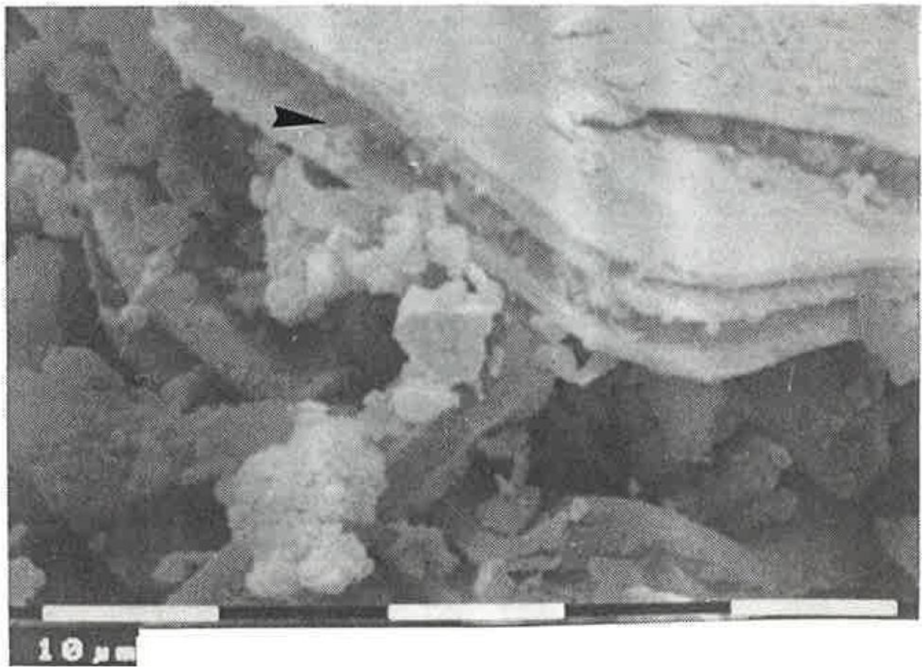


Figure 146. The Scanning Electron Microscope image of the crystalized NaCl in between layers of mica (Nieminen & Jänniskangas 1990).

The weathering resistance of 31 different rock samples was evaluated by exposure to 550 cycles of temperature change between -25 and +55 °C when submerged in water or a solution of 5% sodium chloride (NaCl). The material was tested with the Swedish impact test and Nordic ball mill test. The changes in the samples were presented by expression of gradation in the form of gradation modulus. The surface area and the porosity as well as water adsorption capacity were measured as indicative of changes inflicted by weathering.

The pyrrhotite containing aggregates were found to be the most susceptible for crystallisation of salt crystals within their structure. Coarse granular mica packs expressed similar behaviour. Based on the results a guide for choosing the aggregate type with resistance to weathering was developed. The developed scale was incorporated into the Finnish Asphalt Specifications. However, it is worth underlining that the original study was not investigating the alternative materials (or secondary waste materials) from the power and steel industry as part of the construction materials. For example slags, bottom ashes and other industrial crushed materials were not included in the original study (Nieminen & Jänniskangas 1990). The evaluation of their resistance for weathering in freezing water, as well as in freezing salt solution would be beneficial.

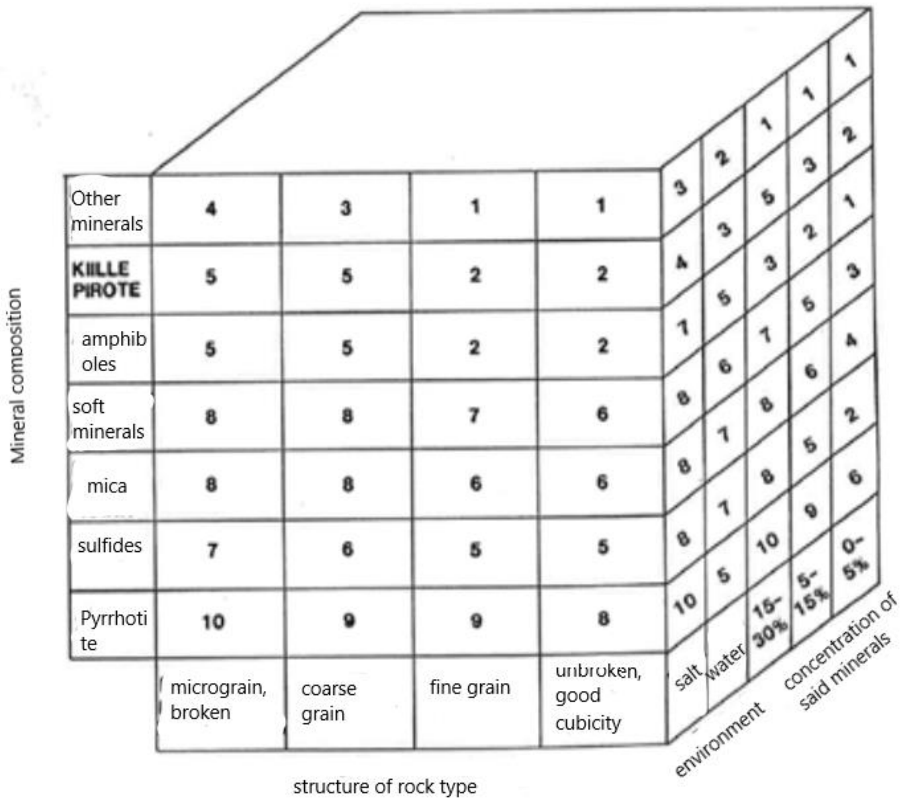


Figure 147. The guideline for choice of the aggregates appropriate for use in pavement construction, where 1 is least susceptible to weathering and 10 is the most susceptible. (Nieminen & Jänniskangas 1990).

Interestingly, the use of deicing chemicals is seemingly improving the adhesion between the binder and aggregates. This phenomenon was assigned to the different surface tension of the brine liquid in comparison to the pure water in the system (S. Anastasio 2015).

11.2.4 Cohesive failure of asphalt mastic

The influence of the asphalt mastic composition on tensile strength of asphalt concrete was postulated. The investigations including multiple filler and gradation type suggest varying uptake of moisture, different maximum moisture uptake and differing tensile strength. The authors postulated that particular mastic composition may fail cohesively only if the adhesive strength is higher than the cohesive one. The authors demonstrated how by the choice of mineral filler the cohesive strength after moisture conditioning can be manipulated in order to achieve ideal or desired behavior (Kringos et al. 2011)

Interestingly, the moisture uptake to mastic composed of polymer modified bitumen with the hydrated lime as a filler increased the uptake of moisture into the sample, while the switch between the limestone and hydrated lime for non-modified bitumen slightly decreased the uptake of moisture in case of hydrated lime.

Hesami et al. (Hesami et al. 2013) investigated the effect of the composition of the mastic on the moisture susceptibility of the asphalt mixtures. The parameters investigated involved size distribution of the filler particles, the concentration of filler particles as well as the addition of bio-modifiers. The measurement of the viscosity of the mastic before and after conditioning in water. It was noted that the type of the modifier may influence the viscosity of the mastic after the conditioning by up to 10%. However, the viscosity comparison between the samples was conducted at 100 °C. There is a possibility that the moisture present at this temperature may influence the rheological parameters in a different way than at room temperatures.

11.2.5 Cohesive failure due to air voids and expansion forces

Some of the construction materials such as slag are capable of volume expansion. The slag aggregate is a by-product of steel industry. From the chemical point of view, the composition of the slag is an aftermath of the processing of minerals at high temperatures. The slags are composed of oxides. For that reason, during the exposure to moisture (e.g. from rain or humidity) and carbon dioxide present in the air, the surface of the slag aggregate may evolve due to weathering.

It has been postulated that the calcium oxide content is the driving force in the volume expansion process. The reactions, which lead to such volume expansion can be described with below chemical reactions:

$CaO + H_2O = Ca(OH)_2$

$Ca(OH)_2 + CO_2 = CaCO_3$

Wang et al. (Wang, Wang & Gao 2010) provide the calculations of the expansion related to just the first reaction. Nevertheless, the volume of the surface layer of such grain can double. This is why the slag is suggested for expansion prior to use in the construction industry. If the expansion occurs in application, then the expansion forces may lead to cracking.

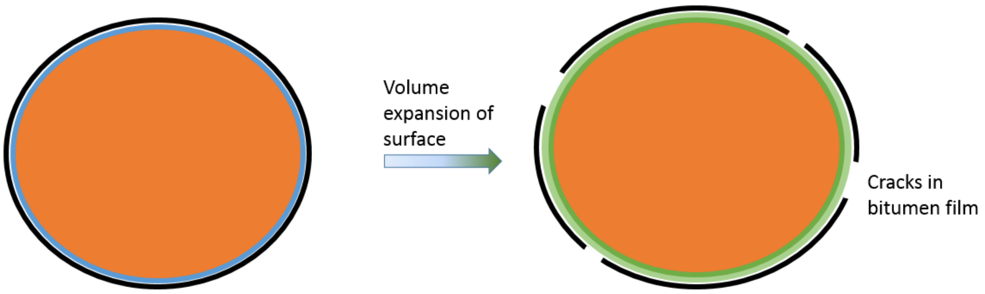


Figure 148. Schematic representation of the effect of surface expansion on the cohesion of asphalt concrete.

12 The effect of air voids on moisture damage

As a result of the studies conducted on the cores collected from pavements in California, the conclusion was reached that air void content, pavement structure, rainfall and pavement age have the highest influence on moisture damage while repeated loading and cumulative truck traffic have a marginal effect (Caro et al. 2008; Lu & Havery 2006).

Intuitively, the presence of air voids in the asphalt concrete is allowing for the infiltration of water into the system, exposing the pavement to the conditions propagating the moisture damage. However, as research suggests there is a **range of air void content** and **the size of air voids,** which determine the maximum propensity of the asphalt pavement to moisture damage (Masad, Castelblanco & Birgisson 2006). This particular air void size and content are referred to as ***pessimum*** **air void size** and ***pessimum*** **air void content**. The pessimum air void content/distribution differs for mixture type and aggregate type (Figure 149) (Caro et al. 2008).

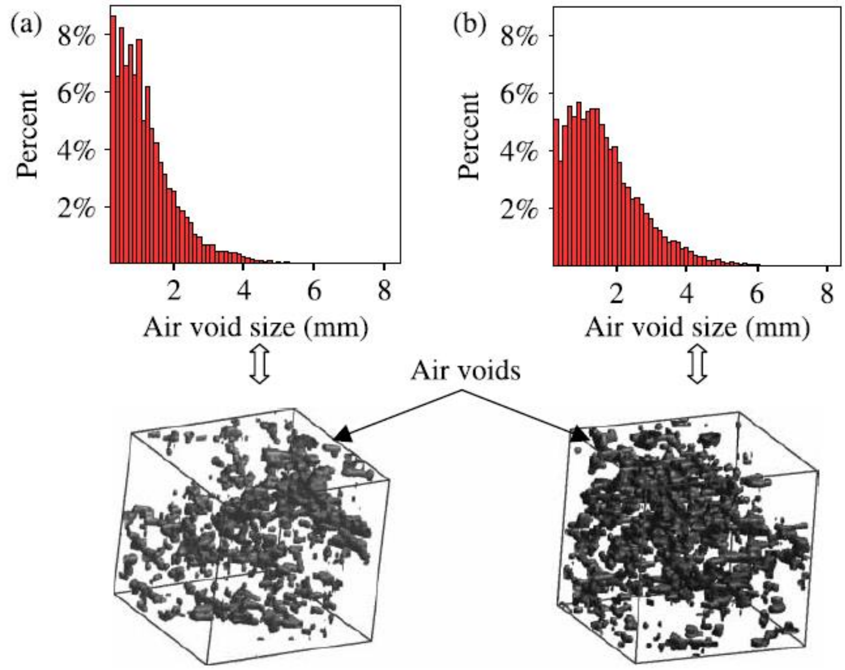


Figure 149. Visualization of air void distribution in asphalt concrete mixture with limestone (a) and granite (b) (Masad, Castelblanco & Birgisson 2006).

It was established that for the asphalt concrete the pessimum air void content is within the range of 5–10% (Caro et al. 2008). In the USA, the target air void content is often at the 7% level. Finnish government targets the air void content on the level of 1–4% for asphalt concrete and 2–5% for the stone mastic asphalt mixtures (Finnish Pavement Technology Advisory Council 2017), which should assure being outside of the pessimum air void content. However, the air void content on the joints and in the segregation spots may be higher than the bulk of the pavement.

The studies on the effect of pore size distribution and connectedness suggest that at the equal air voids content (6–7%) the air void size and connectedness influence the moisture damage susceptibility the most (Arambula, Masad & Epps 2007). The air void size and distribution within the structure can be measured with the Computer Tomography. The air void content and the air void size is differing across the structure, e.g. with depth. An example of the cross section of sample collected from the Ring Road II analyzed with the X-Ray CT in the laboratories of KTH in 2013 is provided in Figure 150. However, the measurement is limited by the image resolution. The series of scans, which allow for the 3D analysis of the sample is able to evaluate the air void content, distribution of sizes and connectedness (Figure 149).

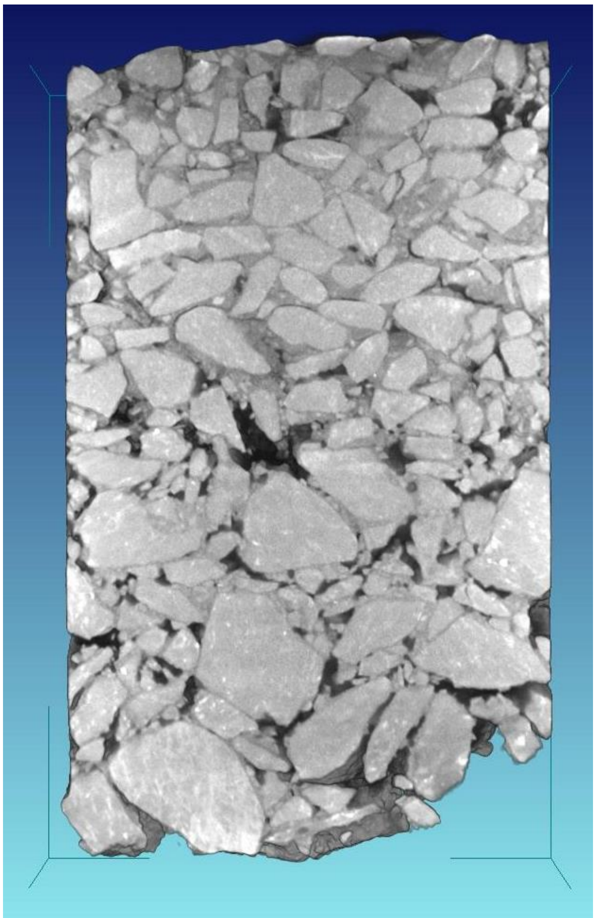


Figure 150. The X-Ray Computer Tomography of core collected from Ring Road II (location M) depicting cross section with visible air voids (Pellinen, Makowska et al. 2013).

Chen et al. (2004) classify the air voids in the asphalt concrete as so-called

- Effective
- Semi-effective
- Impermeable.

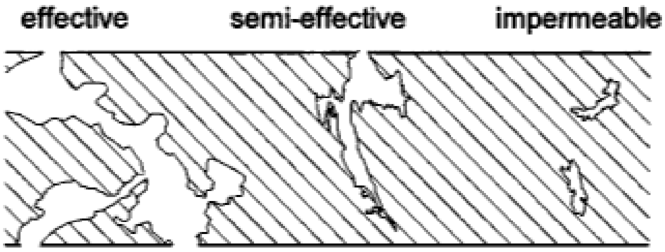


Figure 151. Air void types according to (Chen, Lin & Young 2004).

The effective air voids allow for water to drain through the pavement, and those are usually observed in porous asphalt structures. The semi-effective air voids are assigned to the structures of stone mastic asphalt mixes. The impermeable structure is in line with asphalt concrete mixtures.

Interestingly the air void structure is different when the samples are compacted in the laboratory and different when compacted in field. Some types of roller compactors are known to inflict small cracks, which increase connectedness between the air voids, leading to increased propensity for moisture damage (Caro et al. 2008).

12.1 Permeability, capillary rise, and diffusion

The air void content, size and connectedness influence the permeability and capillary rise in asphalt pavements. The third parameter governing moisture transport within asphalt is diffusion, but it is not only dependant on the air voids.

12.1.1 Permeability

Permeability of the pavement determines how soon the rainfall will drain down from the pavement. The optimum permeability of the pavement, assuring sufficient drainage was estimated to occur in pavements at around 8% air voids. The parameters influencing the permeability in field (Cooley, Prowell & Brown 2002) were proposed to be the maximum diameter of aggregate (D), air void content (Va), thickness of layer (h)

$$k_{field} = -5.335 + 4.61 \ln(V_a) + 0.138D - 0.024\,h \tag{29}$$

In this context, it is possible to optimize mixtures and pavements to either construct them in fully permeable or impervious regimes. The importance of the quality of construction work and mix design is underlined in this case.

Table 18. Classification of voids in terms of eprmeability (k) for asphalt mixtures (after Chen, Lin & Young 2004).

k (cm/s)	Permeable condition	Void	Mixture
≤ 10^{-4}	Impervious	Impermeable	Dense
10^{-4} – 10^{-2}	Poor drainage	Semi-effective	Stone mastic asphalt
≥ 10^{-2}	Good drainage	Effective	Porous asphalt

12.1.2 Capillary rise

The air voids in asphalt pavement can be in form of macropores, but also micropores. The capillary rise occurs. The capillary rise in asphalt pavements allows for water transport into the structure through the capillaries formed by the interconnected voids. The parameters governing the height to which the moisture is travelling above the saturation surface (h) are: the radius of the capillaries (r), the surface tension of water (σ_w), the density of water (ρ_w) and the contact angle between the liquid and solid (θ). The relationship between those parameters is given with the equation:

$$2\pi r \sigma_w cos\theta = \pi r^2 hg\rho_w \quad (30)$$

where g is acceleration due to gravity.

According to the surface tension, the water should not rise in capillaries when the bitumen is on the wall of the air void, due to the hydrophobic nature of bitumen. However, the water is in contact with mastic, and mastic as composed of the mineral material may inflict capillary rise. The studies on mastic types and distribution of air void content and size indicate that capillary suction can be minimized by correct mixture design (Masad, Arambula et al. 2007).

12.1.3 Diffusion of water vapour

The diffusion of water vapor into the asphalt mixture can occur either through the binder, as described in section 11.2.1 , or through pores. The water may adsorb on the surface of the pores and condense inside the structure (Caro et al. 2008; C. Hansen 2007).

It was postulated and proven, that in the regions in which limited rainfall is present the damage due to high moisture content in the air is plausible. The mixtures expressing moisture damage behavior tested in the high relative moisture environment expressed higher content of absorbed water into the structure. Not only the suction of the moisture into the asphalt proved indicative of propensity to moisture damage, but also the storage of moisture or drying capacity (Caro et al. 2008).

13 Introduction to Methods of evaluation of resistance to moisture damage – Part 4

The moisture damage in asphalt concrete leads to the deterioration of pavement performance. Therefore, an ability to predict the performance of the asphalt mixture is of high importance.

Within the Task 1 of Work Package 2 in the project BITU2020 a review of the theories explaining the aspect of moisture damage in asphalt pavements is provided. Both adhesive and cohesive failure is covered in that chapter.

The Task 2 of Work Package 2 in the project BITU2020, focuses on the review of methods applied for the evaluation of material's resistance to moisture damage. The review is divided into three parts:

1. Standardized methods used worldwide, with the focus on European Union and United States of America
2. The methods used in the past for the evaluation, especially if the methods were used during the ASTO project in 1989–1991 for the reason of the development of the Finnish Asphalt Specifications
3. The upcoming methods – described in the literature, but perhaps not widely applied in the industry.

The principle behind each method is described with appropriate literature review.

When possible, the evaluation of repeatability and reproducibility of the method is provided. Potential problems connected with the method, linking to the theoretical background, promising findings related to studied materials from international publications, as well as the research needs statement are provided for each method. This work comprises the scope of Task 3 in Work Package 2 titled "International Experience and challenges with prediction". Additionally, because often during the review of the techniques the modifiers are tested with considered method, the scope of work package 2 Task 6 "The link between the chemistry of the raw materials and moisture damage", is covered within this part of the BITU2020 project report.

The structure of this part of report is focused on collecting the information related to one testing method in relatively close proximity. Therefore, a method is described, discussed, the experiences worldwide and locally are presented, the repeatability of the method is discussed if data is available. After each method, suggestions in the context of Finnish asphalt practice, as well as questions arising from the reviewed literature related to the subject, are gathered.

14 Standardized methods

14.1 Indirect Tensile Strength Ratio (ITSR) (EN 12697-12 Method A)

The ITSR test is one of the most widely spread procedures to evaluate asphalt mixtures in terms of their resistance to moisture damage. The test is understood as performance predicting and is one of the parameters to report during the Type Testing of the bituminous mixtures (SFS-EN 13108-21) when the requirements in specific country of application exist. The test was developed as an alternative of the American Lottman test, which turned after evolution into one of the AASHTO standards (AASHTO T283).

14.1.1 Principle

Method A uses the indirect tensile strength of cylindrical specimens of bituminous mixtures. A set of cylindrical test specimens is divided into two equally sized subsets and conditioned. One subset is maintained dry at room temperature while the other subset is saturated and stored in water at elevated conditioning temperature. After conditioning, the indirect tensile strength of each of the two subsets is determined at a defined test temperature, depending on the grade of bitumen. The ratio of the indirect tensile strength of the water conditioned subset compared to that of the dry subset is determined and expressed in percent (SFS-EN 12697-12:2008).

14.1.2 Method variations and applicability to materials

14.1.2.1 The Lottman and modified Lottman test (AASHTO 7-283)

The Lottman test is performed according to the principle 14.1.1 on the three subsets of samples:

1. dry conditioned (control)
2. wet conditioned (field performance up to 4 years)
3. wet and freeze-thaw conditioned (field performance 4–12 years).

The Tunnicliff and Root conditioning pattern is performed only on two subsets of samples:

1. dry conditioned
2. wet conditioned.

The modified Lottman test, currently used as part of the American specifications (AASHTO T-283) is a combination of the above two methods, and consists of the:

1. dry Conditioning
2. freeze-thaw cycled and water conditioned.

The laboratory prepared sample is aged prior to compaction for a period of 16h in 60 °C in the compaction oven.

The temperatures of conditioning are respectively

1. room temperature but conditioned to 25 ± 0.5 °C prior to measurement for dry samples
2. vacuum suction of water into the pores with applied vacuum of 13–67 kPa
3. -18 ± 3 °C for freeze cycle for a 16 h with 10 ml of water in the plastic bag
4. 60 ± 1 °C for 24 h for thawing
5. 25 ± 1 °C for 2 h.

In the AASHTO T283 the degree of saturation is one of the parameters, which can disqualify the sample as an outlier. Specific dimensions of sample are defined. The differences in sample conditioning and dimensions in comparison to European and Finnish-modified procedures are gathered in section 0.

Finally, the Indirect Tensile Strength of dry and moisture conditioned samples is measured at 25 ± 0.5 °C.

14.1.2.2 European standard (EN 12697-12 Method A)

The European standard uses slightly different conditioning pattern, different testing temperatures, as well as different specifics of the sample. The subsets are conditioned in dry and wet conditions.

The water bath conditioning temperature for bitumen Penetration grade up to 100/150 is (40 ± 2) °C, but softer than 160/220 the conditioning temperature should be reduced to (25 ± 2) °C.

The Indirect Tensile Strength test temperature is chosen based on the bitumen grade

- For Penetration at 25 °C ≤ 70 (0,1 mm) → 15 ± 1 °C
- For Penetration at 25 °C ≥ 70 (0,1 mm) → 10 ± 1 °C
- For bitumens with kinematic viscosity at 60 °C ≥ 4000 mm2/s → 10 ± 1 °C

According to Finnish experience, the Indirect Tensile Strength for soft asphalt concrete samples (SA) prepared with V1500, V3000 or soft penetration grade bitumens 10 ± 1 °C is used (Makowska and Eskola, The bitumen related problems observed on soft asphalt concrete pavements: case study 2020). However, the applicability of the ITSR test is defined in Finnish Asphalt Specifications as only to SA-B, that is SA in which soft Penetration grade bitumen is used – not the viscosity graded bitumen (Finnish Pavement Technology Advisory Council 2017).

14.1.2.3 Finnish requirements and Finnish research

The ITSR test is initially used to establish the compatibility between aggregate and bitumen, but with a definition of the sample properties. Such definition is provided in instruction PANK 4301, suggesting using the aggregate of the gradation adhering to the defined porous asphalt gradation with the maximum aggregate size of 11 mm and a bitumen 70/100. The compatibility of the aggregate and binder is assured if the ITSR result is equal or above 70%.

For the type testing, currently in Finland the test SFS-EN 12697-12 method A, is the test required for the demonstration of the resistance to moisture damage. No specified freeze-thawing cycle is mentioned. The test is performed on the mixture prepared according to the mix design. In order to meet the specifications the AC and SMA mixtures need to retain equal or above 80% of strength after moisture conditioning ($ITSR_{80}$), while for SA-B mixtures the retained strength after moisture conditioning should be equal or above 60% ($ITSR_{60}$).

However, it was observed that in majority of the tests performed on the regular asphalt mixtures (AC, SMA) the retained strength is close to 100%, which causes issues in distinguishing between mixtures and trust in the reliability of the method (Laukkanen, Halonen & Pyy 2012). Additionally, with increasing number of freeze-thawing cycles (Laukkanen, Halonen & Pyy 2012) in Finland, the proposal was to include the step similar as in the modified Lottman test. The researchers proposed to include 10 freeze-thawing cycles and defined the special procedure in instruction PANK 4306.

However, due to the retirement of the primary investigator of the research and development project (Laukkanen, Halonen & Pyy 2012), accompanied by lack of driving force from the industry explained by the necessity to invest in the specialistic conditioning equipment, the method had not become the part of specifications still in 2017 (Finnish Pavement Technology Advisory Council 2017).

Some of the conclusions of the research project focused on localization of the EU method for the Finnish market, included:

- need to control the level of saturation below 100%, due to the fear of water carrying a load during the test
- need to define the target saturation level of specimens
- necessity to group the samples so that their air void content and bulk density were on the similar level in dry and wet subset
- definition of the freeze-thawing cycling procedure, assuring the saturation level was controlled for
- need to conduct further study to redefine the requirements for local mixtures based on the systematic and statistical review of results coming from the quality control testing performed according to the modified method.

The biggest problem identified for the method SFS-EN 12697-12 method A was the oversaturation of samples to the level of 100%. It was argued that when the water fills the pores of specimen, the resistance to momentary rapid loading is higher in the specimen, than in the condition of lower saturation. It was argued that the standard provides systematic incorrect results due to the lack of control of this parameter.

14.1.2.4 Polish research, Instruction and requirements

The necessity of switch towards European standards influenced procedures in other countries, among them Poland (Zofka, Maliszewska, Horodecka et al. 2013). The researchers conducted survey of the requirements related to ITSR results in 15 European countries.

Interestingly, despite the fact, that Norway, Sweden and Estonia took part in the survey in 2012, the presence of freeze-thawing cycle in the testing procedure was reported only by Finland (PANK 4306), Turkey (AASHTO T283) and Poland (WT-2 2010). For that reason, the experiences reported by the researchers (Zofka, Maliszewska, Horodecka et al. 2013) comprise valuable source of comparative experience for Finnish researchers.

What is worth to underline in terms of experiences from other countries, based on the results of the survey, is that Finland is not specifying the moisture resistance of the mixtures for courses other than wearing courses (see Table 19), when compared to other Nordic countries and Poland. The lack of quality requirements for Finnish termed mixtures of type “AC bin” and “AC base” could explain why the moisture damage is occasionally observed beneath the wearing course layers in field studies (Aaltonen 2014) (Pellinen, Makowska et al. 2013). Additionally, considering the allowed staged construction of the pavements, the lack of moisture resistance requirements for the layers left over 1–3 years before the wearing course is applied (Liikennevirasto 2018), may be the culprit of failures.

Table 19. The chosen answers from the survey conducted by (Zofka, Maliszewska, Horodecka et al. 2013).

Country	UE/ nonUE	CEN	Wearing course	Intermediate course	Binder Course	Freeze-thawing cycle
Estonia	UE	Yes	$ITSR_{90}$ $ITSR_{60}$ (PA)	ITSR $_{80}$ ITSR $_{85}$ (if SMA left as wearing)	$ITSR_{60}$ $ITSR_{80}$ (if the layer stays over-winter without over-lay)	-
Finland	UE	Yes	$ITSR_{80}$ (AC, SMA) $ITSR_{60}$ (SA)	-	-	10[1]
Norway	nonUE	Yes	$ITSR_{70}$	$ITSR_{70}$	$ITSR_{70}$	0
Poland	UE	Yes	$ITSR_{90}$	$ITSR_{80}$	$ITSR_{70}$	1
Sweden	UE	Yes	$ITSR_{75}$	$ITSR_{75}$	$ITSR_{75}$	0

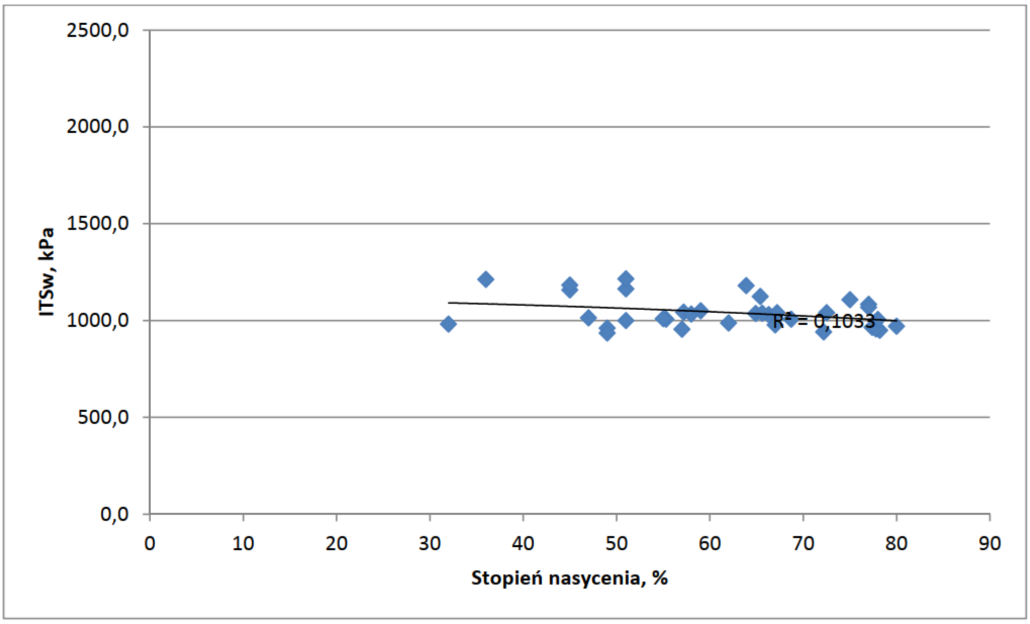
[1] The planned number of cycles in 2012 was 10, yet due to the internal issues the plan was not realized and currently 0 cycles is performed.

As a result of the research project, it was demonstrated that including just one freeze-thawing cycle decreases the ITSR significantly in comparison to non-freeze-thaw cycled experiments (Zofka, Maliszewska, Horodecka et al. 2013). The suggestion was to lower the ITSR requirement for Poland and to remove the freeze-thaw cycling, for the Polish tests to be in line with tests conducted in other European countries. Considering the marketability of the asphalt mixtures and the sales between the countries, such advice is understandable.

The Polish procedure was in fact a combination of the EU and AASHTO procedure. The procedures are compared in Table 23. It becomes apparent that comparison of the results coming from different procedures of sample conditioning, provide differing results.

Interestingly, during optimization of the procedure, it was found that:

- Saturation level is optimal between 70–85%.
- The size of the sample is a significant parameter 63.5 ± 2.5 mm thickness was optimal for laboratory prepared samples.
- That the control of the temperature of the baths in the laboratory may have caused errors – suggested is setting the target, e.g. 25, and the phrase "±2 °C" is defined as a measuring tool uncertainty defined during calibration operations.



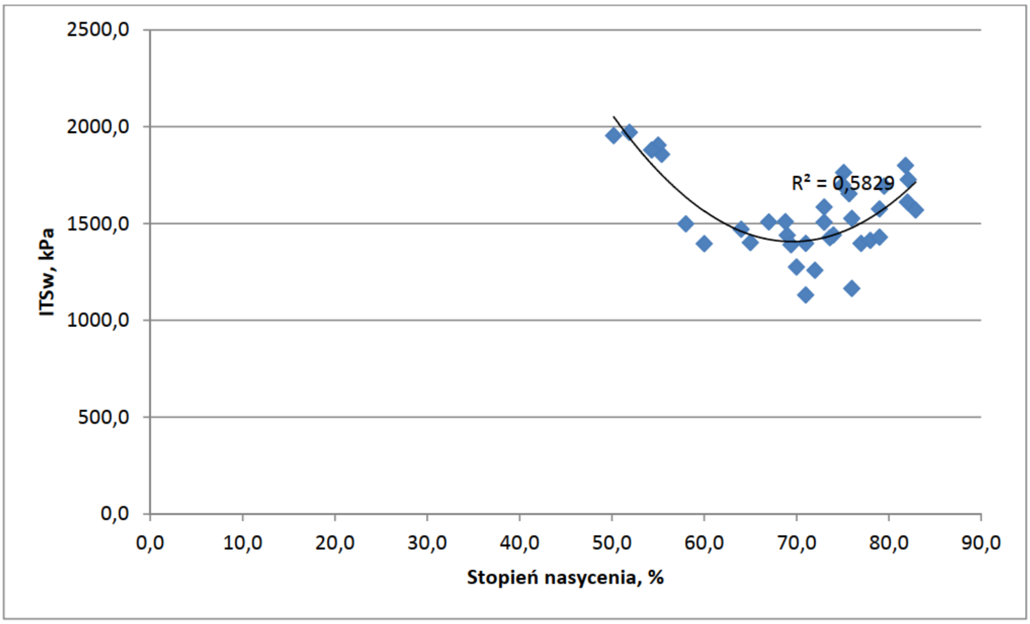


Figure 152. The relationship between saturation level (x axis) in the sample and Indirect Tensile Strength after moisture conditioning for SMA11 (upper frame) and AC (lower frame) mixtures (Zofka, Maliszewska, Horodecka et al. 2013).

14.1.2.4.1 Effect of additives on ITSR

After optimization of the testing method of ITSR described above the resistance of various mixtures to moisture damage was evaluated using a complex experimental matrix (Zofka, Maliszewska and Horodecka et al. 2013). The test performed on the mixtures not only included evaluation of the effect of the aggregate, binder grade, type of mixture, but also the type of an adhesion promoting additive. Additionally, the combinations were evaluated using other characterization methods, such as rolling bottle test, Hamburg wheel tracking and TSRST, and the results from those tests are brought up in the respective sections.

The experimental matrix is presented in the Table 20. The liquid additives were added at 0.3% of bitumen mass, while hydrated lime was added at 1.5% of asphalt mass.

Table 20. The matrix of the sample code names in the Polish study (Zofka, Maliszewska, Horodecka et al. 2013).

Adhesion promotor	SMA11 surf 45/80–55			AC16 bind 50/70			AC22 base 35/50	
	Basalt (B)	Gabbro (G)	Granite (GR)	Dolomite (D)	Amphibole (A)	Quartzite (K)	Limestone (W)	Paleobasalt (M)
-	B	G	GR	D	A	K	W	M
Wetfix BE (W)	B/W	G/W	GR/W	D/W	A/W	K/W	W/W	M/W
Teramin 14 (T)	B/T	G/T	GR/T	D/T	A/T	K/T	W/T	M/T
AD-HERE (A)	B/A	G/A	GR/A	D/A	A/A	K/A	W/A	M/A
INTERLENE IN/400 L (I)	B/I	G/I	GR/I	D/I	A/I	K/I	W/I	M/I
PE-31 (P)	B/P	G/P	GR/P	D/P	A/P	K/P	W/P	M/P
Hydrated lime (WH)	B/WH	G/WH	GR/WH	D/WH	A/WH	K/WH	W/WH	M/WH

As can be seen from Figure 153 – Figure 155 the effect of the aggregate can be strong on the results of the ITSR. The dolomite aggregate expressed the lowest resistance to moisture. However, in Finland dolomite aggregates are rare and their use is insignificant. Additionally, the specifications in regards of ITSR are only defined for the wearing course layers. It was postulated during the stakeholder meeting of BITU2020 project that the results comparable to the raw materials of interest in Finland are connected only with the Gabbro and Granite aggregates. Nevertheless, the situation presented below indicates that for each type of aggregate, different adhesion promotor results in either decrease of the moisture resistance, small improvement or large improvement, as witnessed by the ITSR result. This underlines the necessity to test the adhesion promotors in considered conformation of design parameters, such as mix design, bitumen type and adhesion promotor.

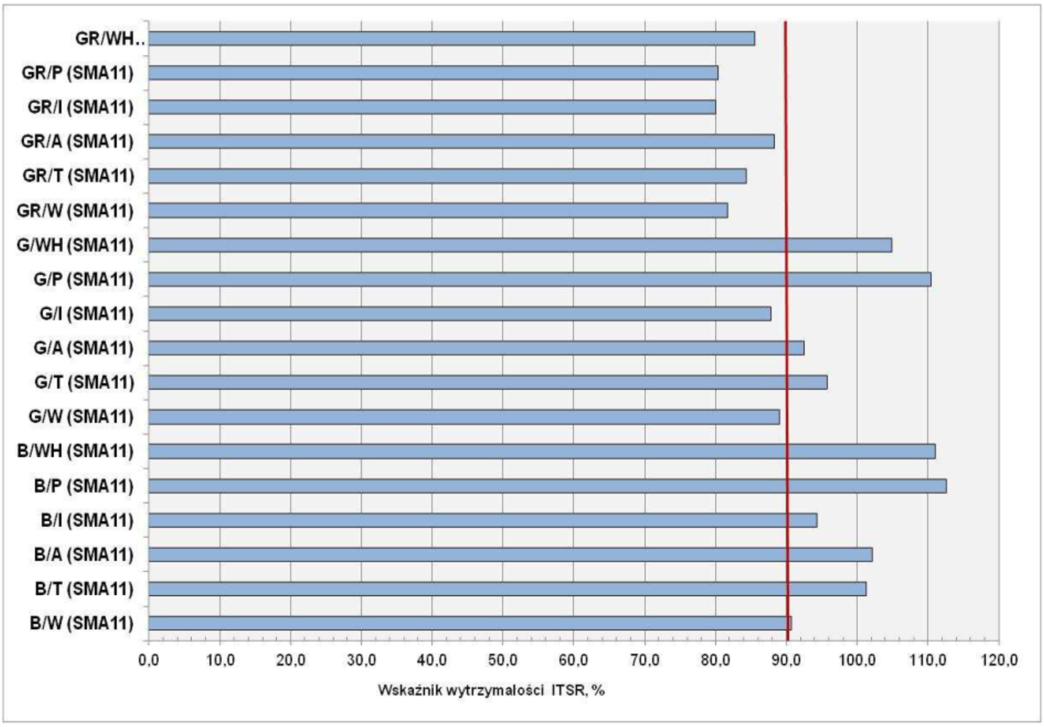


Figure 153. The results of the ITSR procedure for wearing course layer SMA11 plotted against the Polish requirement for considered asphalt mixture type (Zofka, Maliszewska, Horodecka et al. 2013).

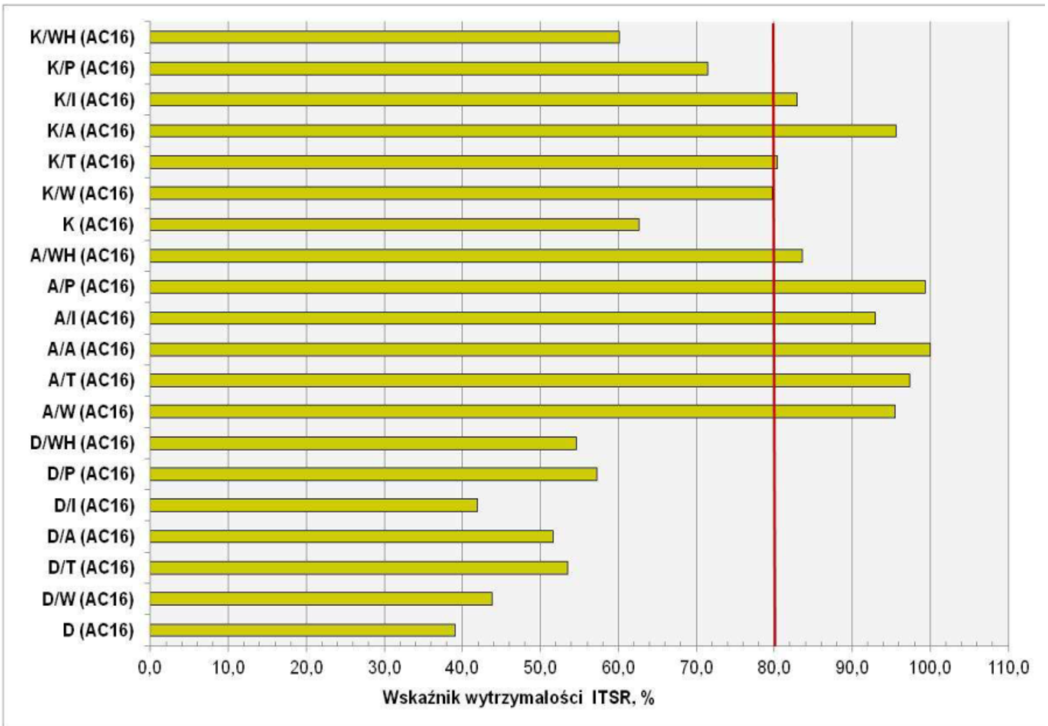


Figure 154. The results of the ITSR test for the binder course layer AC16 plotted against the Polish requirement for considered asphalt mixture type (Zofka, Maliszewska, Horodecka et al. 2013).

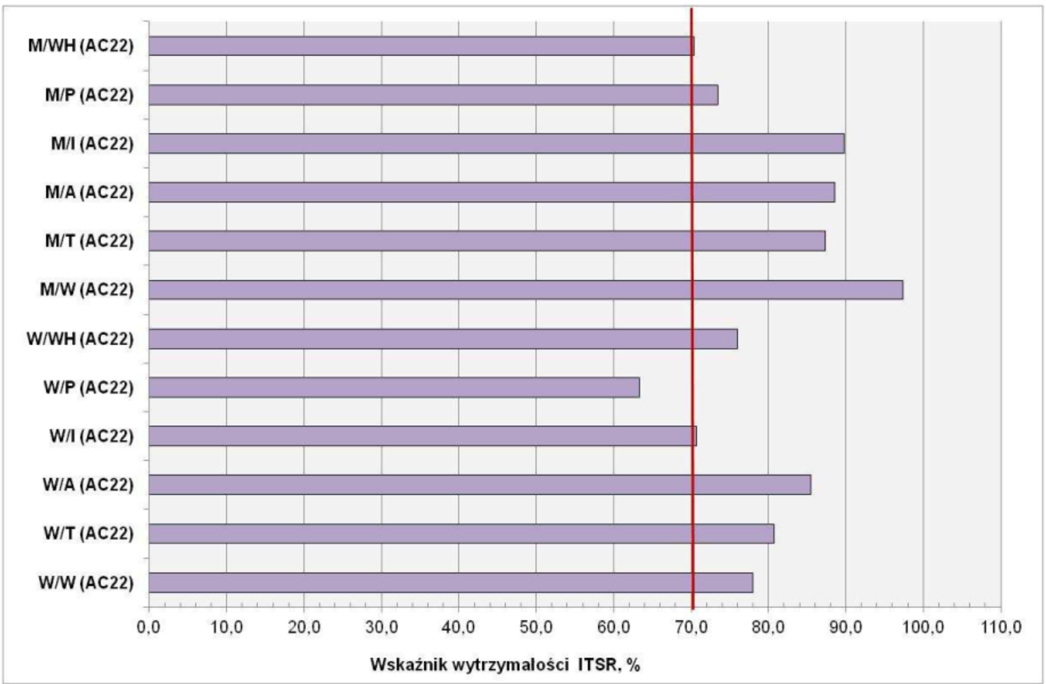


Figure 155. The results for the ITSR test on the base course layer AC22 plotted against the Polish requirement for considered asphalt mixture type (Zofka, Maliszewska, Horodecka et al. 2013).

Based on the above results, the researchers suggested to use Ad-here adhesion promoter, if one type of product is desired for all mixtures. It was not the product improving best for all the mixtures, but it also did not cause decrease the properties in any case. The use of other promotors would require consideration on compatibility with considered aggregate.

Interestingly, as is visible in Figure 153, the SMA11 mixtures also express the ITSR values above 100% in this study. Similar has been observed in ASFADUR project for SMA16 mixtures even after 10 cycles of freeze-thawing (Laukkanen, Halonen & Pyy 2012).

What could be the culprit of this strange behavior? Both countries are suggesting the use of cellulose fibers in SMA mixtures. The elastic modulus of wood depends on the direction of fibers alignment along the axis of applied force. Could such a small amount of fibers, increase the tensile resistance of asphalt concrete, if in one sample set the alignment is along the longitudinal direction and in the other sample in perpendicular direction to the force? However, the wet cellulose fibers lose their tensile strength. Is this strange ratio upkept above 100% simply connected with inhomogeneous distribution of fibers between sample sets, leading to varying mastic composition and strength of asphalt concrete composites?

14.1.2.5 Swedish research related to moisture conditioning

In 2013, a comprehensive project was performed in Sweden in respect of adjustment of the local specifications of bitumen products, and unifying the standards to be in line with the harmonized bitumen classification standard EN 12591: 2009 (Olsson, Krona & Nordgren 2010).

The seven different origin bitumens of the same Penetration grade 70/100 were investigated for their resistance to moisture damage. In 2008–2009, the field tests with the chosen bitumens were conducted. The field tests were accompanied by the laboratory analysis, in order to find the best correlating method to the field performance (Olsson, Krona & Nordgren 2010). In 2016 and evaluation of the state of the test roads and retesting of the samples was conducted (Olsson & Witkiewicz 2017).

Initially, it was found that using the regular ITSR was not differentiating between the bitumens. The results varied between 85–101% of retained ITS after conditioning and, as was provided in Table 19, the $ITSR_{75}$ was an applicable requirement in Sweden at that time. Even after conditioning of the samples for 7 days in a similar manner as was developed in Asfadur project (Laukkanen, Halonen & Pyy 2012), but cycling between -20…20 °C, the ITSR ratios were in between 90–99% (Table 21) (Olsson, Krona & Nordgren 2010).

The biggest differences between the binders were visible in Hamburg Wheel-track test and Rolling Bottle test. However, after 72 h of rolling all binders resulted in 0% coverage, thus the results from 72 h were not differentiating well.

In the study from 2016, conducted on the cores collected from the test roads for each of the bitumen, the situation was different. The cores collected were retested according to the procedure of the standard 12697-12 Method A and results now varied between 67–94% of retained strength (Table 21). This can be compared against all the provided rheological data and chemical composition of binders used in the study (Table 22).

The binder Bit5 was deviating from the binder specifications in force before 2010 in Sweden, by means of too low dynamic viscosity at 60 °C. It was the binder with highest increase in softening point during RTFOT and after 1 year in field, as well as after 8 year in field. However, in terms of resistance to moisture damage, abrasion by studded tires, creep, rolling bottle test and wheel track, when compared to other binders, it was evaluated in the laboratory that binder 5 predicts best performance. The order of performance based on the set of laboratory evaluations was from best to worst: 5 > 7 > 3 > 6 > 4 > 2 > 1.

Table 21. The results of the Indirect Tensile Strength for samples conditioned according to EN procedure, including the 7 days of freeze-thawing (FT) and after 8 years in service, compared against basic binder characteristics.

Binder	Pen	SP	ITSR	ITSR +FT	ITSR 8y	ITS dry	ITS wet	ITS Dry FT	ITS wet FT	ITS dry 8y	ITS wet 8y
	0,1 mm	°C	%	%	%	kPa	kPa	kPa	kPa	kPa	kPa
Bit1	71	47,0	85	90	70	2729	2329	2475	2227	2703	1882
Bit2	83	45,7	91	91	94	2232	2023	2154	1969	1671	1578
Bit3	83	45,9	101	95	67	2059	2088	1965	1863	1971	1329
Bit4	72	46,8	91	94	69	2582	2339	2572	2428	2163	1493
Bit5	83	45,7	90	99	84	2407	2176	2217	2196	1732	1462
Bit6	80	46,6	93	98	73	1877	1753	1832	1801	1663	1219
Bit7	84	45,9	94	93	81	2299	2163	2224	2068	2277	1847

Bit5 – considered not meeting the binder specifications in Sweden prior to 2010

Table 22. The Penetration (Pen) and softening point (SP) of the tested binders before production, alongside SP after 8 years in service, accompanied with the fractional composition (S– saturates, A – aromatics, P1 – polar I/"resin", P2 – polar II/"asphaltenes") of binder before and after 8 years in service.

Binder	Pen	SP	ΔSP RTFO	ΔSP 1y	Va 8y	ΔSP 8y	S 0y	S 8y	A 0y	A 8y	P1 0y	P1 8y	P2 0y	P2 8y
	0,1 mm	°C	°C	°C	%	°C	%	%	%	%	%	%	%	%
Bit1	71	47,0	4	5,0	3,0	4,1	7	9	52	37	26	35	16	19
Bit2	83	45,7	5	3,4	3,8	5,3	3	6	51	40	27	35	19	19
Bit3	83	45,9	5	2,5	4,5	9,7	6	6	55	34	20	42	19	19
Bit4	72	46,8	5	3,0	3,8	4,6	7	6	52	40	25	39	16	15
Bit5	83	45,7	6	5,2	4,3	10,1	8	12	58	37	19	36	18	15
Bit6	80	46,6	5	4,1	2,1	2,0	5	13	58	41	22	28	15	19
Bit7	84	45,9	5	3,7	2,8	8,1	8	6	54	41	20	31	18	22

Bit5 – considered not meeting the binder specifications in Sweden prior to 2010

Because the thickness of the samples in ITS are different between the test sites the comparison of the strength is troublesome. However, it would be worth noting that the strength of those samples after 8 years in field is rather low to start with in the dry form. After further conditioning the decrease of strength is not apparent and on the level of 84–94%. While for the other binder types, the strength of the samples is significantly higher and decreases relatively more due to moisture presence. In line of the results from the Polish researchers, the control of the thickness of the samples compared between sites in the future when considering the research plans, could be of high interest.

14.1.2.6 Summary

The Table 23 presents a comparison between different adaptation of a test known as the Indirect Tensile Strength Ratio test for bituminous mixtures. Based on the survey conducted by Zofka et al. (2013) Poland is considered as having modified procedure to the European one, while Finnish methods are divided into currently used and the developed PANK 4306 method, which is not in use at the moment. The three different European methods are compared against the American AASHTO T283 methodology.

Table 23. Differences between the ITSR methods in Europe and America.

	Unit	American AASHTO T283	EU& Finnish current 12697-12A	Finnish-mod (PANK 4306)	Polish-mod (WT-2 2014 cz. I)
Thickness lab/slab	mm	63.5±2.5		70-75 raw 60-65 cut	63.5±2.5
Saturation degree	%	≥70		Equal and ≤100	Suggested ≥70
Aging T	°C	60 (loose mix)			Optional
Aging t	h	16±1			Optional
Compaction method		Marshall	All	All	Marshall 2x35 or gyratory 40 rounds (150mm)
Precomp. T	°C	Tc±3			Tc according to bitumen producer
Precomp. t.	h	2± 1/6			
Curing PP T	°C	20±5	20±5		
Curing PP t	h	24	16-24		
Air voids of lab samples	%	7	NS	8-10 (by dimension)	Design
Dry cond. T	°C	20±5 →25 °C for 2 h before test	20±5 →25 °C for 2 h before test	5 °C	20±5
Vacuum p	kPa	13-67	6,7	6,7	6,7
Vacuum T	°C		20±5	20±5	20±5
Wet T	°C				40
Wet t	h				68-72
Freeze cond. T	°C	-18±3		-18…-22	-18±3
Freeze cond. t	h	16			16
Freeze-thawing t	h			12 or 24 (method A and B)	
Wet cond. T	°C	60	40 or 25	4…23	60
Wet cond. t	h	24	72±2		24
Number of freeze-thaw cycles		1	0	10	0-1

	Unit	American AASHTO T283	EU& Finnish current 12697-12A	Finnish-mod (PANK 4306)	Polish-mod (WT-2 2014 cz. I)
ITS T	°C	25	15 Or 10	10	25
Rate of movement in ITS	mm/min	50	50	50	50

PP – post production
Tc – compaction temperature

14.1.3 Repeatability and reproducibility

According to the standard SFS EN 12697-12, the repeatability for Method A is on the level of r = 15%, and reproducibility is on the level of R = 23%. Because of that, the reliability or trust in results of the test is quite low.

However, the problem with the test is rather not in the repeatability of the interlaboratory measurements. The repeatability and reproducibility can be improved by unifying instructions (Zofka, Maliszewska, Horodecka et al. 2013).

The problem is more connected with the lack of the link between the results of ITSR test and field performance. The mixtures evaluated in ITSR often result in values close to 100% (Laukkanen, Halonen & Pyy 2012; Olsson, Krona & Nordgren 2010; Pylkkänen & Kuula-Väisänen 1989). This does not allow the practitioners to distinguish between well and bad performing mixtures, which results in lower trust towards the methodology.

It is worth to note in this place, that the typical research studies investigate artificial mixtures in which typically fillers are not used, and an aggregate comprises of one material type. The mixtures prepared for field applications may consist of such additives as limestone fillers, which are considered based on the results as improving the moisture resistance of the mixtures (Kurki, Halttunen et al. 1993). Similar problem is observed for rolling bottle test, which also usually does not include the fine material during the analysis (see section 14.3) (Finnish Standards Association 2018).

14.1.3.1 Semi-circular Bending Test (SCB) as an alternative to ITS

It has been also underlined that the repeatability of the Indirect Tensile Strength test itself is limited. During the test the sample may dislocate, which is called "wedging" (see Figure 156 right). The failure in some samples was suggested to occur in a compression rather than tensile mode. For that reason a simpler and more repeatable test, known as semi-circular bending test was developed.

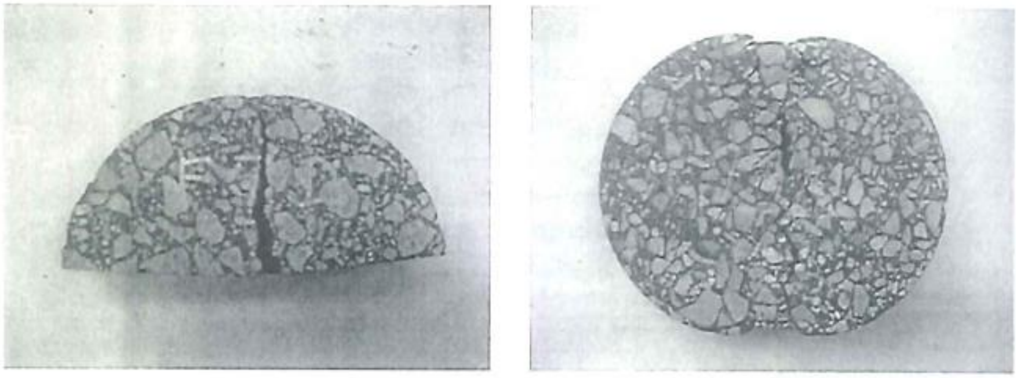


Figure 156. Differences in cracking of a SCB and ITS test (after Molenaar et al. 2002)

The sample can still be prepared in the compactor or drilled from the road. Then the sample is cut in two pieces to result in two semi-circles. In the middle of the semi-circle a notch (crack initiating point) can be prepared, by e.g. sawing. Afterwards, the sample is placed on two points, while the load is applied from the third point on top (see schematic in Figure 157). The crack propagation happens in much more predictable way, with the failure mode being mostly due to indirect tensile forces (Molenaar et al. January 2002). The repeatability of this test was found much better than for the ITS (Figure 158) (Li & Marasteanu 2010). The value of the tensile strength from SCB is numerically bigger than from ITS, and this was postulated to be due to the lack of the wedging.

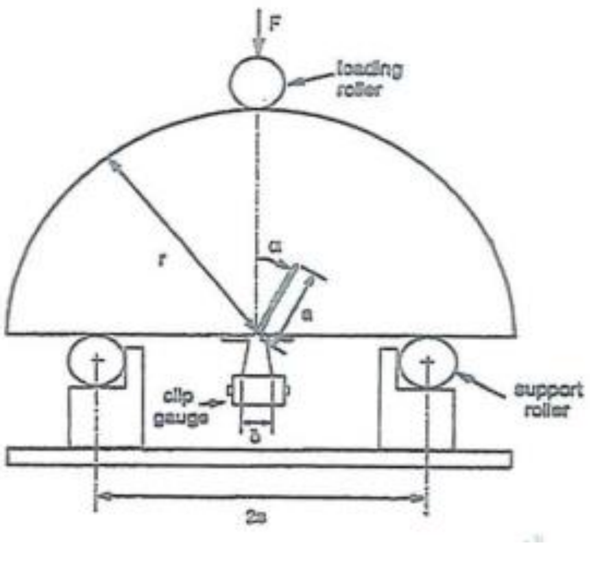


Figure 157. Schematic of the semi-circular bending test set-up. Reprinted from Molenaar et al. (2002).

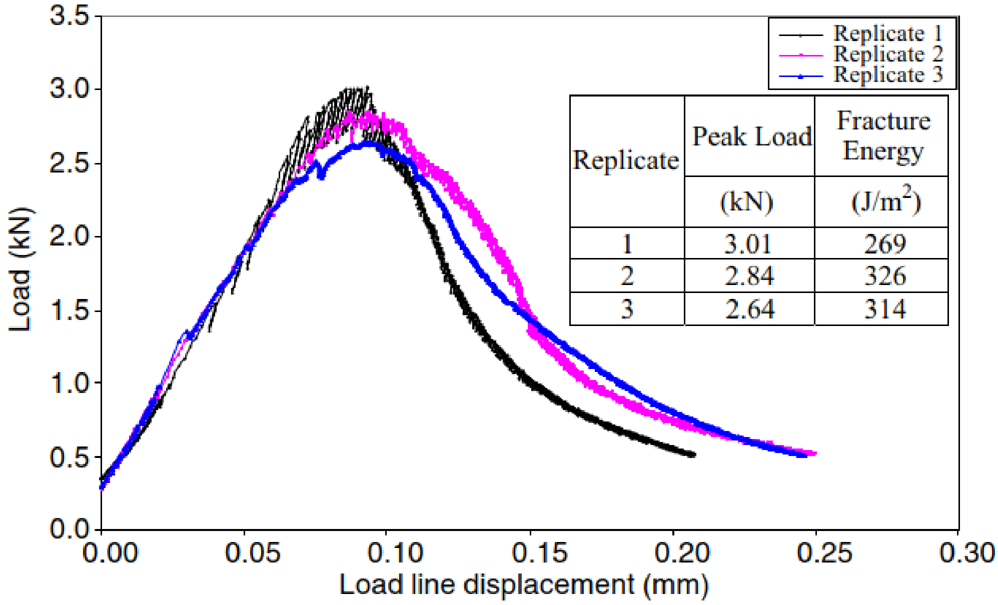


Figure 158. Repeatability of the SCB test performed on the three replicates. Reprinted from Li & Marasteanu (2010).

The test can be also utilized for studying of the low temperature cracking. The same equipment could be used to study fracture energy, the tensile strength and low temperature cracking (Li & Marasteanu 2010). The three-point bending, typically used in studies of fatigue can be performed in the same equipment.

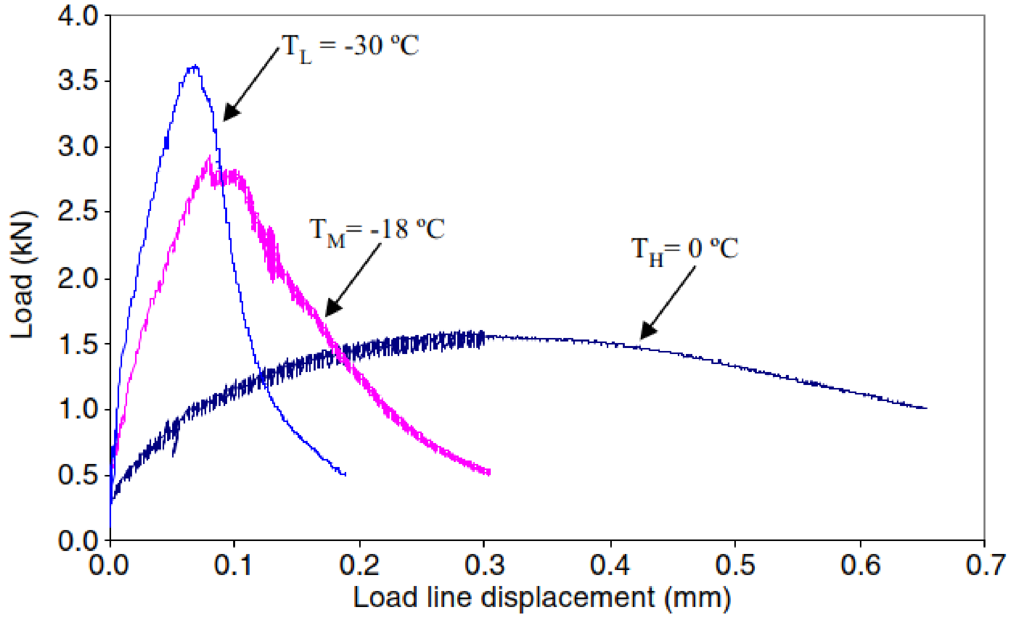


Figure 159. The typical plot of loading with load line displacement, demonstrating the effect of the temperature of the load-displacement curve shape. Reprinted from Li & Marasteanu (2010).

In terms of suggestion for the Finnish Transportation Agency, it would be advisable to investigate if this testing method could replace the current ITS method in the ITSR tests in the future. The moment when the mixture would be optimized for low temperature cracking, fatigue and resistance to moisture, ideally one testing equipment could provide analytical capabilities for a laboratory.

The method was demonstrated to distinguish between binder types, aggregate types, content of air void in terms of tensile strength and fracture energy (Li & Marasteanu 2010). The method has its own parameters, and the most important one is the notch depth.

14.1.4 Other international experience

Some of the other experiences connected with ITSR from Norway (Anastasio et al. 2015) and China (Li & Li 2016) were also found, but are reported in the section connected to Cantabro test (16.1.1). The authors of those articles focused on underlining how ITSR is perhaps a not the best differentiator between mixtures and how the Cantabro test, or even looking at the numerical value of strength (not the ratio) could be of more use in prediction of propensity for raveling.

14.1.5 Problems related to method and research needs statement

Based on the above research efforts, the conclusion is clear that control of the test parameters in laboratory is crucial to achieve repeatable result. However, not much of the literature exists on the actual link between the results of the ITSR and the field performance. The optimization of the procedure, if considered, should take that aspect into account.

14.2 MYR-test (EN 12697-12 Method C)

14.2.1 Applicability to materials

The name of the test used in Finland comes from the abbreviation of the name of the developer, that is Matti Ylä-Rautio (MYR). The Finnish Asphalt Specifications use the MYR-value to determine the moisture damage susceptibility of Soft Asphalt Concrete (SA) (Finnish Pavement Technology Advisory Council 2017).

The soft asphalt concrete can be prepared using lower temperatures than typical hot-mix asphalt. This is linked with the rheological properties of the binder, namely V1500 or V300 in case of SA-V, or 250/300…650/900 Penetration grade soft bitumen in case of SA-B. The previous research indicated that road oils, cutbacks and emulsions can be used as a binder in the SA types of mixtures (Apilo 1996; Simonen 2011), but the curing time needs to be considered in case of those structures.

As a result of the fact that the SA has a very low tensile strength, the reliability of the ITSR for this type of asphalt concrete is very low. The MYR-test is considered simple and quick, as it does not require special mechanical equipment and lasts for up to few minutes. It was performed for year and its results can be linked empirically to the well performing pavements (Apilo & Eskola 1998). It is suggested for use during the mix design, as well as during the production as a quality control. The need to use the adhesion promotor in the mixture is based on the results of this test, if the bonding value is above or equal to 2g (Finnish Pavement Technology Advisory Council 2017).

14.2.2 Principle

The 1000 g of loose asphalt mixture stabilized for a period of 60 minutes from preparation to allow the initial aging and adhesion. The whole mass is placed into the graduated beaker and a 1500 ml of water (temperature of 25 ± 3 °C) is poured into it as well. The beaker is sealed with a rubber stopper.

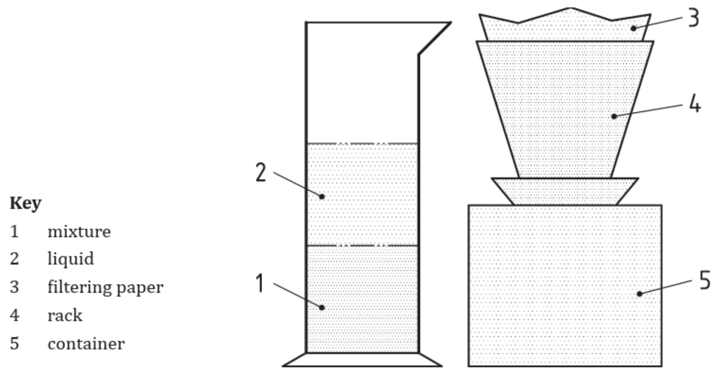


Figure 160. The equipment used for the MYR-test (SFS-EN 12697-12 Method C) (Finnish Standards Association 2018).

The beaker is moved upside down relatively fast 10 times, allowing each time for the mass of asphalt to drop to the bottom. A rest period of 60 seconds is applied, and a series of 10 upside down turns is repeated. Afterwards, immediately after assuring that all loose debris from the wall is in the water, the liquid is poured over the previously prepared filter paper until the mark on the beaker is 1000 ml.

The bonding value is defined as a mass of debris that stays on the filter paper after drying to a constant mass. This debris can constitute of bitumen, or bitumen and fines, etc., but connects to the loss of adhesion and cohesion in asphalt concrete sample.

14.2.3 Repeatability and reproducibility

The information provided in the SFS-EN 12697-12 indicates that for the bonding value below or equal to 2 g, the value ≤ 0.3 g provides 95% confidence interval.

14.2.4 National experience

The main requirements for resistance to moisture damage of fines are defined by water adsorption values (Finnish Pavement Technology Advisory Council 2017). Performing water adsorption test (PANK 2108) requires complicated equipment (BET-surface area testing) (PANK 2401), which requires bottles of nitrogen and liquid nitrogen in the laboratory. Therefore, the threshold to perform the water adsorption test may be high in the industry. The MYR-test on the other hand, requires a large glass beaker, access to water, rack, beaker and a filter paper.

Nevertheless, the link between MYR and water adsorption value exists. However, the value of water adsorption defined for SA types of pavements is lower than the suggestion for general mixtures. This may originate in the difference between the studied fractions, as discussed further in connection with past Finnish research projects (see section 15.3.4.3 This was the reason why the study of SA pavement and the cut-off value of water adsorption for them was studied separately (Apilo & Eskola 1998).

Because the maximum grain size studied in PANK 2108 is smaller than that in previous studies (passing 0.075 mm) (Kurki, Halttunen et al. 1993), it is envisioned that both surface area of the fines passing 0.063 mm is larger, and so is the adsorbed mass of water into the mass of fines. However, this is not resulting in the water adsorption value increase for finer material (Pylkkänen & Kuula-Väisänen 1990).

Nevertheless, 27 different aggregate sources were investigated for the purpose of establishing the performance predicting parameters for the SA type mixtures (Apilo & Eskola 1998). The study defines the design method for this type of pavement based on the concept of voids filled with bitumen, comparing the fine material volumetric properties. Additionally, the comparison of the fine material properties such as surface area and water adsorption was compared with the experience related to field performance, and the MYR-value obtained during the production of the pavements. The conclusion was that the water adsorption value below 5 mg/m^2 was related with good performance and good MYR-values. In the case of the water adsorption above the 7 mg/m^2, both MYR-value and performance related to moisture resistance were inadequate. In the cases with water adsorption above 7mg/m^2 the use of adhesion promoting additives was necessary. The good MYR-value was defined as below 0,5 g, acceptable on the level of 0,6–2 g, and the unacceptable on the level above or equal to 2,1 g (Apilo & Eskola 1998).

As a result of the study, for the SA type pavements, the moisture resistance is considered adequate when the water adsorption (PANK-2108) (Päällystealan Neuvottelukunta ry 2009) is below the level of 5 mg/m^2. For the mixtures with water adsorption value above 7 mg/m^2, the MYR test is required to establish necessity for adhesion promotor, as well as to establish the required amount of the adhesion promotor. The MYR-value for PAB-V and PAB-B mixture should be below or equal to 2.0 g (Finnish Pavement Technology Advisory Council 2017).

However, the requirements for the water adsorption are related to the raw materials, compatibility of materials and mix design stage. It was observed during the research, that the biggest effect on the adhesion between unmodified bitumen and aggregate came from the production process and temperature of production. One of the production options for this type of mix include the use of steam for heating purposes. Additionally, because SA type mixtures are considered the cold or warm mixes (SA-V mixtures can be prepared at 40–120 °C, while SA-B at 110–155 °C (PANK ry 17.4.2018)), the risk of the insufficient drying of the aggregate prior to coating and presence of water at grain-binder interface is high. Due to the choice of the process, the need for adhesion promotor may be higher than it would be apparent from the raw material characterization. It was advised to use the MYR-test during the production to assure that used process is still resulting in an asphalt mixture with adequate moisture resistance properties (Apilo & Eskola 1998).

14.2.5 Problems related to MYR method and research needs statement

The method itself is considered simple and fast. There is low threshold for the quality control laboratory to conduct the measurement when requested. The evaluation of repeatability and reproducibility could be documented in a more transparent way, tough. However, it is apparent that the MYR-value seems to be the internal evaluation criteria within Finland alone. The studies on the SA type pavements in other countries tend to test the ITSR rather than MYR-value for SA pavements (Vaitkus et al. 2016).

The economic and social problem related with the SA pavements is related to the decreased production of this type of wearing courses in the past decade in Finland, due to the optimization of the infrastructure maintenance budget towards repairs of the high-volume roads. There is a risk that the know-how related to the production of this material may be decreasing over time in the industry with the retirement wave or the professionals born soon after World War II. Additionally, the reformulation of the Finnish Asphalt Specifications between 1995 and 2017 removed some of the requirements set forward to the SA pavements from the original work of Apilo and Eskola (1998), reformulated the recommendations and spread them into different sections of the Specifications (Finnish Pavement Technology Advisory Council 2017; Rakennustietosäätiö RTS 2010), which may lead to misunderstandings and confusion.

According to the Table 31 in Finnish Asphalt Specifications (Finnish Pavement Technology Advisory Council 2017), the MYR-value must be reported for the mixture during production. Additionally, according to the Code of Building Practice for Infrastructure (Rakennustietosäätiö RTS 2010) publication, which lists the quality control requirements for the road related materials, it is stated that:

- For SA-V mixtures after the addition of the adhesion promotor **and** every 500 tons of mass, the "bucket test" (*fin.: sankokoe*[2]) should be performed.
- If the resistance to moisture is low, the MYR-test **should** be performed.
- For SA-V prepared using emulsion as a binder, the MYR-test **must** be done always during mix design.
- For cold or warm mixed SA-V mixtures and emulsion-based SA, MYR must be done every 5000 tons apart.
- Every asphalt plant (preparing the SA mixture) must do at least one MYR-test.

The need to establish the level of adherence to the requirement came forward during the preparatory projects prior to BITU2020 (Makowska & Eskola 2020) and became one of the focus points of the Work Package 2 – Task 4 of project BITU2020. The clarification of the specifications may be advised.

Additionally, the raw material change has not been taken account over the last two decades. After the research program from 1989–1992, the main source of the bitumen on the market had been supplied by one refinery until 2014, with a few other producers. The research project related to the SA-V pavements focused on differentiating between 27 different aggregates (Apilo & Eskola 1998). No differentiation between bitumen sources or producers occurred.

In the following years, the research on the SA type pavements focused rather on the development of the binders, and again rather one source of bitumen and dif-

[2] "the bucket test" (*fin.: sankokoe*) – the mass of SA is placed into the bucket filled with water and stirred with the hand or stick; if the water is dirty the adhesion is weak

ferent fluxing solvents were used in the investigations. The MYR-values reported for the mixtures were low (0–0.2g), but unfortunately the value for the reference mixtures, without fluxes or not emulsion based, was not reported (Simonen 2011). Again, in the cutback bitumen study (Simonen 2011) the base bitumen source was provided by one producer. Therefore, it is hard to conclude whether the bitumen source affects the adhesion for SA mixtures. The effect of the binder source as a parameter should be established.

14.3 Rolling Bottle Method (RBT)

The Rolling Bottle Method is one of the three methods focused on the determination of the affinity between bitumen and aggregate (SFS-EN 12697-11 Method A). The two other methods, namely Static Bottle and Boiling Water Stripping method are discussed later on.

14.3.1 Principle

An aggregate sample of a grain size 8/11 mm (SFS EN 13043) is covered with bitumen while hot. The grains are then cooled down assuring that they stay separate. A bottle (Figure 161) is filled with loose covered particles and a portion of clean distilled or deionized water (temperature of 5 ± 2 °C). Such prepared material in a bottle is placed on the rolling bottle machine, and rolled with the speed dependent on the type of binder (40 or 60 min^{-1}).

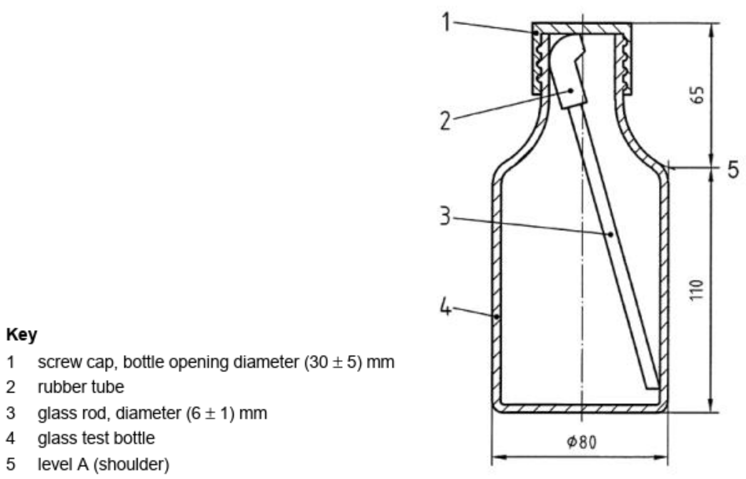


Figure 161. The schematic of a bottle used during the Rolling Bottle test (SFS EN 12697-11)

The rolling continues for 6 h, before first visual evaluation occurs. The liquid is poured into the beaker, while the aggregates with bitumen are placed onto the transparent plate (Petri dish). The evaluation of the percentage of coverage of the aggregate is performed by minimum of two evaluators independently (without communication) using the guide provided in the standard (Figure 162). Afterwards the aggregate and bitumen, as well as the liquid from the beaker are placed back into the bottle and the rolling continues. The next observation occurs after 24 h from start. Test can be continued for 48 and 72 h, especially for mixtures in which adhesion promoting additives are used.

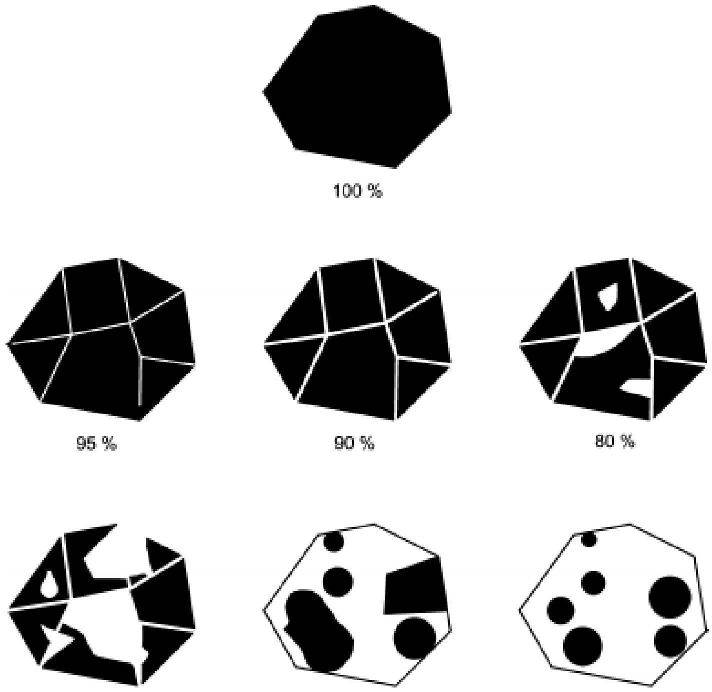


Figure 162. Reference for estimation of the degree of bitumen coverage on the aggregate.

The results comprise the average of at least two observatory evaluations, rounded to the nearest 5%. The result comprises either a singular value, e.g. coverage percent after 24 h, or a stripping curve in which the coverage percent is plotted against the rolling time.

14.3.2 Method variations and applicability to materials

The test is used in the Nordic countries (S. Anastasio 2015). For example, Norway was reported to limit percentage of coverage to minimum 25% after 48 h of rolling (Anastasio et al. 2015), yet the RBT is not a part of specification in all of the countries (Finnish Pavement Technology Advisory Council 2017). The affinity of bitumen and aggregate is especially important for low volume road mixtures, which contain soft bitumen and aggregate heated to lower temperatures than in the typical hot mix asphalt. In comparison with SMA mixtures, the SA mixtures have less adhesion promoting filler if any. For the evaluation of soft asphalt concrete pavements, a separate affinity test is specified for contractors (Finnish Pavement Technology Advisory Council 2017; Finnish Standards Association 2018) method A on specially prepared samples, or (Finnish Standards Association 2018) method C, though. Despite that, the RBT is used sometimes to evaluate the additives in a binary way (promoting increased adhesion, or not) during mixture design process.

Overall, the benefit of using the RBT as opposed to the Indirect Tensile Strength ratio (ITSR) for evaluation of the resistance to moisture damage, are related with an ability to test aggregate type. In ITSR for asphalt mixtures a ready sample, which includes fines and aggregates potentially from different quarries and of different mineralogy, is complicated for analysis, especially if the density of the aggregate influences the overall AC design. The RBT allows the differentiation between sources of aggregates. RBT is not an ideal test method to study mineral fillers as adhesion promotors such as hydrated lime, and yet it is often used in this context (Pylkkänen & Kuula-Väisänen 1990; Zofka, Maliszewska, Horodecka et al. 2013). The problem one could raise between comparing the binder modified by amines or hydrated lime and comparison by RBT is related to differences in having a mastic and not having a mastic. There may be some small stiffening increase due to addition of fine material, thus the viscosity of the mastic differs to start with. Nevertheless, it could be assumed that hydrated lime added at the level of 1–1.5% by asphalt mass will have rather low stiffening effect on the binder.

The interesting part about the RBT, is that some special configuration of parameters can be investigated in RBT easily, e.g. behavior in brine with de-icing chemicals (S. Anastasio 2015). This can also be combined with types of additives (Pylkkänen & Kuula-Väisänen 1990). Studies on the effect of the thermal history of the aggregate are also subject to less influence from fine and filler material in comparison of the results (Pellinen & Makowska 2018).

14.3.3 Repeatability and reproducibility

The Rolling Bottle test (Finnish Standards Association 2012) is one of the tests used to study the interaction between the bitumen and aggregate in the moisture environment, but less confidence is applied to its results. Recently, research effort was placed on determining the suitability of the test for use in specifications (Porot, Besamusca et al. 2015; Błażejowski et al. 2016). The methodology has a quite low repeatability values reported in the standard, and because of that is often considered unreliable. Various aspects were discussed in the past to affect the numerical result of the test, including the personal factor of the visual observer (operator's skills), the effect of the aggregate color onto the evaluation, the light conditions (Lantieri et al. 2017), the sample preparation procedure (Porot, Besamusca et al. 2015; Błażejowski et al. 2016), the geometry of bottles and temperature of solution. The necessity of multiple visual evaluators of the results to be present in the laboratory during the evaluation times, complicates the procedure and increases the cost.

The replacement of visual evaluation was suggested by means of photogrammetry and good results were obtained (Lantieri et al. 2017). However, during the interlaboratory comparison it was established that evaluation by skilled human evaluator from a picture was also an acceptable procedure with good repeatability (Porot, Besamusca et al. 2015). This inter-laboratory observation raised the importance of the sample preparation procedure on the result (Porot, Besamusca et al. 2015).

Nevertheless, the STAR report from the round robin evaluations suggested that the highest differentiation between samples occurs at the point of 24 h of rolling (Porot, Soenen et al. 2018). The conclusion is that the aggregate has more effect on the result than the bitumen, thus the test should differentiate between the aggregates (Porot, Soenen et al. 2018).

One of the issues observed in the laboratory of Aalto University during the tests is related to the lumps, especially in the evaluations of soft binders. If lumps exceed 10%, then the test is invalid, and results should be discarded. Soft binders have higher tendencies to still express stickiness between the grains. When binders of 160/220 or V1500 are compared, this aspect is more visible. The hard bitumen grade 50/70 or 20/30 after cooldown is relatively stiff and behaves as more elastic material, whereas the soft binders express viscous behavior at room temperatures, which leads to gluing of particles during the test.

The standard also allows the use of 5.6/8 mm fraction of aggregate, but the observations in the standard mention higher propensity to lumping, and the same is reported in the work by (Pylkkänen & Kuula-Väisänen 1990).

Last, but not least the studies related to test improvement suggested the effect of bitumen adsorption into the aggregate, that is storage of bitumen and aggregate at elevated temperatures as in silos, as one of the most affecting aspects during the sample preparation (Błażejowski et al. 2016).

14.3.4 International experience

As mentioned previously, the project conducted by the Road and Bridge Institute in Poland (Zofka, Maliszewska, Horodecka et al. 2013; Bankowski et al. 2011; Maliszewska et al. 2012) investigated the affinity between the aggregates and binders of different grade with and without the addition of adhesion promoters using the RBT test. The experimental plan was based on the similar matrix as that presented in Table 20.

The results presented in the final report (Maliszewska, et al. 2012; Zofka, Maliszewska, Horodecka et al. 2013) were plotted in Figures Figure 163–Figure 165. Results indicate that every aggregate and binder combination tested, has its own optimal adhesion promotor. Some adhesion promotors were decreasing the affinity, as witnessed with RBT. Therefore, suggesting the use of one type of adhesion promotor across all types of mixtures, binders and binder types could prove challenging. On the other hand, the aggregate types presented in Figure 164 and Figure 165 are not the types of aggregate typically used in Finland for road construction.

What is worth to underline, is that in Finland there is no regulations for the binder course and bound layer courses in respect of resistance to moisture. In those layers, the resistance to studded tire wear is a parameter of negligible significance. Therefore, the use of other, lower quality materials is likely. Proposed is the review of all the aggregate-binder couples used currently for bituminous mixtures production in Finland in order to determine the actual necessity for testing.

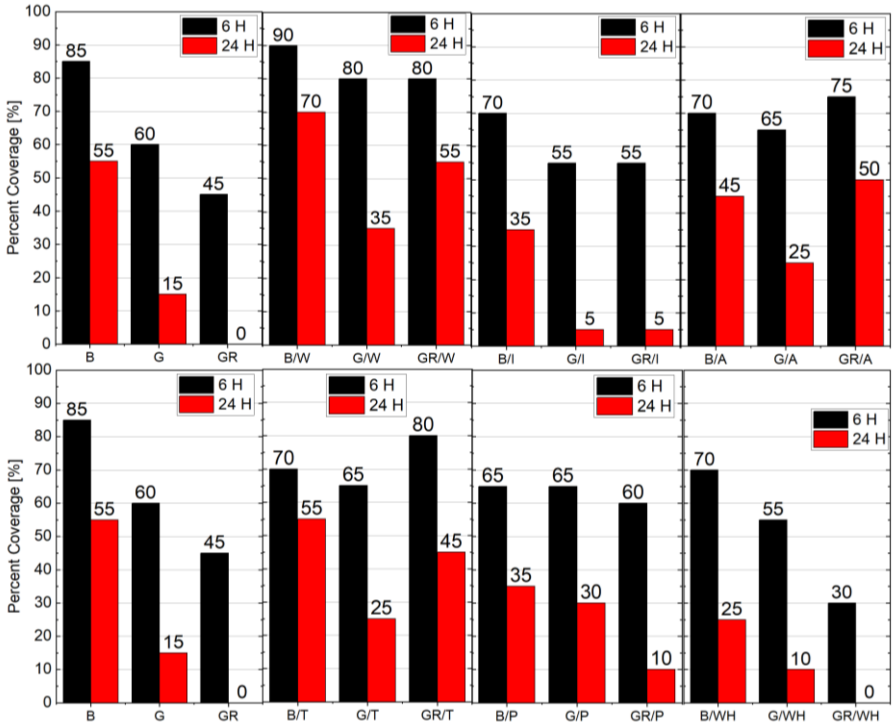


Figure 163. The RBT results for Basalt (B), Granite (G) and Granodiorite (GR) with binder 45/80–55 without and with adhesion promoting modifiers (Zofka, Maliszewska, Horodecka et al. 2013).

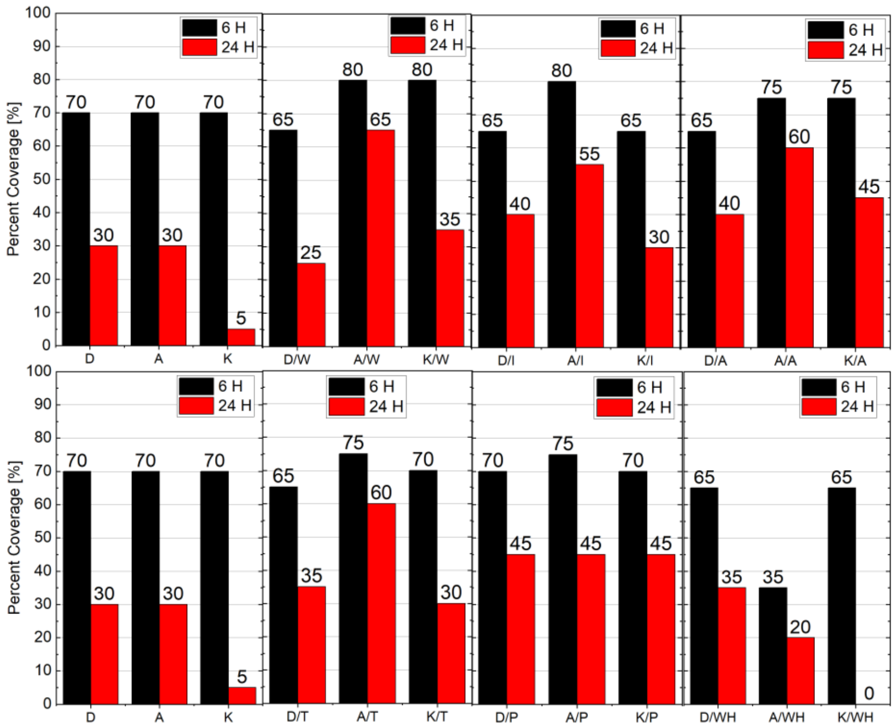


Figure 164. The RBT results for Dolomite (D), Amphibole (A) and Quartzite (K) with binder 50/70 without and with adhesion promoting modifiers (Zofka et al. 2013).

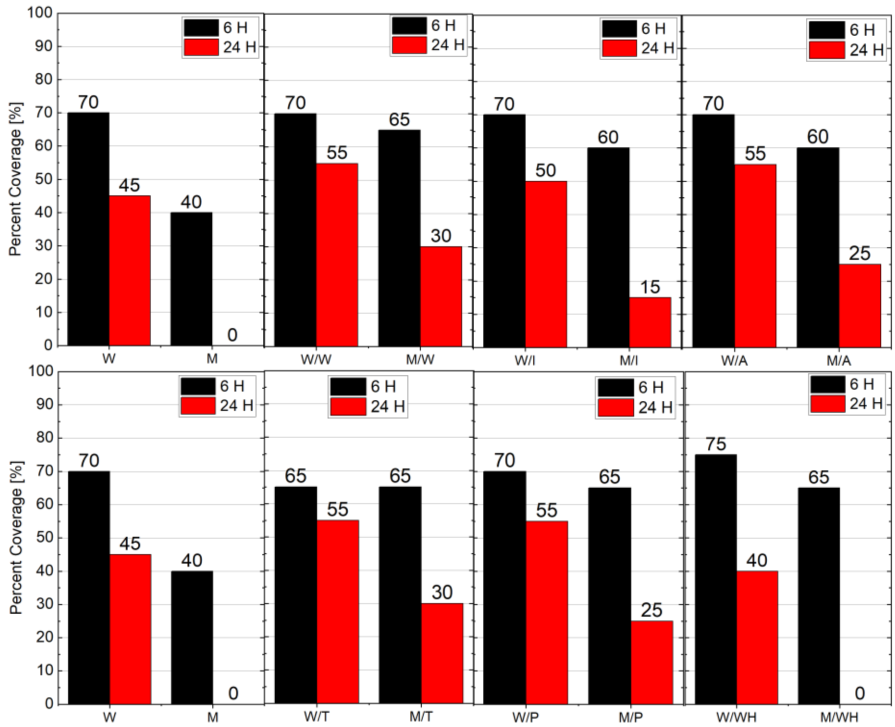


Figure 165. The RBT results for Limestone (W) and Paleobasalt (M) with binder 30/50 without and with adhesion promoting modifiers (Zofka et al. 2013).

In the context of the BITU2020 project, one of the most interesting research project investigated the effect of the producer of the binder on the affinity to the aggregate, as well as compatibility with the adhesion promotor (Paliukaitė et al. 2016). In the study, the bitumen 50/70 and PMB 45/80–55 from two sources were investigated as binders, while the Iterlene IN/400-L and Antrocelbond were used as adhesion promotors, but coded due to confidentiality (Paliukaitė et al. 2016).

The polymer modified binder was apparently performing better in terms of adhesion to aggregates tested (granite and dolomite) (Figure 166). However, it became apparent that **the choice of appropriate amount of adhesion promotor depends on the binder source.**

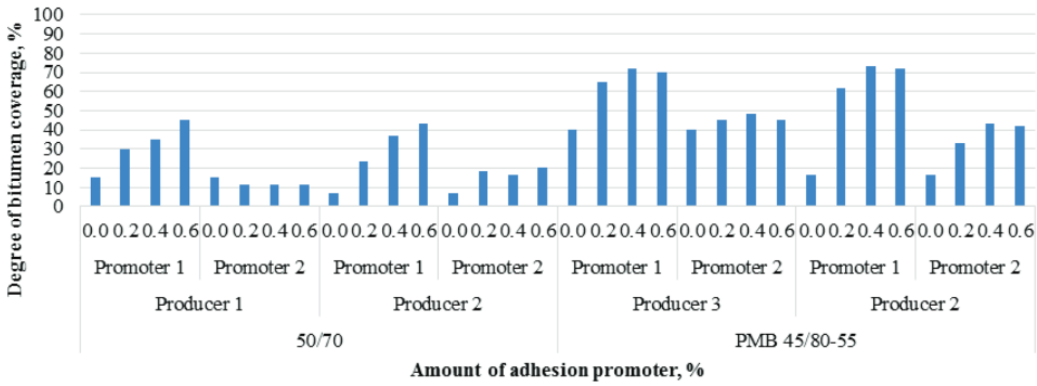


Figure 166. The coverage percentage of granite aggregate by bitumen after Rolling Bottle test (24 h) (after Paliukaitė et al. 2016).

Interestingly, in the studies on the action of de-icing chemicals on the result of the RBT, the coverage degree was higher in bottles filled with brines, rather than deionized water (S. Anastasio 2015). The lower stripping in the presence of de-icing chemicals was assigned to the difference surface tension of brines in comparison to the surface tension of the deionized water (see section 15.2).

14.3.5 National experience

The rolling bottle test was used during the ASTO project (Pylkkänen & Kuula-Väisänen 1990) to evaluate the differences between bitumen sources and their effect on stripping. As is described in section 15.3.4.1 , the bitumens of the same penetration grade were found to differ in acidic and basic numbers. Figure 167 presents results in which the differences between binders are underlined. The behavior of binder from source A and LAG express the opposite behavior in terms of affinity with chosen aggregate types.

Additionally, the evaluation of the effect of the adhesion promoting modifiers was performed. The tests were unfortunately not summarized numerically, but in the form of pictures, which are reprinted here (Appendix 1).

The adhesion promoting additives, tested using similar techniques also indicated varying levels of acidity (Pylkkänen & Kuula-Väisänen 1990). It was proposed that acidic aggregate and acidic bitumen (LAG) would not be compatible. The addition of basicity increasing promotor was postulated to increase the affinity between the components. Granite and granodiorite were characterized by the lowest affinity with reference bitumen AH (more basic) and LAG (more acidic) after 72 h of RBT. Addition of acidic adhesion promotors resulted in decreased affinity or no improvement in case of AH type binder (Appendix 3). In terms of LAG binder the improvement was seen with amine based promotors and not the more acidic promotors (Appendix 3).

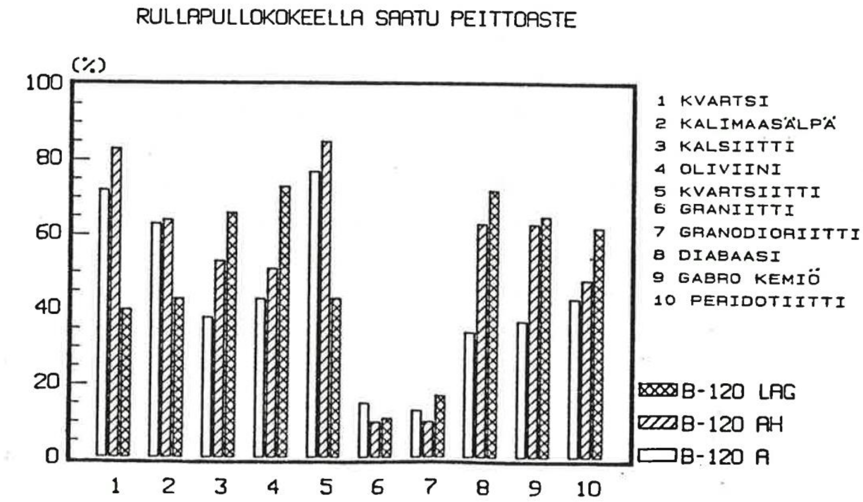


Figure 167. The comparison of the results of the RBT (percent covered on y axis) performed for 72 h on 10 aggregate types using 3 same grade bitumen from varying sources (LAG – Venezuelan, AH – Arabian Heavy, A – Russian, 1 – Quartz, 2 –Quartz feldspar, 3 –Calcite, 4 – Olivine, 5 – Quartzite 6 – Granite, 7 – Granodiorite, 8 – Diabase, 9 – Gabro from Kemiö, 10 – peridotite) (Pylkkänen & Kuula-Väisänen).

14.3.6 Problems related to method and research needs statement

Some of the questions which would be worth evaluating for the production purposes in Finland are formulated as below questions:

- Is the time between the production and cool down in fact affecting the adhesion between bitumen and aggregate?
- Are the production parameters such as heating, control of moisture in the inflowing aggregate, and the storage of the mixture prior to laydown, influencing the adhesion?
- The aggregates in Finland are not always stored under tents or facilities in which the moisture content is controlled within stock-piles. Provided that a mixture remains longer above the 140 °C, the steam trapped in the aggregate has an ability to bubble through bitumen and evaporate during storage. If the temperature drops down fast, the moisture may be trapped inside the asphalt concrete. The effect of sample storage of binder-aggregate affinity should be evaluated using also other techniques.
- The effect of the acidity of the bitumen, as characterized by the acidic number, is a likely cause or indicator of the increased or decreased affinity. Monitoring the chemical properties of the binder using titration techniques is fairly easy for a trained laboratory technician to perform and should be considered as a screening/quality assurance test for bituminous products. Currently there is on performance predicting parameter of bituminous binders which indicate anything about the binder influence on water sensitivity in Finnish Asphalt Specifications (Finnish Pavement Technology Advisory Council 2017).
- Since the use of de-icing chemicals, such as regular sodium chloride brine or calcium chloride brine seems to positively influence the affinity of bitumen with aggregate due to the alteration of water's surface ten-

sion, a suggestion is made to investigate possible preventive maintenance procedures, for example spreading brine prior to expected large precipitation in spring and during warm winters.

14.4 Static Method (SFS-EN 12697-11 method B)

The static method was used in Switzerland as part of the requirements for asphalt pavements with soft bitumen types. This method development for local market was adding more focus on long term behavior of asphalt mixtures in warm water, as it is expected that 90% of binders in Switzerland have a softening point below 40 °C and temperatures in that range are observed on the pavements during the summer-time (Hugener, Beltzung & Angst 2012).

14.4.1 Principle

A portion of the aggregate (150 particles) after being coated with bitumen is submerged into the deionized or distilled water at room temperature. The sample is left in this state for up to 48 h. After 48 h, the operator removes the water and visually evaluates the number of particles not fully covered by binder. The number of particles is divided by total number of particles, and the result expressed as a percent of coverage. If 1 in 3 particles is fully uncovered, the test needs 3 more replication.

14.4.2 Method variations and applicability to materials

This method is similar to the so-called “bucket test” used for the soft mixtures. In this test the soft asphalt concrete or oil gravel, were submerged into the bucket filled with water. If the materials were prone to moisture damage or incompatible, the separation between binder and aggregate was visible. <u>This test is still required for PAB-V mixtures during the production.</u>

14.4.3 Repeatability and reproducibility

No precision data is available.

14.4.4 International experience

The Switzerland initiated a project focused on evaluation of this standardized method B and comparing it to the boiling water standard used in Swiss specifications (Hugener, Beltzung & Angst 2012). The differences between all the methods in standard 12697-11 and the Swiss instruction are described in Figure 169.

The results suggested that the low temperature of water conditioning applied in method B was not sufficient to induce the stripping within the considered time. The mixtures prepared using 50/70 and 70/100 binders expressed stripping on the level of 5–10% in those conditions. Increasing the temperature to 40–50 °C resulted in decreased covered area to the level between 60–75% depending on the time of exposure. The suggestion by the researchers was to increase the water temperature in method B in order to simulate the summer conditions of intense precipitation after hot summer day, and a temperature of 40 °C was proposed. The suggestion was also to revisit the current requirements applied in

Switzerland to the materials, by retesting the well and bad performing combinations (Hugener, Beltzung & Angst 2012).

The procedure after the change of the conditioning temperature was validated using various binders and interestingly the differences in affinity became more apparent between binders.

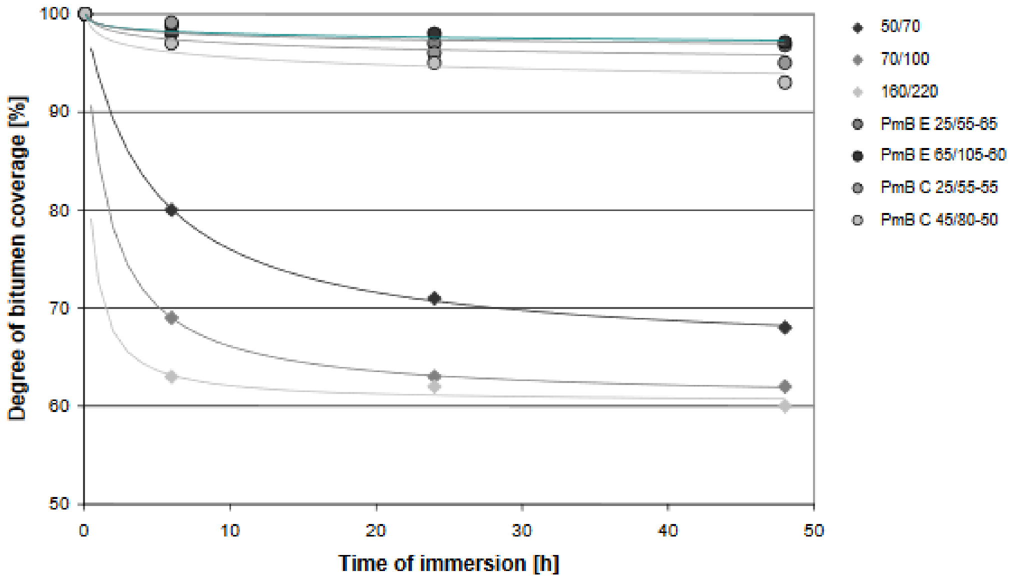


Figure 168. The results of the evaluation of reference mixture composed of 7 binders studied using the static method (SFS-EN 12697-11 method B) after the adjustment of the water temperature to 40 °C during conditioning.

	EN 12697-11:2005 [1]			**SN 670460 [2]**
	Part A	**Part B**	**Part C**	
Aggregate coating				
Number of samples	3	1 or 4	2	2
Binder amount	16g (3.1%)	4% (±x·0.5%)	31.5 g (2.1%)	8 g (5.3%)
Aggregate size	8/11.2 (5.6/8, 6.3/10) mm	6/10 mm	7/14 mm	8/11.2 mm
Aggregate amount	510 g for 3 sets	150 particles	1500 g	150 g
Mixing temperature	variable, according to EN 12697-35 + 25°C [8]	130°C	variable, according to EN 12697-35 [8]	variable, according to EN 12697-30 [9]
Cooling time	12 h	60 min	Immediate quenching in cold water	5 min
Water conditioning				
Conditioning type	rolling bottle	static	static	static
Water temperature	20 ±5°C	19 ±1°C	boiling water (90 - 100°C)	60 ±0.5°C
Conditioning time	24 ±1h	48 ±1h	10 min	60 ±1 min
Assessment of the coating degree				
Condition of the aggregates	wet	dry	in hydrochloric or hydrofluoric acid	wet
Validation of the result	visual estimation of the coating proportion after 6 and 24 h	number of not completely coated aggregates (visual)	titration of the reaction solution	visual estimation of the coating proportion with graphical aids
Result	coating coefficient in 5% steps	passing yes/no	coating coefficient in 1% steps	coating coefficient in 1% steps

Figure 169. Differences between methods of the standard 12697-11 and the Swiss instruction (Hugener, Beltzung & Angst 2012).

14.4.5 National experience

Even though the methodology is suggested for use during production by (Rakennustietosäätiö RTS 2010) the last published data on the use of the method is related to the studies of SA type pavements from 1997 (Apilo & Eskola 1998) as "sankokoe" (bucket test). No effort to evaluate how does the standard 12697-11 method B compares to the previously used bucket test was found.

14.4.6 Problems related to method and research needs statement

If there is any deviation in Finland in comparison to the standard reported above, it should be formulated into a proper instruction. Evaluation of the correlation between the performance and results on a large scale from materials produced would be beneficial for future formulations of specifications. Therefore, it would be suggested to start collecting the documentation related to the bucket tests performed during production.

14.5 Boiling Water Striping Method

As mentioned during the introduction of the Static method, the boiling method is more targeted at evaluation of the behavior of soft asphalts during precipitation occurring during the warm season.

14.5.1 Principle

The aggregate samples after covering with bitumen is boiled in hot water. The liquid is decanted. The evaluation of the stripping level occurs by acid etching of the aggregate exposed, and then titrating the obtained solution.

14.5.2 Repeatability and reproducibility

A repeatability coefficient of variation of 15 % of the determined value has been found, with an absolute precision threshold on the determination of the stripping percentage of 2 %.

Reproducibility has not been established for this test.

14.5.3 Problems related to method and research needs statement

In the opinion of the authors of hereby report, the possibility of transfer of this technique into the industry is unlikely. The method uses dangerous chemicals, such as hydrofluoric acid, which is currently discouraged in laboratories. It is one of the most corrosive acids on the market and it dissolves glass, hence has a possibility to damage laboratory premises, if insufficient precaution is taken. Additionally, the laboratory worker is required to be able to distinguish the types of the aggregates prior to test based on their mineralogy in order to choose the most appropriate dissolving chemical.

The professional evaluation of the state of laboratories in Finland suggests that it is highly unlikely to be an accepted test by the community. The method would require specialist in the laboratory just for this purpose, on top of safety protocol and dangerous chemicals, which relates to the investment in the upkeep of station and disposal of chemical waste. For that reason, it will not be discussed further. If the community is interested, more detailed evaluation can be pursued.

14.6 The compressive strength after moisture conditioning (EN 12697-12 Method B)

Unfortunately, insufficient amount of information was found on the use of the method. However, the test is applying the load to the sample in a similar direction as in field (by traffic). The compressive strength and resistance to deformation is evaluated between the samples. Similar principle is used in the Hamburg Wheel Test Device as well as the Water Compression test evaluated during the ASFADUR project (Laukkanen, Halonen & Pyy 2012).

14.6.1 Principle

A cylindrical sample, obtained by any method of compaction is first saturated with moisture under vacuum and then submerged into water for 7 days at 18 °C. A separate test of samples is kept in dry conditions. After the conditioning the compression test is performed on the sample. The maximum force recorded is used in the comparison between dry and wet sets. The resistance to moisture is expressed as a compressive strength ratio between wet and dry subset of samples.

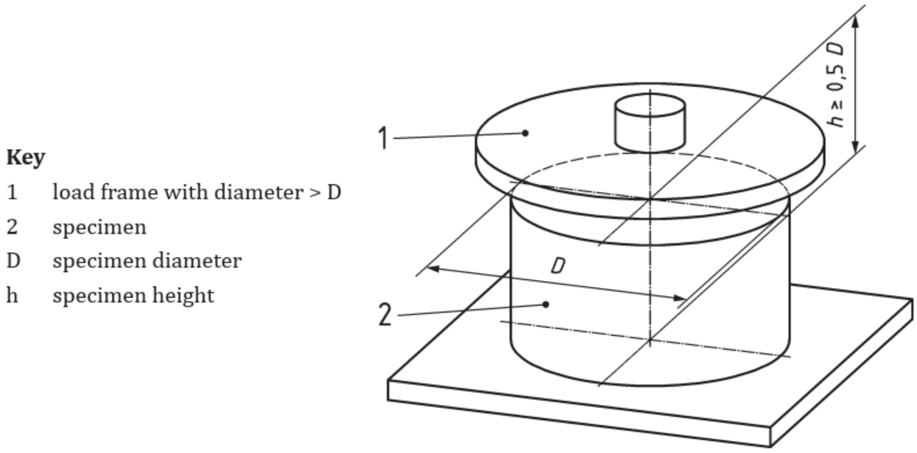


Figure 170. The specimen type and loading regimen in the Compressive strength after moisture conditioning (SFS-EN 12697-11).

14.7 Hamburg Wheel tracking (HWT) device

Due to the fact, that in field the loads are applied perpendicular to the surface, the idea of testing the resistance to damage by following the effect of wheel rolling on top of the surface is of concern. The traffic is inducing both creep and compaction of material in dry conditions, while in the wet conditions it was proposed that the water pumping inflicted by the action of wheels may be a contributing factor to the moisture induced damage.

14.7.1 Principle and Method variations

The susceptibility of a bituminous material to deform is assessed by measuring the rut depth formed by repeated passes of a loaded wheel at a fixed temperature. The wheel tracking can be performed in a large device or small device. The Hamburg wheel-tracking device is considered the small device. The tests performed in the small device can be conducted in dry and wet conditions.

The wheel-tracking apparatus (Figure 171) consists of loaded wheel and a sample securely attached on top of the tabletop. In this test, the table moves underneath the wheel, or the wheel moves on top of the table. The load applied to the wheel and number of passes per sample are specified in the standard (SFS EN 12697-22).

Figure 171. The Hamburg Wheel-tracking device (Olsson, Krona & Nordgren 2010).

After the test is performed, the rut depth is recorded initially every 500 cycles, and then at least after 1000, 5000 and 10000, or until 20 mm rut depth is observed. The rut develops initially following the logarithmic trend, and after approximately 5000 wheel passes a linear change is observed. A graph of recorded ruth depth after wheel tracking cycles is prepared. The rest result is the slope (rate of rutting) of the line between 5000–10000-wheel passes (WTS – wheel tracking slope, mm), and the mean average rut depth after *N* cycles in relation to sample height (PRD – proportional rut depth, %).

The method A is conducted in air, and Method B is conducted in water. The results of method A are reported as WTS_{air} and PRD_{air} while method B as WTS_{w} and PRD_{w}. The minimum two tests is required to provide results.

14.7.2 Repeatability and reproducibility

The precision data related to the test is limited. The precision will depend on the conditioning parameters (temperature, air, water, etc.), but also on the sample preparation technique. Below are the tables provided in the standard EN 12697-22.

Table 24. The precision values for PRD from laboratory prepared specimens.

Number of cycles	Test result level %	Repeatability, r %	Reproducibility, R%
100	3.5	0.76	0.97
1 000	4.8	1.05	1.32
10 000	6.4	1.08	1.20
30 000	7.0	1.11	1.16

Table 25. The precision values for wheel-tracking from cores (WTS).

Samples	Test result level microns per sample	Repeatability, r microns per sample	Reproducibility, R microns per sample
Laboratory made	2.1	0.5	1.0
	1.7	0.6	1.1
From site	6.4	2.5	4.7
	10.7	3.2	4.5

According to the literature, the test is sensitive to the quality of aggregate, the stiffness of binder, the duration of short-term aging process, the source of bitumen and the refining process used for its production, the additives presence as well as to the compaction temperature used for manufacturing of samples (Chaturabong & Bahia 2017).

14.7.3 International experience

14.7.3.1 Wisconsin

The tests evaluated the effect of moisture on the results from HWT. The experimental design included two mixtures (for MT– Medium and HT– Heavy Traffic), two binders (V – very heavy traffic / 64 °C, S – standard traffic grade / 56 °C) and two aggregate types (Granite and Limestone), to be tested by HWT in both dry and moisture conditioned state.

The binders were tested with DSR for the base parameters such as G*, δ and G*/sin(δ). This was supplemented by MSCR test performed at 50 °C on both unaged and RTFOT aged samples. The choice of the MSCR temperature usually comes from the PG grade. In this case, the HWT was performed at 50 °C and for that reason, the same temperature was used in MSCR test. The chosen binder parameters are presented in Table 26.

Table 26. The summary of the properties of binders studied in Wisconsin (Chaturabong & Bahia 2017).

Binder	PG	Complex modulus at PG [G* (kPa)]	Phase angle at PG [δ (°)]	G*/sin(δ)	Recovery (3.2 kPa) [%]	AVERAGE Jnr (3.2 kPa)	Pass /fail
S-28	58	1275	72.9	1275	0.89	2.29	Fail
V-28	64	1658	86.9	1735	34.64	0.51	Fail
S-28_RTFOT	58	3719	82.9	3748	0.81	2.38	Fail
V-28_RTFOT	70	2315	68.4	2492	30.80	0.96	pass

One of the binders was more susceptible to rutting, so was one of the mixtures. The air void content was kept at approximately 4%. The mastic of those mixtures was additionally tested using the PATTI® (see section 17.1.1). The failure mode due to the conditioning of samples was found cohesive, based on strength and PATTI® tests.

In the HWT the initial wheel runs cause so-called consolidation. After consolidation, the creeping occurs and the slope of the line is defined as an inverse of creep. However, after certain amount of wheel runs the curve changes shape and a stripping point is observed. Afterwards, the wheel run depth develops following a different slope, known as the inverse of stripping (Figure 172).

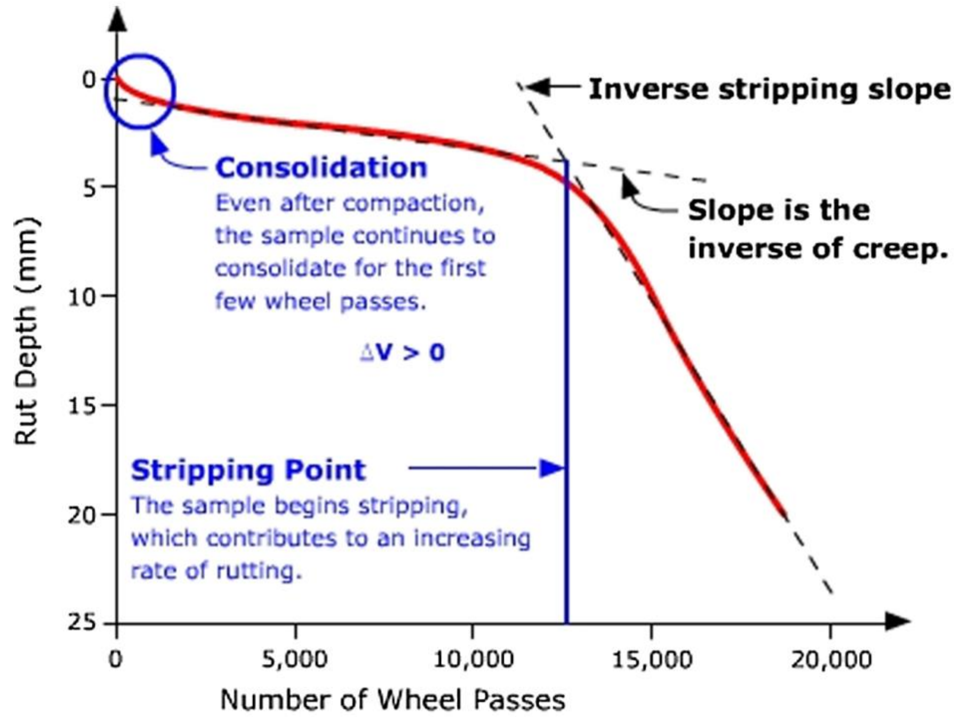


Figure 172. Explanation of the terms related to analysis of the curve obtained from the HWT (Chaturabong & Bahia 2017).

The wet and dry conditioned samples behave differently in HWT device. The moisture conditioned samples reach the stripping point much faster than the dry conditioned samples (Figure 173).

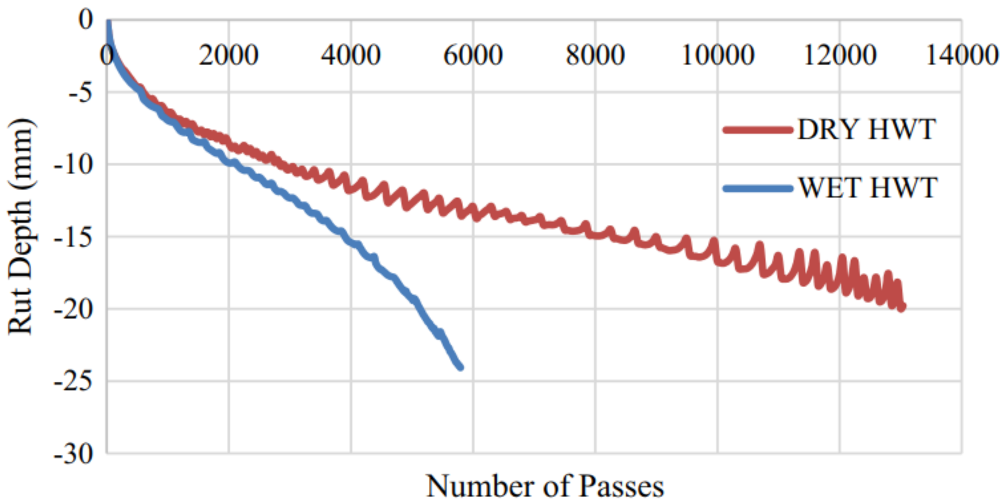


Figure 173. An example curve obtained during the HWT test for Granite and S-28 bitumen using the MT mixture (Chaturabong & Bahia 2017).

The researchers developed a novel parameter for the moisture susceptibility of the mixtures based on the derivative analysis of the curves. Analyzing the first derivative of the curve the range of creep and strip were defined, and the slopes of those lines were related to each other. The ratio between the first derivative derived slopes is the moisture susceptibility.

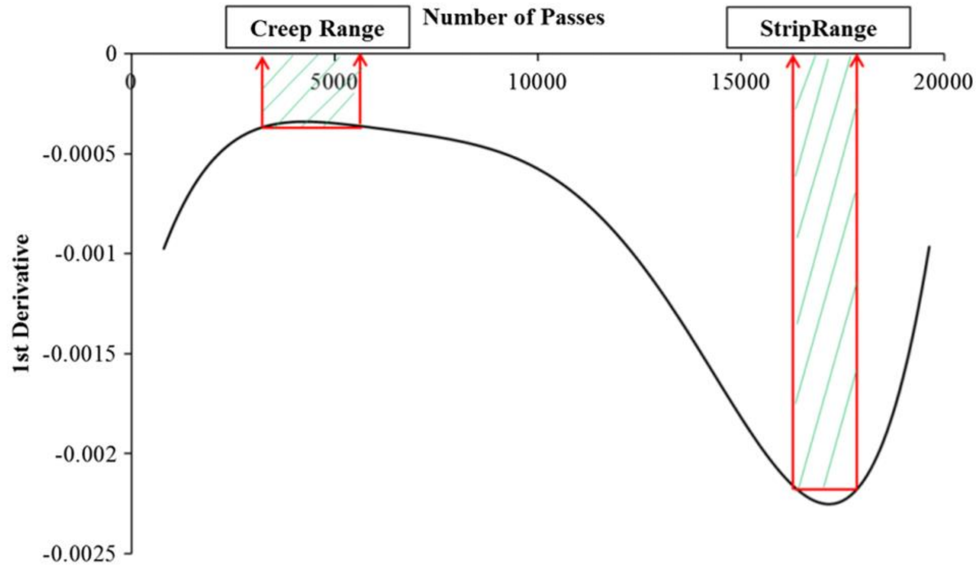


Figure 174. The first derivative of the 6-order polynomial fit of the HWT results with the definition of stripping and creep slopes (Chaturabong & Bahia 2017).

The idea of this calculative attitude is based in the assumption that creep slope in the early stage of wheel tracking is similar, or within repeatability between dry and wet samples. Therefore, running the HWT only in wet state, could allow determination of both properties – the creep and stripping susceptibility.

Despite the fact, that the researchers mention that the mixtures are chosen based on the knowledge coming from field in respect of their moisture susceptibility, unfortunately no clear link to the performance in the field is presented in this study. Nevertheless, based on the previous specifications, the ratio between the slope of stripping and slope of creep should be lower than 2 for the mixture to be moisture resistant. All of the tested mixtures had this value higher than 2, thus were all deemed as moisture sensitive. (Chaturabong & Bahia 2017)

14.7.3.2 Texas

The researchers work with the HWT performed on the two cylindrical samples of diameter 150 mm and a thickness of 61 mm, connected to form a path for the wheel to pass on. The slabs are submerged in water at 50 °C. Approximately 52 passes of wheel per minute are applied (AASHTO T324). Each specimen is loaded for at least 20 000 cycles or until 12.5 mm rut depth develops.

The definition of terms related to the output curve from the HWT are slightly different between different publications. The nomenclature applied by (Yin et al. 2014) is explained visually based on the diagrams in Figure 172. The initial rut depth defined as "consolidation by (Chaturabong & Bahia 2017) phase is referred to as the "postcompaction". The creep range and creep phase are similar in the works by both groups. The stripping point and stripping number are defined in a similar way as the point of deflection of the curve from the projected rut development due to the viscoplastic deformation Figure 176.

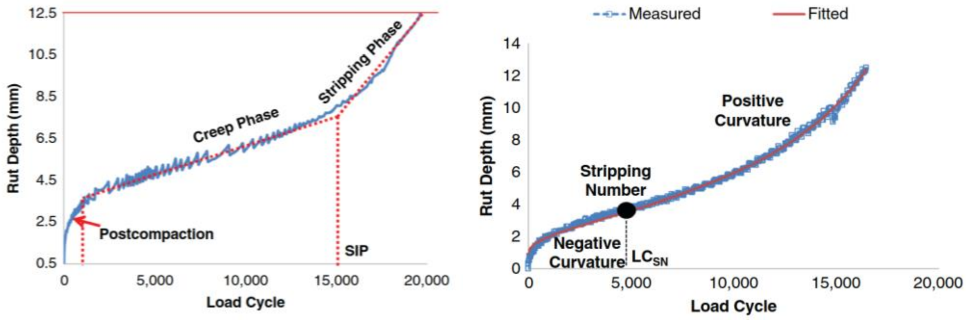


Figure 175. The typical HWT output of the rut depth versus load cycle with defined nomenclature in the study by (Yin et al. 2014).

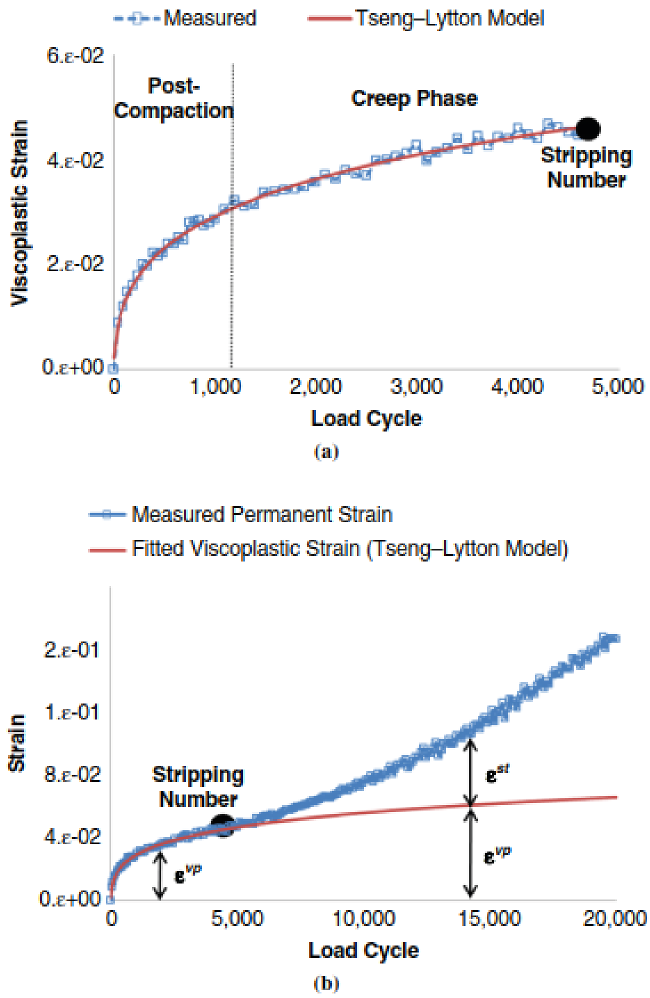


Figure 176. Typical viscoplastic strain behavior versus load cycle in HWT (a) fitted to the post compaction phase of the curve and (b) projected viscoplastic strain in stripping phase (after Yin et al. 2014).

Conducting the analysis of the curves in this simple manner allows one to compare between mixtures. The researchers compared Hot Mix Asphalt (HMA) mixtures and Warm Mix Asphalt (WMA) mixtures. The typical mix design used in Texas was evaluated in form of reference, and in two mixtures an addition of antistripping additives was used – hydrated lime (lime) and liquid anti-stripping additive (LAS). For comparison, the mixtures containing 15% of recycled asphalt pavements (RAP) and recycled Asphalt Shingles (RAS) were prepared.

Interestingly, using this approach or the approach in which the stripping point described in previous section were used, the results of evaluation seemed similar. The resistance to moisture damage with use of adhesion promoters increased as witnessed by the stripping point or stripping number (Figure 177). The advantage of the use of the stripping number may be in the relatively lower number of cycles prior to its occurrence. The stripping point and strip range defined in the studies by (Chaturabong & Bahia 2017) require longer test duration (see Swedish experience in section 14.7.3.3).

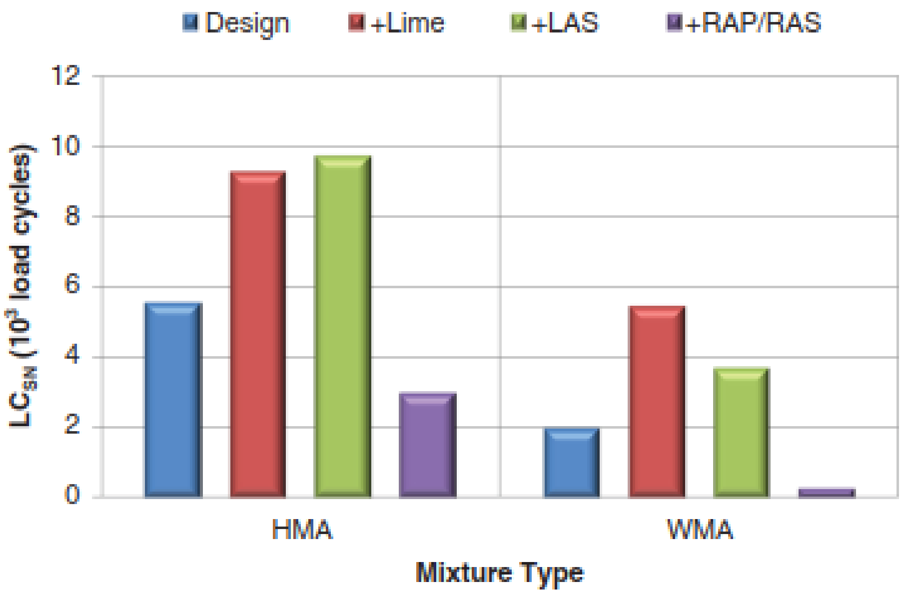


Figure 177. The number of cycles necessary to reach stripping number (LC_{SN}) of HMA and WMA mixtures with additive and RAP modification against reference mixtures (after Yin et al. 2014).

Interestingly, the use of the RAP and RAS material in this study (Yin, et al. 2014) decreases the resistance to moisture susceptibility of tested mixtures. In the context of increasing content of recycled materials in Finnish mixtures over the last decades in Finland (and worldwide), and the perceived decreasing resistance to moisture damage the effect of RAP on this, performance criteria should be considered for studying. It is underlined that if the mixture in Finland is allowed to be characterized by the SFS-EN 12697 method A performed on the PA11 gradation type with 70/100 binder (PANK 4306), considered as the aggregate moisture susceptibility test locally, the effect of RC is not measured. The mixtures containing RC should be evaluated from final compositions.

14.7.3.3 Sweden

As mentioned in section 14.1.2.5 Swedish researchers investigated the effect of bitumen source on the moisture susceptibility using a myriad of research methods, among them the HWT at 50 °C. Interestingly, looking at the results presented in the report (Olsson, Krona & Nordgren 2010) and comparing the dry and wet rutting curves, we can observe the differences between the mixtures. It seems, that the mixture 1 expresses the so-called stripping point before the level of 10000-wheel passes is applied to the moisture conditioned sample (Figure 178 and Figure 179). This could indicate that the test can differentiate between the bitumen types and their moisture susceptibility.

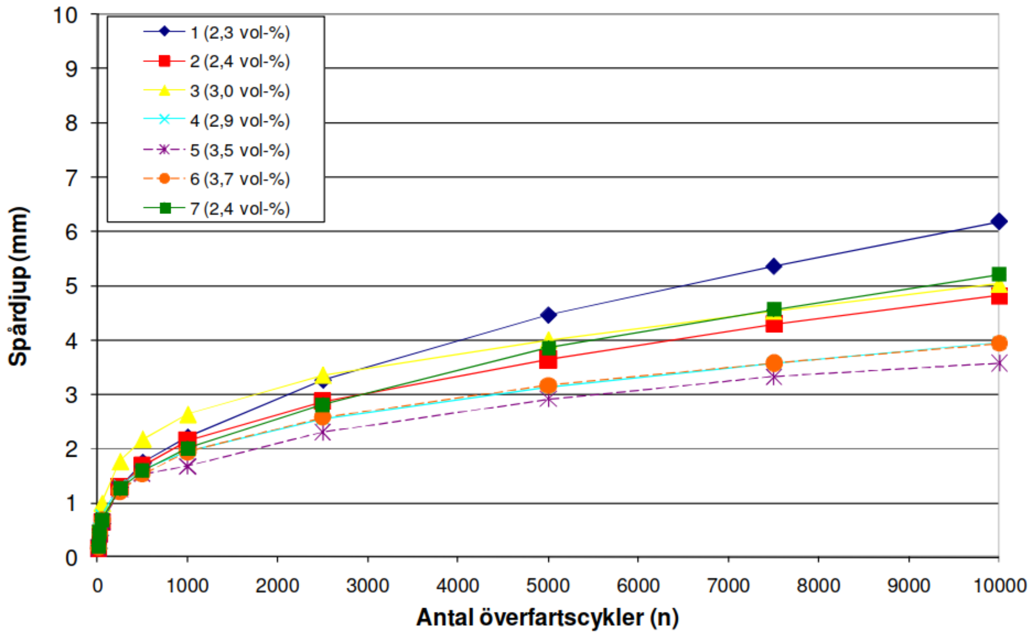


Figure 178. The result of dry conditioned samples measured by HWT for different binders described in Table 22 for asphalt mixtures compacted to air voids on level defined in the legend of the figure (Olsson, Krona & Nordgren 2010).

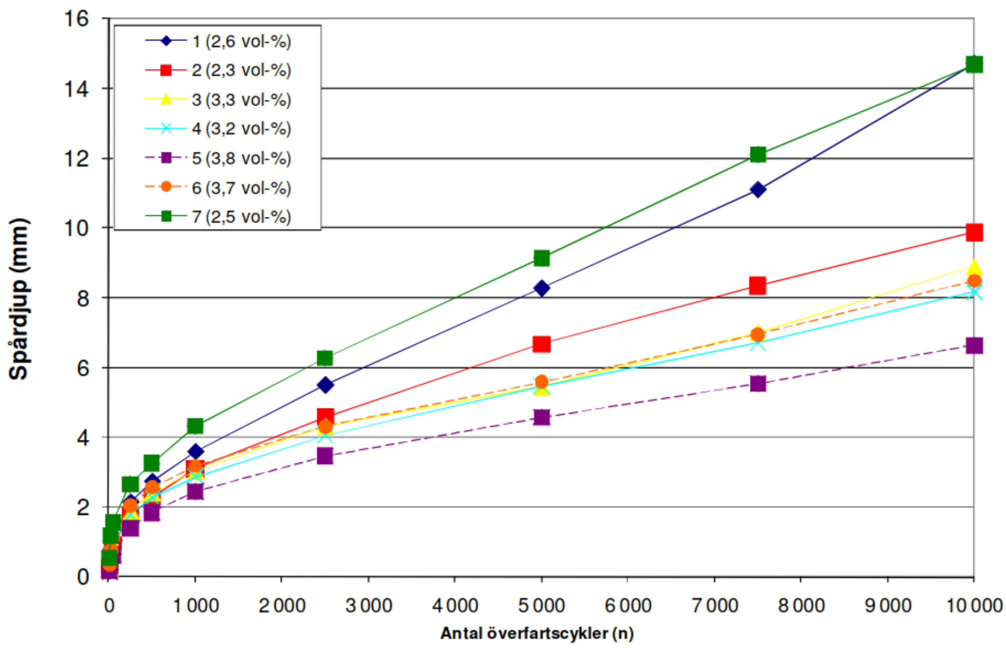


Figure 179. The results of the moisture conditioned samples measured by HWT for different binders described in Table 22 for asphalt mixtures compacted to air voids on level defined in the legend of the figure (Olsson, Krona & Nordgren 2010).

Interestingly, the rutting speed or slope of creep differs between the binders tested. This could indicate the different susceptibility of the binder to rutting. In the Swedish research unfortunately the MSCR test was not performed and the only knowledge available is related to the penetration, softening point and viscosity of the tested binders. Researchers argued as well, that the effect of different rutting speed could be coming from the differences in the air voids level between the prepared samples. However, when the correlation between the speed of rutting and air void content was attempted, the results indicated no correlation. Indeed, the expected high rutting speed could be expected for samples containing 3.5% or 3.7%, but those have in fact the lowest rutting of the tested samples (Olsson and Witkiewicz 2017).

Considering that the only differences between mixtures were in the origin of the binder, HWT is a promising tool to establish both influence of the binder source to the regular creep as well as to the resistance to moisture. Accompanying the HWT with MSCR test on binder could comprise interesting combination of tests for differentiation between the products based on their actual performance, as both tests are relatively simple and fast.

14.7.3.4 Polish experience with HWT and adhesion promotors

The Polish study used the small wheel-tracking device as well to investigate the mixture properties. The bitumen producer was Lotos Asfalt for all tested mixtures, but the different grades were used.

The experimental approach was unfortunately different in terms of the HWT. Based on the results from ITSR, the mixtures with lowest and highest resistance to moisture damage were chosen for HWT evaluation, plus few interesting combinations. Because of that approach, the reference samples were not tested. Additionally, the HWT test was performed only in air, thus the information related to moisture susceptibility for those mixtures is missing. Neither of the presented curves obtained in dry conditions expressed the stripping point up to 20000 cycles.

The results of the test are summarized in Table 27, indicating that the rutting resistance of the mixtures is affected to some extent by the adhesion promoter. Looking at those results, it could be suggested that in differentiation between two mixtures of the similar moisture resistance assured by the ITSR test, the resistance to rutting should be evaluated as a secondary differentiation parameter.

Table 27. The summary of the results of HWT test compared against the ITSR results from the various mixtures modified with adhesion promoters (Zofka, Maliszewska, Horodecka et al. 2013). The samples marked in grey comprise couples of similar ITSR result for visualization.

Bitumen and mixture type	Aggregate type	Modifier type	ITSR	PRD_{air} [%]	WTS(d10000-d5000) [mm/1000 cycles]
SMA 11 45/80–55	Basalt	WETFIX	90,8	12,70	0,202
	Basalt	PE-31	112,6	6,00	0,064
	Gabbro	WETFIX	89,0	7,60	0,062
	Granite	WETFIX	81,7	10,00	0,116
	Granite	INTERLENE	80,0	8,70	0,084
AC16 bind 50/70	Amphibole	WETFIX	95,6	7,20	0,156
	Amphibole	AD-HERE	100,0	7,10	0,134
	Dolomite	WETFIX	43,8	7,90	0,136
	Dolomite	INTERLENE	41,9	7,40	0,154
	Quartzite	WETFIX	79,8	10,30	0,234
	Quartzite	-	62,7	10,80	0,254
	Quartzite	AD-HERE	95,6	10,40	0,270
	Quartzite	WH	60,1	14,00	0,380
AC22 base 35/50	Limestone	W	78,0	7,20	0,172
	Limestone	P	63,3	10,60	0,312
	Paleobasalt	W	97,4	4,60	0,076
	Paleobasalt	WH	70,4	7,10	0,138

14.7.4 Problems related to method and research needs statement

The dry test evaluates the resistance of the mixture to rutting due to the repetitive loading inflicted by the tyre. This rutting type is related primarily to permanent deformation. However, the test performed in the moisture conditioned regime, is affected by the presence of moisture. The pores are filled with water and the load of the tyre inflicts compression forces onto the water in the filled air voids. As a result the stripping may occur.

In the previous sections it was evaluated that the test is reported to be sensitive to bitumen sources and refining processes used for the manufacture of binder. Additionally, the effect of additives can be evaluated not only as affecting the moisture damage resistance, but at the same time the creep properties of the modified asphalt concrete. For that reason, using the Hamburg wheel tracking device could be interesting and replace two tests currently used in Finnish Asphalt Specifications (ITSR and Creep). It is suggested to include this test in the future research efforts, as no report which would investigate typical Finnish mixtures was found in the literature.

15 The test methods of the past

15.1 The ASTO project at the origin of FAS2017

Due to the increasing damages observed on the pavements in Finland during 1980s, the research project titled Asfalttipäällysteiden Tutkimusohjelma (ASTO) (transl. The research program for the asphalt pavements) was inaugurated in Finland and took place between 1987–1992. Within this program development of the asphalt production process, laydown, compaction, design of the asphalt mixtures, testing of the raw materials as well as the quality control of the asphalt pavement occurred. To support the findings the large number of test roads was included in the program to investigate the link between laboratory tests and performance in field. Many local aggregates, multiple bitumens, fillers as well as rejuvenators, fibers and additives were investigated.

Based on the findings presented in the ASTO a series of rules and requirements was set forward for both the raw materials and produced pavements, which then became a Finnish Asphalt Specifications in 1995. From then forward, Finnish Asphalt Specifications have been updated by the organization known as Päällystealan Neuvottelukunta (PANK) (transl. Pavement Industry Advisory Council) based on the results from additional research projects performed in Finland after ASTO, as well as based on the regulations mandatory for the European Union Members (Finland from 1995).

Some of the aspects investigated within the program was the resistance to moisture damage, resistance to studded tires and resistance to freeze-thawing, especially in the presence of deicing chemicals. In the Part 2 of BITU2020 report series, all three of those aspects of influence of water on the asphalt concrete were identified as factors contributing to the moisture damage in the pavement or decrease of the cohesive strength due to the presence of moisture.

A few tests, which currently are not part of the CEN framework or widely popular in literature were performed and found adequate. Because the data exists for the local materials, it is considered important to at least summarize findings from said tests. In case the new materials are introduced on the Finnish market, characterizing them using tests used for development of FAS (Finnish Pavement Technology Advisory Council 2017), could be of interest in the future.

15.2 The surface free energy from contact angle measurements

The theory related to the concept of surface free energy and the work of stripping is presented in Part 2 of the BITU2020 report series.

15.2.1 Principle

The contact angle between a droplet of a liquid in contact with a solid substrate is measured with the use of photogrammetry. The contact angle between solid substrate and model liquids with known surface energies is collected. From those measurements the surface energy parameters of the solid are calculated.

Afterwards the liquid desired for evaluation is tested on the surface of the solid. The surface energy of the tested liquid is calculated using the theory presented in BITU2020 – Phase 1, Part 2 in the section 3.1.4. (Surface tension and adhesive bond). The energy of adhesion, or work of stripping in the system, can then be calculated when the parameters of aggregate, bitumen and water are known.

15.2.2 Method variations

The surface tension between liquid and solid can be measured in various ways. The equipment used for this purpose is called tensiometer. The common methods include:

- Sessile drop tensiometer (goniometer)
- Du Nouy ring tensiometer
- Wilhelmy plate tensiometer
- Du Nouy-Padday method
- Bubble pressure tensiometer.

The last method is used mostly for characterizing differences between surfactants, requires somewhat a transparency of the liquid to observe a bubble inside of it and thus will not be discussed in here.

15.2.2.1 Sessile drop tensiometer (goniometer)

The sessile drop technique evaluates the surface tension of liquid, when the liquid is in form of a pendant. The method can be also used for studies on surface tension between liquid and solid.

In the case of surface tension between liquid and solid, the measurement is based on a basic principle, in which a picture of a droplet on the surface is collected (Figure 180). Alternatively, a shadow of a droplet on a blank background was photographed. Processing of the picture allows to determine the contact angle between the studied solid sample and used liquid (Figure 181).

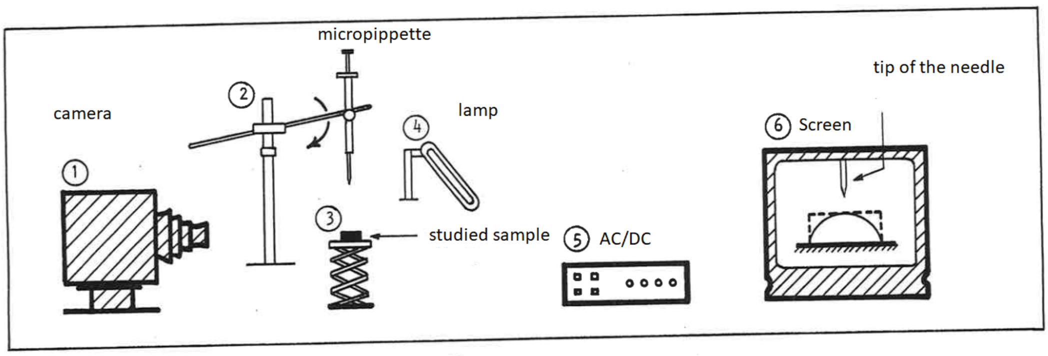


Figure 180. The schematic of the equipment set-up used during ASTO research project to conduct the contact angle measurements (Lyyra 1990).

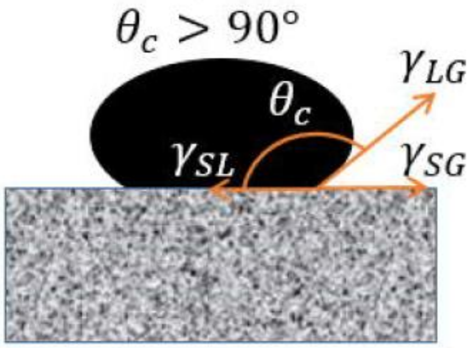


Figure 181. The example of the contact angle between bitumen droplet and aggregate in the context of surface free energy calculation.

In principle at least three liquids of known surface tension in air, need to be used to characterize the properties of aggregate first. In the second stage, the aggregate sample with known surface tension characteristics is tested against the liquid (in this case bitumen). The surface tension between aggregate and bitumen can be calculated.

By adding the third component into the system, namely water, an evaluation of the propensity for stripping can be performed by calculation of the work of adhesion.

15.2.2.2 Receeding and progressing angle (Wilhelmy Plate)

In principle the solid plate is pressed into the liquid and then pulled out of it with a defined force. The liquid into which a solid is pressed, advances on the surface creating the angle between the liquid and the solid. The receding contact angle manifests through hysteresis between the two modes: advancing and receding (Hefer, Bhasin and Little, Bitumen Surface Energy Characterization Using a Contact Angle Approach 2006). The surface tension can be calculated from receding or advancing angle, but the values are varying numerically.

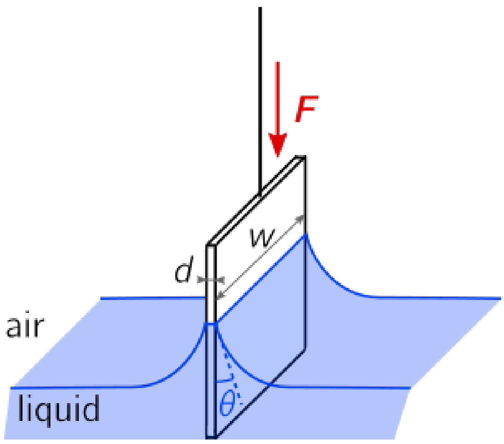


Figure 182. The schematic representation of a Wilhelmy Plate method (wikipedia.org n.d.)

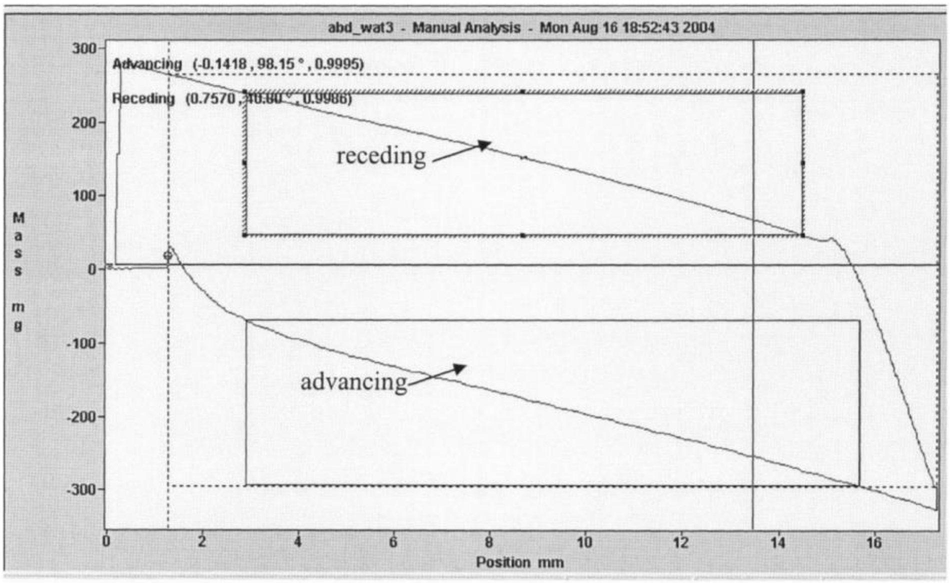


Figure 183. The selection of representative area for regression analysis to determine the Surface Energy from advancing and receding angle using Wihelmy Plate (Hefer, Bhasin & Little 2006.

15.2.2.3 Du Nouy-Padday method

The method uses a small rod with a tip of a shape of a ring, made of composite material, which is immersed into the liquid into certain depth (e.g. 1 mm). Then the rod is pulled out and the maximum force with which the rod is pulled is recorded. The method is not standardized but is referred to as robust and reliable in the industries of surfactants and polymers.

15.2.3 Applicability

The test is being conducted by testing the sample against the number of liquids with known polar and dispersive parameters. They are called reference liquids. Based on the contact angle measurement between sample and reference liquid we can calculate the surface tension of the sample.

Knowing the surface tension of aggregate and bitumen, we can then calculate the work of stripping in the system, as described in Part 2 of the BITU2020 report series. As mentioned in that section, the calculations may vary if the humidity level is different. Additionally, the content of deicing chemical ions in the water will affect the surface tension of the liquid in which the aggregate-binder couple is submerged (Cupples 1945).

15.2.4 Repeatability and reproducibility

Currently the sessile drop method is standardized as SFS-EN 828:2013 *Adhesives. Wettability. Determination By Measurement Of Contact Angle And Surface Free Energy Of Solid Surface.* No repeatability data is provided in the standard.

The solids and bitumen are typically characterized by interaction with liquids of known polar and dispersive component. From that interaction the calculation of surface tension is conducted.

During the review it became apparent, that the number of liquids used in the analysis may influence the numerical results reported by researchers.

- The reports of investigations using 1, 3, 5 or 10 reference liquids were found.
- The time between application of the droplet and collection of the reading is an important aspect due to imbibition into the porous surface of the aggregate.
- There are at least three ways to calculate the surface free energy of material using the measurements of the contact angle as well as other techniques which can be used (Khan, Redelius and Kringos, Evaluation of adhesive properties of mineral-bitumen interfaces in cold asphalt mixtures 2016). Therefore, comparing the numerical results in between the articles, should take account this as a parameter.
- Comparing between the results from the drop method and Wilhelmy plate method is also not advised.
- It is thus suggested to focus on the analysis of the trends recorded by researchers in between the materials studied and to incorporate it into own analysis. However, in order to make conclusions about materials used by the operators, it is advised to perform the measurement using the technique available to operator, testing own raw materials.

One of the problems in looking at the numerical values is connected with temperature. The surface tension is calculated from values of surface tension of reference liquids at temperature of measurement. This is typically in the room temperature or in normal laboratory conditions.

As has been also discussed in Part 2 of the report, the surface tension of water depends on the humidity level in the air. It is likely that similar is observed for reference liquids. The level of vapor saturation with the reference liquid, and also relative humidity, probably will influence the measurements. The problem is not as straight forward as making a picture of a droplet.

15.2.5 Conclusions about surface energy from ASTO

During the ASTO project, one bitumen was tested and 35 aggregate surfaces using the drop method. 10 reference liquids were used to conduct the calculations. Three methods of calculations were used: PNS, Kaelble and Zisman (Peltonen et al. 1989).

The conclusions were that the increasing silicon dioxide content on the surface is increasing the polarity of the surface. Sodium and potassium content on the surface correlated with increased polarity and decreased adhesion. (Biotites contain Na and K ions, and were afterwards limited in the materials allowed for road construction.) Increase of iron (Fe) was increasing polarity but not decreasing the adhesion. Increasing calcium ions were decreasing polarity, but the adhesion was not increasing with increasing Ca content (Peltonen et al. 1989).

The strict connection between polarity of aggregate and adhesion was not found. It was proposed that production may have the effect not measured in this test, e.g. the temperature at which the adhesion happens (Peltonen et al. 1989).

One of the more interesting things investigated during the ASTO program, which perhaps did not come forward in the literature reviewed from international sources, was connected with the effect of temperature of the aggregate and bitumen during the measurement on the contact angle (Lyyra 1990). Obviously, the bitumen needs to be heated before application in form of a drop onto the aggregate. The Contact angle decreases with increasing temperature (Figure 184 and Figure 185). Since we cannot drop stiffer bitumens at temperatures between 5–40 °C, the value of contact angle may be misleading, when obtained at higher temperatures.

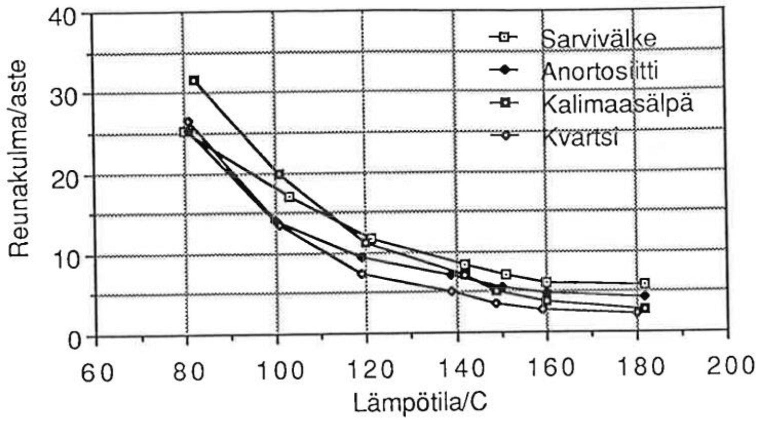


Figure 184. The contact angle of Arabian Heavy (AH) bitumen measured at different temperatures against multiple minerals (Lyyra 1990).

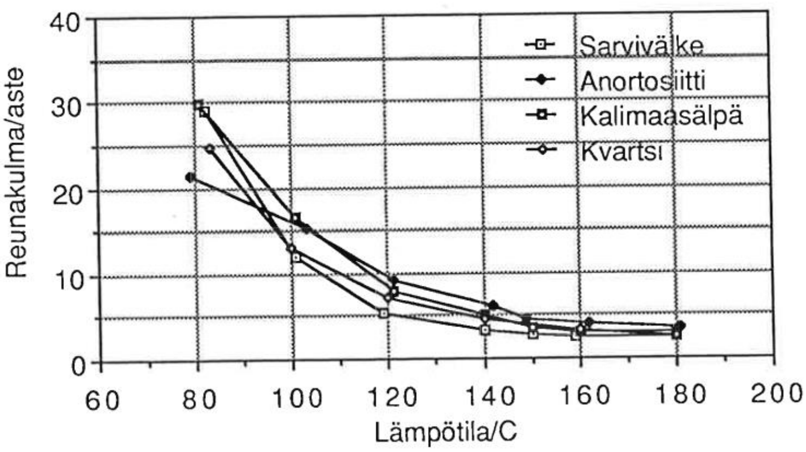


Figure 185. The contact angle of Russian bitumen (A) measured at different temperatures against multiple minerals (Lyyra 1990).

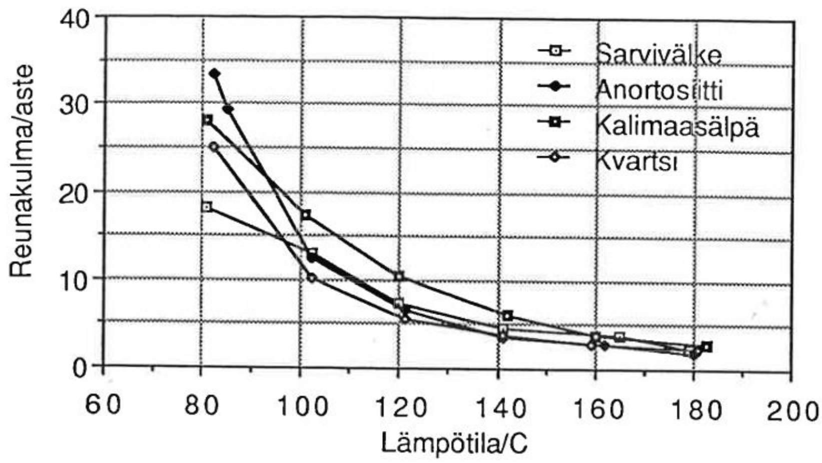


Figure 186. The contact angle of the Laguna bitumen (LAG) measured at different temperatures against multiple minerals (Lyyra 1990).

15.2.6 Conclusions from international literature after ASTO

The surface tension concept has been evaluated internationally to figure out if differentiation between binders can be achieved via the contact angle measurements (Hefer, Bhasin & Little 2006). The differentiation between the binders can be achieved when using a myriad of solvents (A. Hefer 2004). The use only one solvent is not advised to differentiate between the bitumens. However, in order to study the effect of the additive on the contact angle, i.e. following of the relative change, such approach could be acceptable.

As shown in the Figure 187 and Figure 188 addition of the adhesion promotors such as silanes can alter the contact angle of the binder obtained after the modification. This in turn affects the surface tension and work of adhesion. However, it is worth to note on the example of presented figures, that an optimal amount of additive needs to be established using the materials considered for use. In the case of the studied silane-based modifier, the significant improvement can be observed at the levels between 0.6–0.7% (Liang et al. 2011). Using more of the modifier did not have significant further effect on the blend. Using too little or too much of the additive is possible if the mix design is not optimized. The contact angle test could be a simple technique to establish such optimal binder composition for a given set of asphalt components.

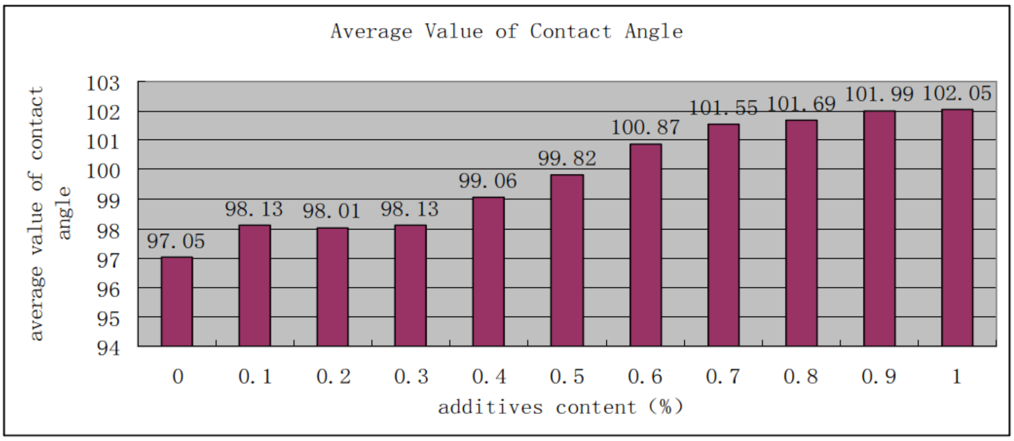


Figure 187. The average value of contact angle of bitumen with Penetration 65 dmm and Softening Point of 63,9 °C with modifier γ-(methacryloyloxy) propyl trimethoxysilane used at respective volume (Liang et al. 2011).

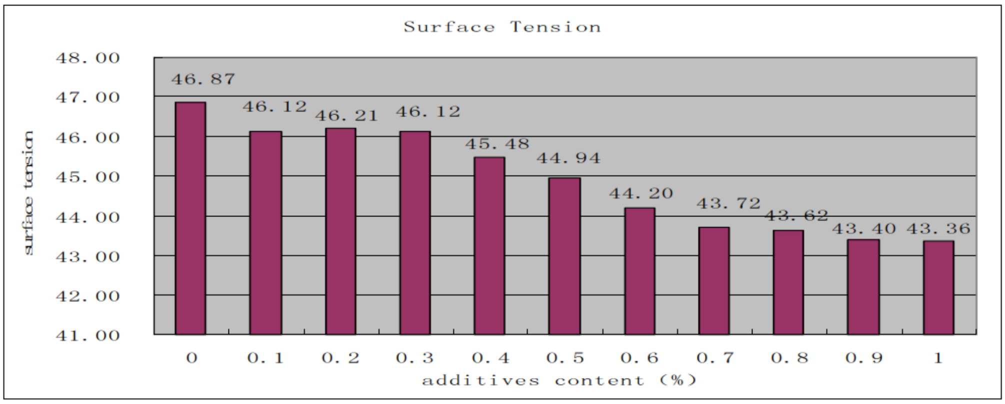


Figure 188. The average surface tension of bitumen with Penetration 65 dmm and Softening Point of 63,9 °C with modifier γ-(methacryloyloxy) propyl trimethoxysilane used at respective volume calculated using the contact angle data.

One of the biggest research on the subject in terms of binders, have used only two polar liquids to characterize the materials. This is the weakness for comparison of the results numerically (Zhang et al. 2018). Nevertheless, the research compares different binders and the effect of aging on the stripping. The lower the work of stripping, it is considered that the material is less susceptible to moisture damage. Aging of different binders influences the stripping work in a different way (Zhang et al. 2018). The aspect of oxidation of binder and its propensity to oxidation (increasing the polarity), is proposed to influence the stripping and moisture damage. The effect on the adhesion due to aging is also different between binders when different types of aggregates are considered (Figures Figure 189–Figure 191) (Zhang et al. 2018).

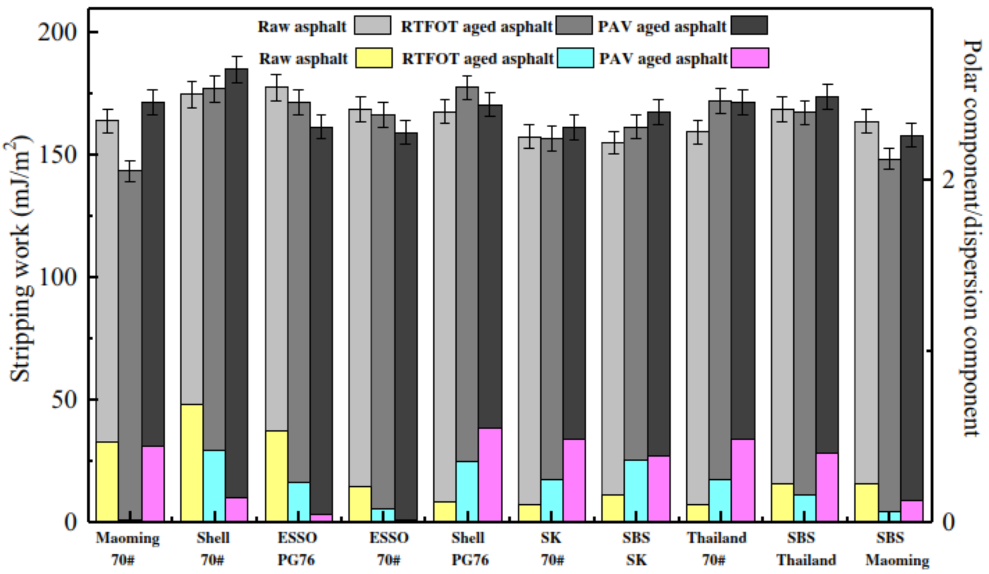


Figure 189. The stripping work and the ratio of the surface free energy component of asphalt-limestone in pre and post aged version (Zhang et al. 2018).

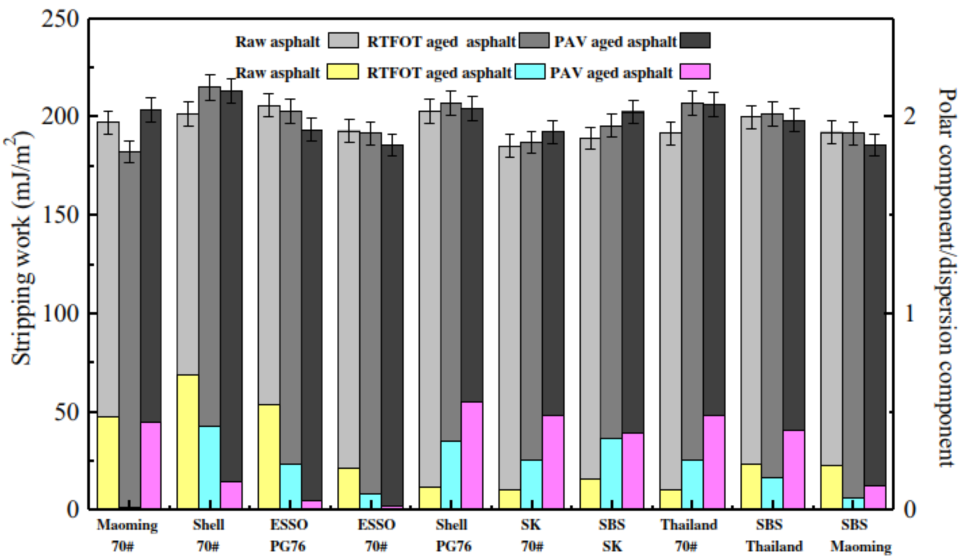


Figure 190. The stripping work and the ratio of the surface free energy component of asphalt-granite in pre and post aged version (Zhang et al. 2018).

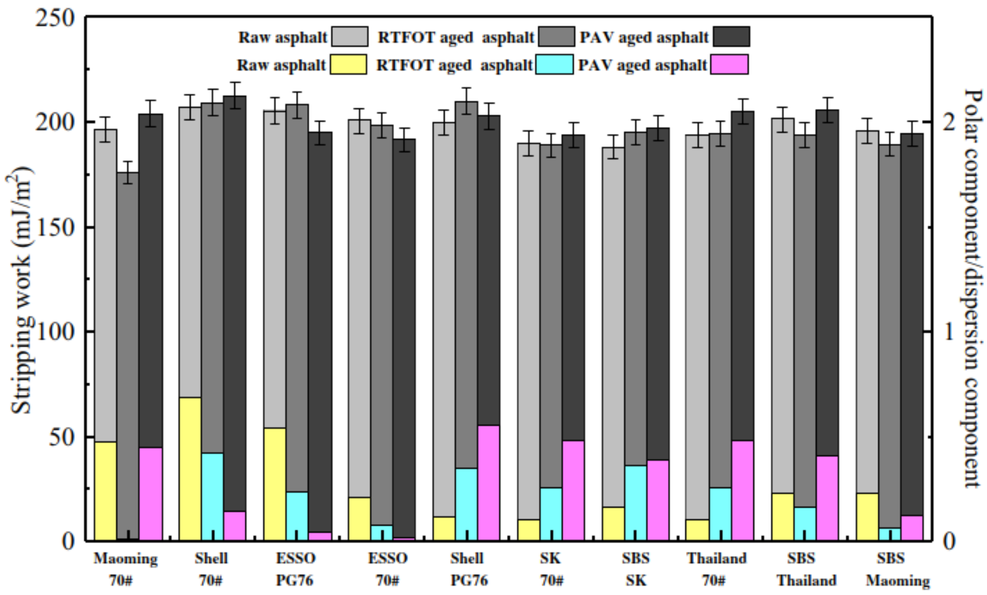


Figure 191. The stripping work and the ratio of the surface free energy component of asphalt-diorite in pre and post aged version (Zhang et al. 2018).

15.3 The Hallberg test

15.3.1 Principle

The test allows for determination of the adhesion force between binder and aggregate. According to this test the adhesion force is the highest force inflicted by the pressure of water, which the binder resists without detaching from aggregate surface. The adhesion force is expressed in mN/m (Pylkkänen & Kuula-Väisänen 1989).

In the test two ranges of aggregate gradation are used 0.5–1 mm and 0.074–0.125 mm. The binder is dissolved into the kerosene in the volumetric ratio of 1 part to 3 parts of kerosene (Pylkkänen & Kuula-Väisänen 1989; Kannisto et al. 1979).

Figure 192 presents the schematic of the equipment necessary for the execution of the test. Into the glass tube, opened from both ends, a layer of 0.8 cm of aggregate with gradation 0.5–1 mm is compacted as a filter layer. On top of this layer a second layer of 0.8 cm of aggregate size 0.74–0.125 is compacted. Such prepared column is placed on the U ring in a glass dish of 5 cm diameter. The binder solution is applied into the dish, to the level of filter layer, and the solution is allowed to cover the fine aggregate by capillary suction. Afterwards the obtained sample is allowed to stabilize for 1 h. After stabilization (also referred to as aging) takes place, the water is applied slowly by pouring onto the wall. Such prepared sample is left for 24 h (Pylkkänen & Kuula-Väisänen 1989; Kannisto et al. 1979).

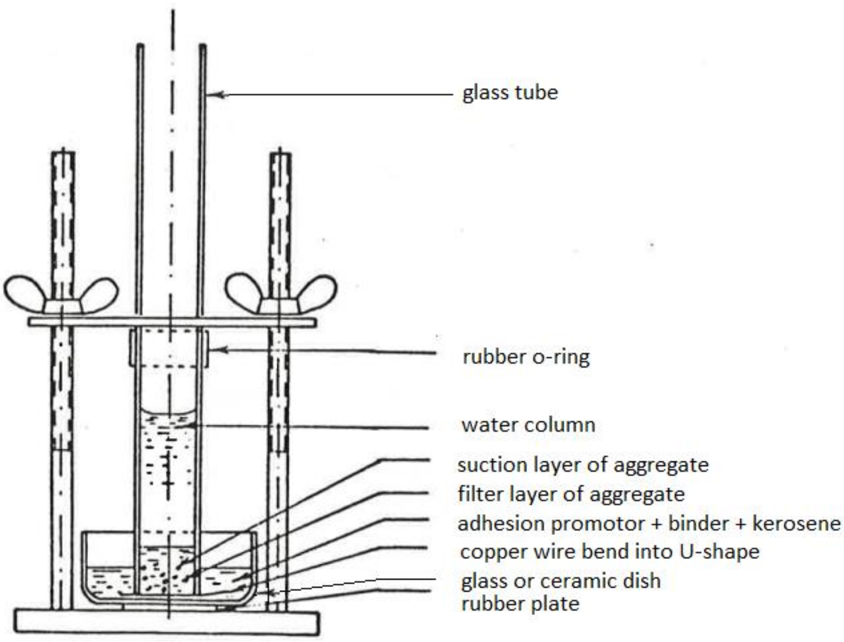


Figure 192. The equipment necessary for evaluation of adhesion force according to Hallberg test (Kannisto et al. 1979).

A series of tubes is prepared from one aggregate, with an incrementing height of water column. Every next tube has additional 20 mm higher water column on top of the sample. The evaluation after 24 h looks which samples gave way to water (allowed the drain down). The highest resisting column height is considered in further calculations (Pylkkänen & Kuula-Väisänen 1989; Kannisto et al. 1979).

The adhesion force can be calculated from equation

$$\Sigma = \frac{d*981}{4}(hv + 0.5),$$

where

Σ – adhesion force (mN/m)
hv – the largest water column which is not passing through the sample (cm)
d – the average capillary diameter of the aggregate in the suction layer (Kannisto et al. 1979)

The average capillary diameter can be determined using the capillary flow meter (Pylkkänen & Kuula-Väisänen 1989).

15.3.2 Applicability

Prior to the ASTO program, the Hallberg test was used to study compatibility between aggregate and bitumen cut-backs or road oils. The grain fraction limits its applicability for mixtures containing large stones. The preparation of the fines may be required, which may necessitate large volume sieving or crushing of the rock.

During the ASTO program ten aggregate types with a specifically defined mineralogy were studied in combination with three bitumens of different origins (Russian, Venezuela and Arabian Heavy). The bitumens were of the grade B120, which is currently corresponding to bitumen 100/150 Penetration grade. The differences in adhesion force between bitumens and minerals were observed for those combinations. However, for each bitumen the correlation to mineralogy was different, as well as the relation to other methods such as Rolling Bottle or ITSR (Pylkkänen & Kuula-Väisänen 1989).

15.3.3 Repeatability and reproducibility

The aspects affecting to the uncertainty, repeatability and reproducibility of the measurement were identified as:

- The diameter of the capillary
    - The measurement of the capillary diameter depends on the tube in which the measurement is performed, thus it should be conducted on compacted sample in the tube of the same diameter (Pylkkänen & Kuula-Väisänen 1989).
    - The measurements performed during the ASTO program were performed using the mercury porosimeter, and the difference in this method is that the aggregate is not compacted prior to measurement, as it would be with air-pressure flow meter. The authors argue that the maximum height of the water column above the compacted and uncompacted samples is similar during the test, but advise that their numerical values may differ from those reported by Hallberg himself. However, all of the measurements for the purpose of ASTO program were conducted in a similar way, which allows for comparison between materials. It is also advised to take this account when designing any further testing on additional materials (Pylkkänen & Kuula-Väisänen 1989).
- The aging time of the solution of bitumen in kerosene
    - Aging occurs logarithmically, and 1 h was chosen for Finnish test as an approximation of stability (Kannisto et al. 1979),
    - but in the ASTO research program it was observed and decided that 4 h is the sufficient aging time (Pylkkänen & Kuula-Väisänen 1989).
- The time of the water standing over the prepared sample – according to Hallberg the detachment happens after 24 h and that is the time chosen.
- The temperature of the test – the temperature of aggregate, solution of bitumen and water should be kept constant at approximately 20 °C.
- The level of moisture in the aggregate prior to test – the moist aggregate is not getting as high adhesion with binder as the completely dry aggregate.

It was determined that the test is almost trustworthy. The improvement of the repeatability and reproducibility should be done by the conduction of replicate tests and interlaboratory comparison. Unfortunately, the standard deviation of the resulting tests was not provided (Pylkkänen & Kuula-Väisänen 1989).

15.3.4 Summary from the ASTO report

The Table 28 presents the results obtained for ten tested aggregate types and three bitumens. The bitumens were all of the penetration grade B-120 and were of three origins: Arabian Heavy (AH), Venezuelan (LAG) and Russian (A) (Kurki, Halttunen et al. 1993). In the following stage an addition of adhesion promoters was investigated in those combinations.

Table 28. The results of the Hallberg test in ASTO program (Pylkkänen & Kuula-Väisänen 1989).

		B120 AH		B120 LAG		B120 A	
Aggregate [name]	d [cm]	hv [cm]	Σ [mN/m]	hv [cm]	Σ [mN/m]	hv [cm]	Σ [mN/m]
Olivine	0,0028	16	11,33	15	10,64	22	15,45
Peridotite	0,0030	20	10,06	10	7,73	20	15,08
Gabro Kemiö	0,0023	10	5,92	7	4,23	10	5,92
Diabase	0,0028	5	3,78	8	5,84	9	6,52
Quartzite	0,0025	18	11,34	1	0,92	23	14,41
Quartz	0,0040	5	5,37	0	0,49	15	15,13
Calcite	0,0024	16	9,51	12	7,20	17	10,09
Feldspar	0,0029	0	0,36	0	0,36	5	3,91
Granodio-rite	0,0020	0	0,25	2	1,23	2	1,23
Granite	0,0023	0	0,28	2	1,41	5	3,10

The results of the test were compared with results of ITSR and Rolling Bottle test. It was found that:

- For bitumen B120 A the relation between Hallberg and ITSR test results was insignificant (r = 0,05), in comparison the relation between ITSR and Rolling bottle test results were also doubtful (r = -0,23)
- For the bitumen B120 AH the correlation between Hallberg and ITSR test results was meaningful (r = 0,72), in comparison the relation between ITSR and Rolling bottle test results was less apparent (r = 0,57)
- For the bitumen B120 LAG the relationship between Hallberg and ITSR test results was meaningful (r = 0,9), in comparison the relation between ITSR and Rolling bottle test results were also meaningful (r = 0,74)

The Hallberg test was shown to capture the differences between bitumens. The ITSR test also captures some differences between the three used bitumens. However, the standard deviation or repeatability range for the measurement is suggesting that the difference between the behavior of bitumen with aggregate is indistinguishable by ITSR test. Unfortunately, the repeatability of Hallberg test was not defined in the report, thus it is hard to establish how meaningful is the differentiation by Hallberg test (Pylkkänen & Kuula-Väisänen 1989).

For the B120 A it was determined that the results should be compared using Hallberg test, ITSR and Rolling Bottle, for B120 AH using ITSR and Rolling Bot-

tle, and for B120 LAG using only ITSR test. This is based on the correlation coefficients. The LAG and AH type bitumen were correlating easily to Hallberg test results with the r values close to 1.

Perhaps based on this evaluation it was established that ITSR is the best test for all bitumens. However, it is underlined hereby that the Russian bitumen was analyzed using three test methods because of the conflicting results coming from each of the methods applied. **This is why perhaps a three-method evaluation of affinity between bitumen and aggregate would be more appropriate to understand the behavior when a new binder or aggregate is tested.**

15.3.4.1 The acidity of the aggregate

The acidity on the surface of the aggregate is coming from Brönsted acids (e.g. -Si-OH) or Lewis acid (e.g. -O-Al^+-O-). The basicity on the surface of the aggregate may come from -Si-O- or -O-Al^--O- bonds.

The acidity of the aggregate was established by titration of the fines passing 0.074–0.125 mm with trichloroacetic acid (CCl_3COOH) and n-butylamine ($CH_3(CH_2)_3NH_2$), when the materials BET-surface area was known. The titration is followed either with the use of indicators or with the use of calorimeter, as during the neutralization reaction a heat is excreted from the system.

The acidity of the aggregate was correlating meaningfully for Hallberg test for B120 A (r = 0.82), for Rolling Bottle test of B120 A (r = 0.73) and B120 AH (r = 0,75). However, the relationship between acidity and ITSR tests were not apparent (r = -0,46 …. 0,37).

It was concluded that with the increasing content of SiO2 in the aggregate, the adhesion between aggregate and binder becomes weaker. However, the Russian bitumen B120 A behaved differently, with the increasing SiO_2 the adhesion was improving. This was explained using the titration of the bitumen samples with the hydrochloric acid (HCl) and potassium hydroxide (KOH) to establish their acidity and basicity (Figure 193). The bitumen A and AH has the least acidic species and they were all in the form of phenols, while LAG had rather many acidic species in the form of carboxylic acids. In terms of basicity of the bitumen, the A type had the highest value of the three (Pylkkänen & Kuula-Väisänen 1990).

Addition of the amine-based additives was increasing the basicity of the prepared binders regardless of the base bitumen used (e.g. Figure 194). The use of acidic additive, such as pine oil was increasing acidity (Figure 195). When studied with Rolling Bottle test it becomes apparent that addition of basic additives (including hydrated lime) was improving the adhesion between LAG and aggregates. Adding amine to AH improved adhesion, while adding pine resins decreased the adhesion, both for granite and granodiorite (the most problematic from the point of view of moisture damage, yet the most abrasion resistant).

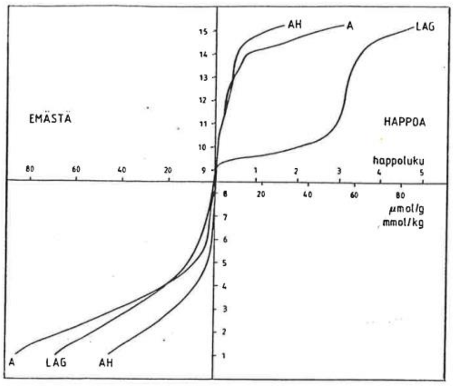


Figure 193. The titration curve of bitumen A, AH and LAG (Pylkkänen & Kuula-Väisänen 1990).

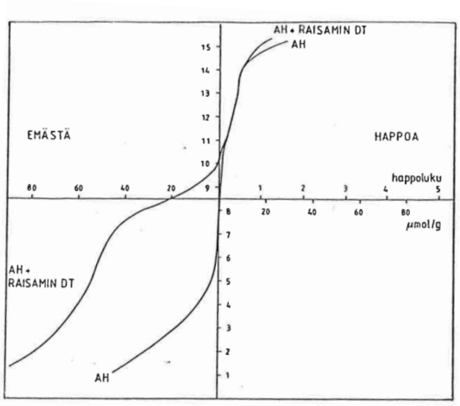


Figure 194. The titration curve of bitumen AH with an addition of Raisamin additive depicting increased basicity (Pylkkänen & Kuula-Väisänen 1990).

It is advisable to consider evaluation of the acidity and basicity of the bitumen prior to mix design and choice of aggregate in the market of uncontrolled bitumen sources, based on the previous experiences reported above.

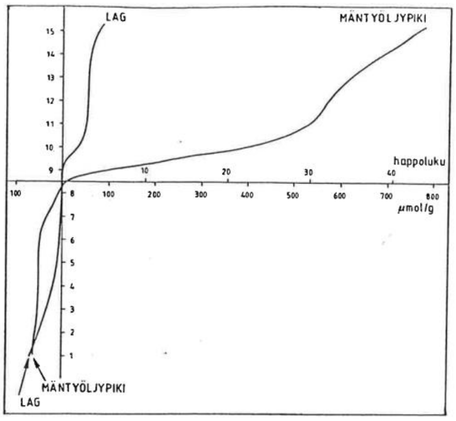


Figure 195. The titration curve of bitumen LAG compared to the titration curve of pine based tar, indicating that the acidity of this product is higher than of the most acidic of considered bitumens (Pylkkänen & Kuula-Väisänen 1990).

15.3.4.2 Zeta-potential

The zeta-potential describes a charge on the surface of either bitumen or aggregate. One of the mechanisms expected to influence the loss of adhesion is connected with repulsion and attraction forces between the molecules (dipols) at the surface of bitumen and aggregate (Caro et al. 2008). As presented in the Figure 196, the higher the difference between the zeta-potential of component A and B at given pH, the more likely the attraction between them, especially if A and B are of the opposite charge. If A and B are of the same charge, the repulsion is likely to be observed.

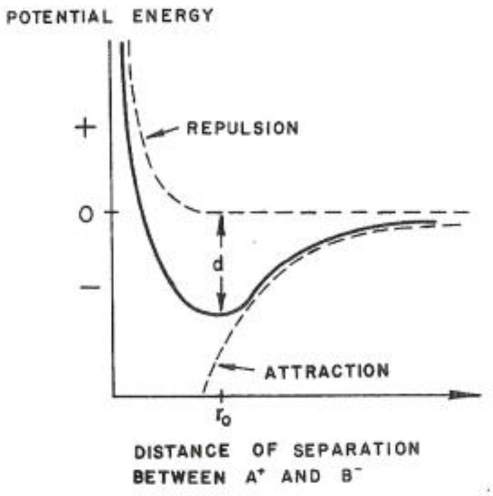


Figure 196. The potential energy as a function of separation distance at a temperature of absolute zero (Kittrick 1977).

Due to the formation of the so-called Stern layer on the surface of minerals due to the internal diffusion of cations and anions inside the mineral, the surface has a charge. Depending on the pH in which the mineral, or a liquid, is measured, the charge is different due to different interactions of surface with anions and cations of the environment (Lyyra 1990).

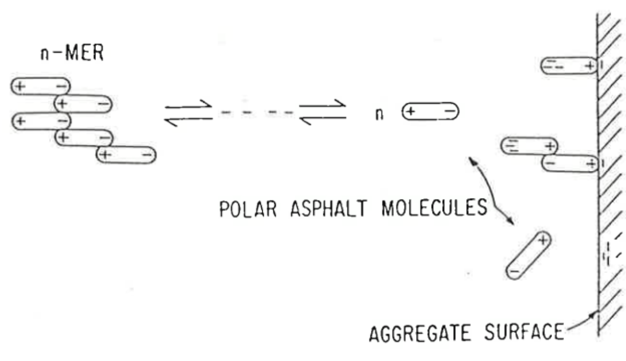


Figure 197. The schematic of the multilayer absorption (Lyyra 1990).

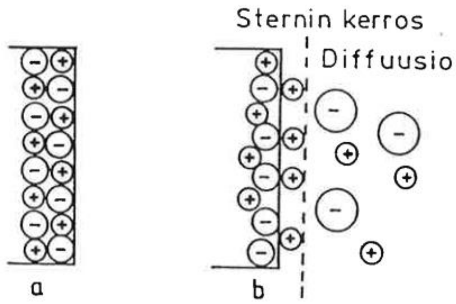


Figure 198. The formation of the charge on the surface of the mineral in a) dry and b) moist environment (Lyyra 1990).

15.3.4.2.1 Principle

The measurement is performed so, that a tube filled with liquid of known pH is placed between anode and cathode. The material is applied in form of a powder from one edge. The time it takes the powder to travel from one end to another end of the tube is then recalculated into the electric potential on the surface. (Lyyra 1990)

15.3.4.2.2 The effect of mineralogy of ASTO aggregates on the zeta-potential

The minerals characteristic for the granites and granodiorites were tested to study their separate effects on the adhesion. The anorthosite (mainly composed of plagioclase feldspar) had the highest zeta-potential, especially in the pH range of 4–10 (Lyyra 1990), which could be observed in field.

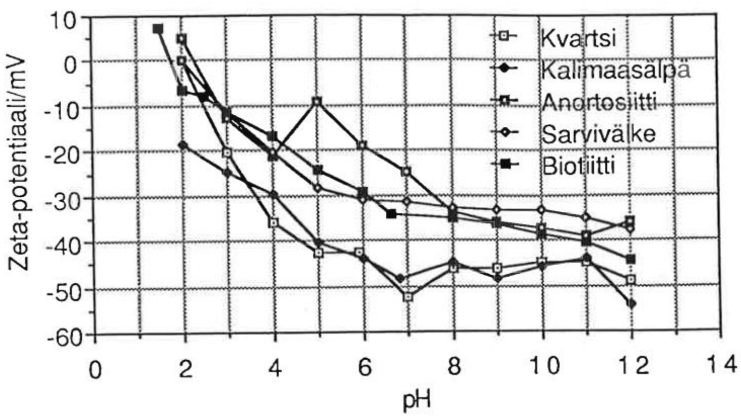


Figure 199. The zeta potential of tested minerals in the function of pH (Lyyra 1990).

To provide the context of the value of the research conducted during ASTO project, it is worth to demonstrate the level of the research reported in the international literature on the subject. Indeed, different bitumens within SHRP program were characterized and it is implied that their properties are different, indicating that each bitumen has a different propensity to stripping (Labib 1992). The work performed during the ASTO program, presented in the following section, about the effect of the heating on the zeta potential could still be considered novel knowledge for the industry.

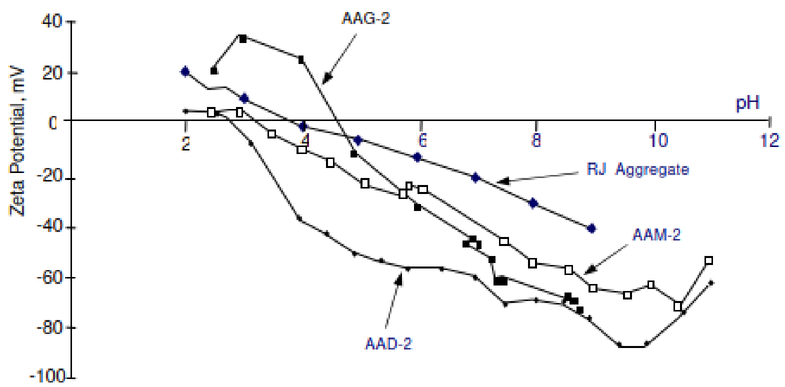


Figure 200. Interactions diagrams for granite aggregate (RJ) with bitumen (Labib 1992).

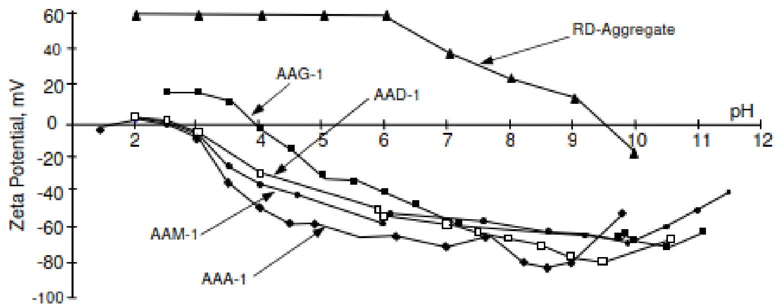


Figure 201. Interactions diagrams for limestone aggregate (RJ) with bitumen (Labib 1992).

15.3.4.2.3 The effect of heating aggregate on their zeta potential in ASTO

The zeta potential was applied to study the effect that the heating of the aggregate may have on the adhesion. The aspect of the effect of heat on the aggregate properties is of importance due to the heat that is inflicted on the aggregate during drying inside of the drum. Especially, in the context of recent advancements in terms of increasing use of recycled asphalt pavements, often a method called superheating of the aggregate is used to assure the proper temperature of final mixture when RAP is cold-feeded into the production (Kandhal & Mallick 1997).

According to the study performed within ASTO some aggregates are not susceptible (potassium feldspar, biotite, anorthosite), but some express different behavior after heating – hornblende decreases its zeta potential, while quartz increases (becomes more susceptible to repulsion) (Lyyra 1990).

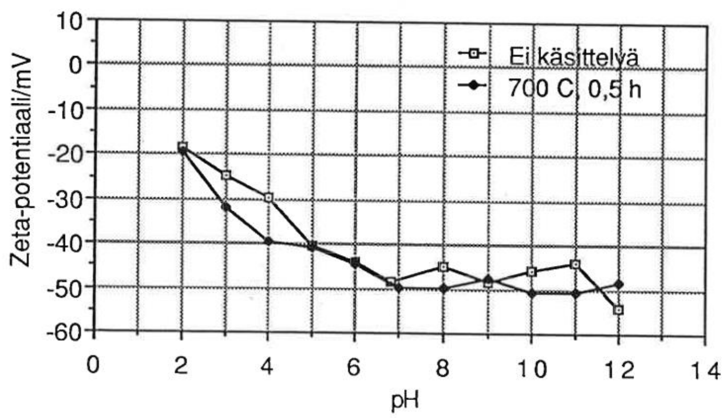


Figure 202. The effect of the heating to 700 °C of potassium feldspar on its zeta potential (Lyyra 1990).

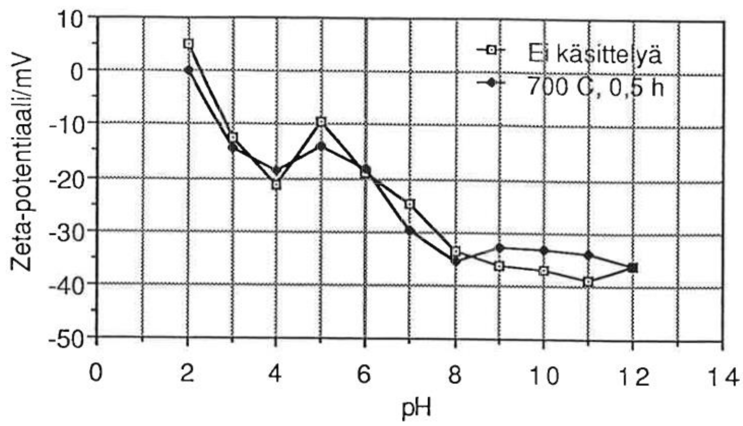


Figure 203. The effect of heating anorthosite to 700 on its zeta potential (Lyyra 1990).

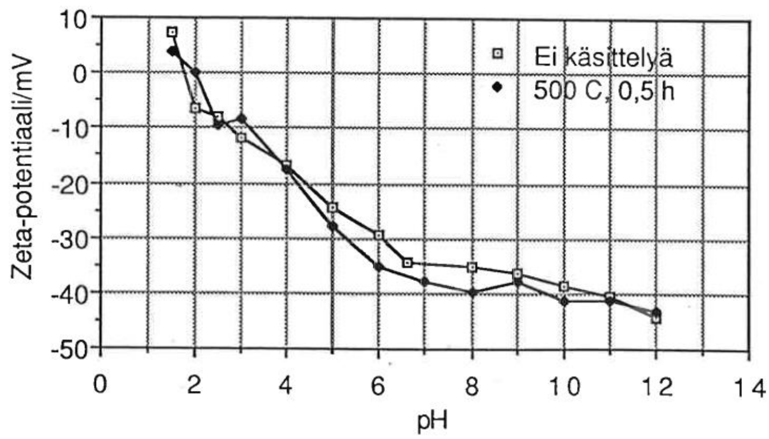


Figure 204. The effect of heating to 500 °C of biotite on its zeta potential (Lyyra 1990).

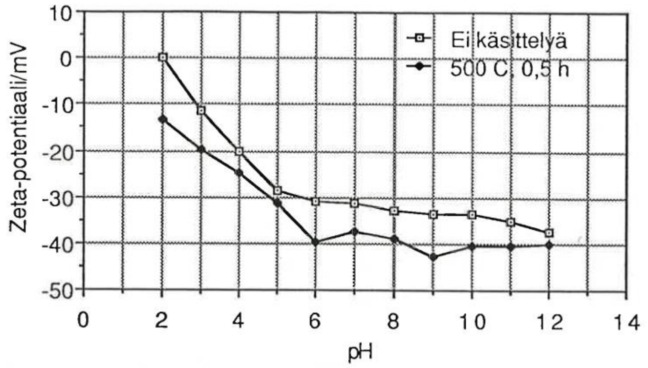


Figure 205. The effect of the heating to 500 °C on the zeta potential of hornblende (Lyyra 1990).

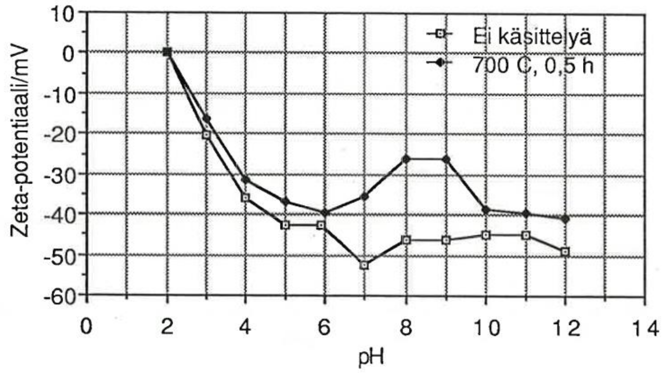


Figure 206. The effect of the heating to 700 °C on the zeta potential of quartz (Lyyra 1990).

15.3.4.2.4 Supporting tests for the zeta potential evaluation during ASTO

The author of the work package investigating the zeta potential of bitumen and aggregates, discussed in his work also the effect of the overdose of the adhesion promotors to the mixture, or the wrong choice of the adhesion promotor for the aggregate surface (Lyyra 1990).

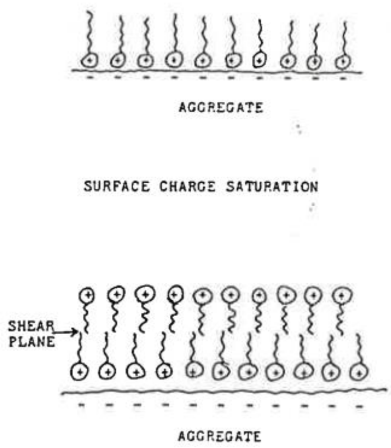


Figure 207. The schematic explanation of the potential negative effect of the overdose of amine as an additive.

For the purpose of the demonstration, Lyyra developed a test investigating the level of desorption of bitumen from the loose mixture (Lyyra 1990). The samples were stored in water, and the bitumen which detached in the process and transferred into the water was removed. Afterwards the bitumen from the remaining mixture was extracted and its mass compared to the initial mass applied into the mixture (3.5% by weight). The detached bitumen (Irronut bitumi, %) was then evaluated for reference from 18 couples obtained by blending 3 bitumens (Russian, Arabian Heavy and Laguna) and 6 different minerals (Figure 208 and Figure 209).

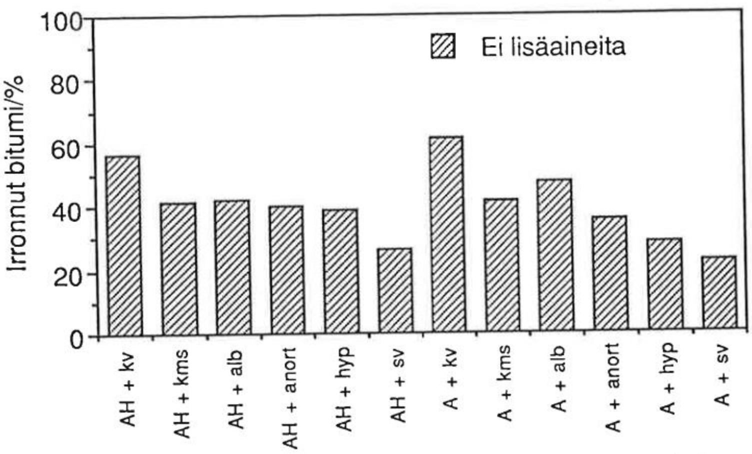


Figure 208. The detached bitumen for AH and A bitumens when mixed with various minerals as reference.

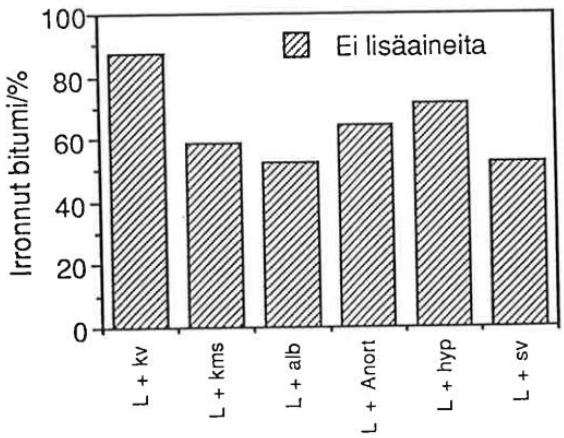


Figure 209. The detached bitumen in reference samples using laguna bitumen with different minerals.

The author demonstrated how adding a fixed amount of Raisamin 200, for example 0.5% or 1.5% to all mixtures would affect the percent of the detached bitumen (Lyyra 1990) (Figure 210 and Figure 211). Lyyra compared it to the optimal dosage of this additive. The study (Lyyra 1990) underlined strongly, why the idea of suggesting to use one amount of adhesion promotor for all mixtures in Finland is not a viable solution, when the mineralogy of aggregates and sources of bitumens differ. Every mix design needs to be conducted separately. Different adhesion promotors are also going to have a different effect at the same percentage of addition to the mixture.

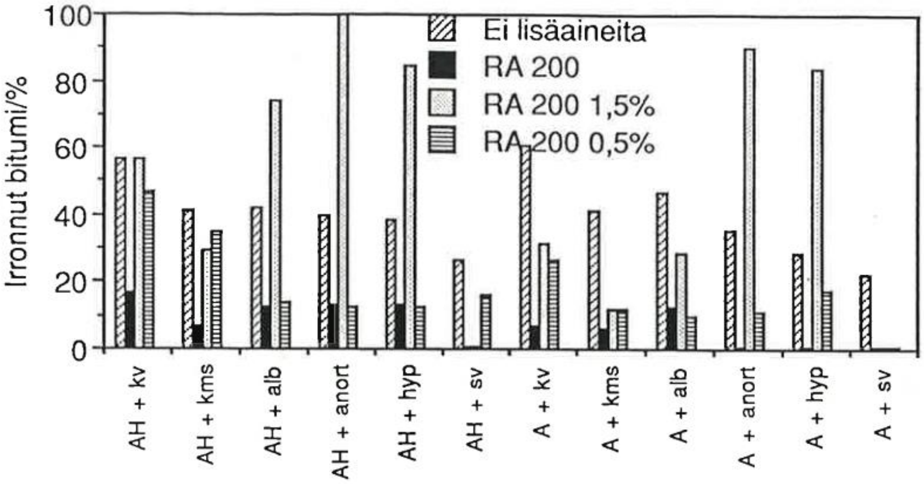


Figure 210. The detached bitumen levels when Raisamin 200 was added as adhesion promotor at 0.5%, 1.5% and optimal dose levels in mixtures using Arabian Heavy and Russian bitumen.

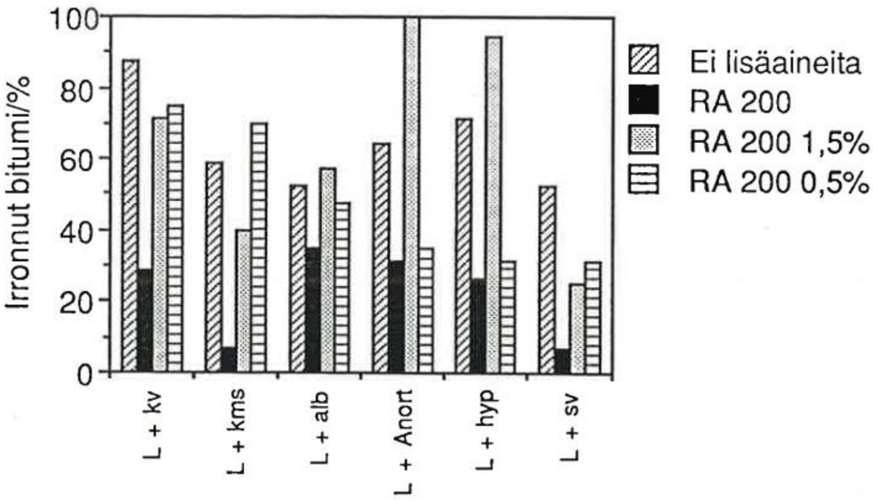


Figure 211. The detached bitumen level from mixtures in which Raisamin was used at optimal level, at 1.5% or 0.5% level in mixtures with different minerals and Laguna bitumen.

15.3.4.2.5 Suggestion

The surface potential of the mineral in the aggregate will determine the amount of the additive to be added. Not only the type of additive, but the amount of it may influence positive and negative outcomes.

The heating of the aggregates or overheating of aggregates, may alter the surface properties of the minerals and the adhesion between bitumen and aggregate. Therefore, in the systems in which the aggregate is overheated, this should be simulated in the laboratory as well prior to evaluation of the adhesion properties. A sample collected from the plant mixing is suggested for testing of the affinity or resistance to moisture damage.

Bitumen composition (polarity), chemical charge and acidity affects the chemical adhesion. The acidity of the binder seems to influence the interaction in terms of chemistry. The acid to acid contact between such materials as quartzite and laguna binder, caused the most stripping. Hence the knowledge of the aggregate surface properties, as well as acidity and basicity of the binder should be most indicative of propensity to stripping. This knowledge could also contribute to the choice of proper adhesion promotors. The level of acidity should be related to the amount of adhesion promotors necessary to achieve good adhesion.

15.3.4.3 The effect of the water adsorption

As mentioned above, the acidity of the aggregate can be established in multiple ways. One of the investigated ways was the absorption of vapors of five solvents, namely:

- chloroform ($CHCl_3$) and carbon tetrachloride (CCl_4) as acidic molecules
- benzene (C_6H_6) and butylamine ($C_4H_9NH_2$) as basic molecules
- water in order to establish hydrophilicity and hydrophobicity of aggregate. (Pylkkänen & Kuula-Väisänen 1989).

The method applied in the study is currently described in PANK 2108 instruction (Päällystealan Neuvottelukunta ry 2009) using water as a solvent, as the significance of the hydrophilicity became apparent. The difference between the results obtained in 1989–1990 (Pylkkänen and Kuula-Väisänen, Bitumin ja Kiviaineksen välinen tartunta asfalttipäällysteessä - OSA I 1990) and the current instruction of the procedure (Päällystealan Neuvottelukunta ry 2009) lay in the fine fraction definition. During the ASTO research program the fines were defined as material passing 0.074 mm, while currently they are defined as 0.063 mm.

The findings of the research project indicated a strong relationship between water adsorption per surface area of the studied aggregate and decreasing adhesion between binder and aggregate as tested by all the methods. Still, the Russian origin bitumen tested by the ITSR test was the least susceptible to this parameter (Pylkkänen & Kuula-Väisänen 1990).

The materials such as quartz and feldspar had water adsorption values of 12.88 and 11 mg/m^2, respectively. For those materials the adhesion between bitumen and aggregate was the lowest, thus a conclusion to set the cut-off value for water adsorption at 10 mg/m^2 was logical.

15.3.4.4 The effect of the mineral composition

The relationship between calcium and magnesium in the aggregates was not apparently correlating with resistance to moisture damage. However, the increasing content of silica (SiO2) correlated with decreasing affinity in all tests. However, the effect was most significantly visible for less basic bitumens, such as AH and LAG.

Interestingly, the increasing iron content was correlating with increasing resistance to moisture damage in all tests for aggregates (not pure minerals) (Pylkkänen & Kuula-Väisänen 1990). This effect was also studied in the parallel project, where the increasing amount of iron was linked to increased ratio of the polar component (γ^p) to the dispersive component (γ^d) of the surface free energy of the aggregate (see report BITU2020 – Part 2, equation 5) (Peltonen et al. 1989). It was expected that the adhesion resistance would decrease with increasing polarity of the aggregate surface as bitumen was characterized by a relatively low polar component (Pylkkänen & Kuula-Väisänen 1990). The theory of polarity as an explanation of behavior at the interface of the aggregate and binder was thus disproved due to conflicting results.

15.3.5 Influence to the Finnish Asphalt Specifications

Based on the above discussed (section 15.3 and studies on weathering of aggregates (Nieminen & Jänniskangas 1990) (discussed in WP2_T1), the formulation in the Finnish Asphalt Specifications states that feldspars, quartz and micas are especially influencing the lower adhesion. The effect of the composition of the aggregate was found higher than the effect of bitumen composition. However, the adhesion is governed by the physico-chemical composition of the interface between the two. Suggestion is to study the water adsorption value prior to mix design to evaluate a need of adhesion promoter (Finnish Pavement Technology Advisory Council 2017).

Specification requirement based on the findings of ASTO research program was defined as good affinity, if the water adsorption is below 10 mg/m^2. (Finnish Pavement Technology Advisory Council 2017)

15.4 Abrassion by studded tyres and bitumen

15.4.1 Test tracks in Finland

During the ASTO program between 1988 and 1991 a series of test roads was prepared: 16 asphalted roads, 6 oil gravel roads and 2 gravel roads. For the evaluation of the influence of various parameters on the abrasion, the test road always comprised a short stretch of reference mixture AB20/IV in which Teisko granodiorite and Arabian Heavy B-120 was used. The test roads included:

- Aggregate test roads (different sources, different methods of crushing)
- Fiber test road (arbocel, dacron and wolmix)
- Different modulus mixtures (using different bitumens and gilsonite)
- Filler test road
- Bitumen content test road
- Adhesion promoter test road
- Influence of the aggregate type in fraction below 8 mm and above 0.125 mm
- Thermal cracking test road (using different binders).

The tests performed in laboratory, as well as the tests performed in the test track were compared to the field tests in order to evaluate the performance predictive strength of the tests. When the test results are reported in comparison (fin. suhtelinen) to the reference mass, they are always related to the AB20/IV/granodiorite and AH B-120 mixture.

15.4.2 Test tracks in Neste Oy

The speed of the wheel at the test track was between 50–65 km/h, but the desire was to achieve 65 km/h speeds. The speeds investigated were 30 and 65 km/h. At low temperatures the impact of the traffic was explained to had contributed to the loss of asphalt particles (Blomberg 1991). Even though the proceeding related to the reports on the wear tracks and pavement wear resistance testing of mixtures using various binders, indicate that binder has no effect on the abrasion, the results in fact indicate that in the temperatures around -5 °C, the difference could be significant (Figure 212) (Blomberg 1991).

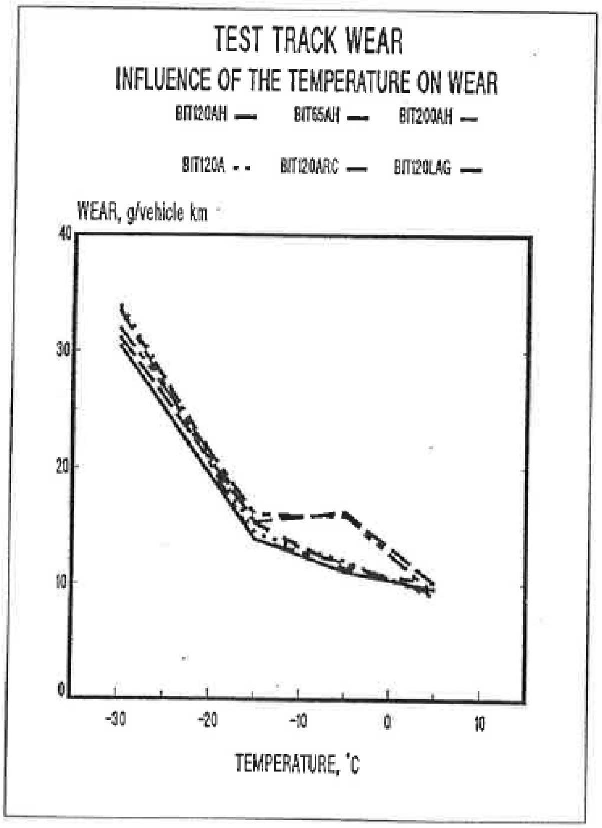


Figure 212. The results from the test track wear at 65 km/h for various bitumen samples at temperatures between -30 and 5 °C.

Nevertheless, the observations suggested that both the increasing speed and decreasing temperature in dry conditions were contributing the increase in wear (Blomberg 1991) (Figure 212 and Figure 213). However, as is visible in Figure 213 and Figure 214 the influence of moisture at +5 °C seemed to have the most significant effect on the abrasion. The results were reported as a ratio, rather than absolute values from the test tracks, and unfortunately it is impossible to conclude if the wear was in fact different between the bitumens. The numerical results are provided only for Pavement Wear Resistance (PWR) tests, where the differences in ml abraded from samples at low temperatures can be in the range of 100% between tested bitumens (Blomberg 1991). It would be worth to challenge the conclusions of the origin of the assumption in Finnish industry that bitumen does not influence the wear.

Considering the information presented here, in light of our current knowledge of the bitumen behavior at high frequencies and low temperatures, we should investigate if the low phase angle measured from bitumen at those conditions would correlate with increased abrasion at said conditions.

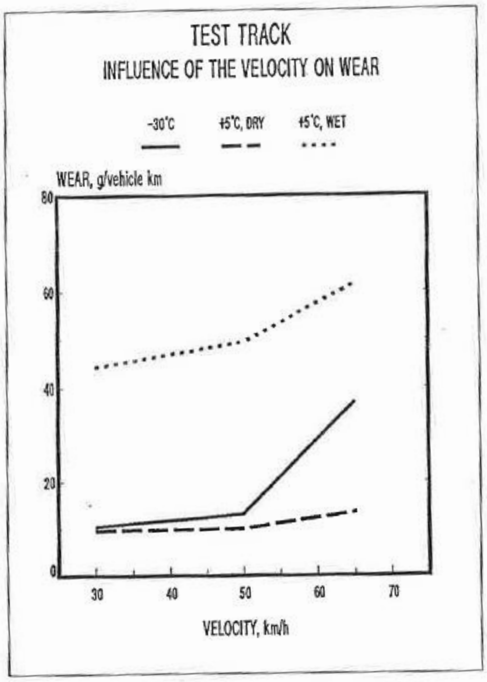


Figure 213. The influence of the velocity on the test track on the wear (Blomberg 1991).

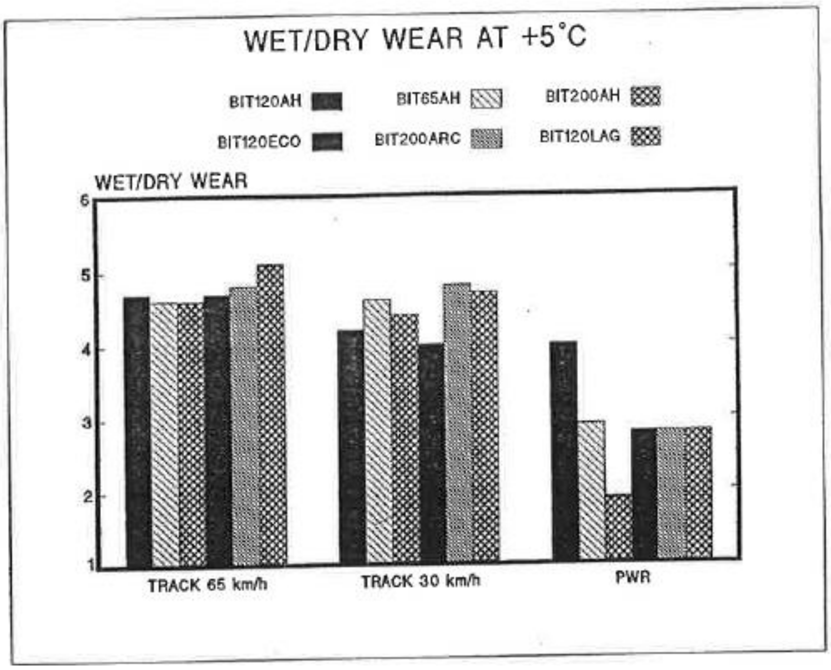


Figure 214. The wear ratio in wet and dry conditions measured at speed of 65 km/h and 30 km/h on the truck, and compared to the results from the Pavement Wear Resistance tester (Blomberg 1991).

15.4.3 Abrasion by Studded tyres (PWR)

The name of the testing device investigated as a testing method during ASTO is known internationally as Pavement Wear Resistance testing device. The equipment and test are specified currently by a standard SFS EN 12697-16. Inside Finland the equipment is referred to as “SRK” due to the abbreviation of the Finnish name “Sivurullakulutuskoe” (side-rolling wearing test).

The test is performed on the core or compacted sample attached from top and bottom. The three wheels with studs are rolling for a fixed number of cycles on the circumference of the sample, while water is applied on the side of the sample as well. The chamber is conditioned during the test to set temperature. The temperature used to establish values necessary to meet requirements in specification is 5 °C.

The speed of the rolling wheels is equal to 5 km/h and it is postulated that the mechanism of failure is mostly abrasion and polishing. The impact of the wheel was postulated unimportant. The frequency of this speed at temperatures above 5 °C applied onto bitumen was postulated to be lower than in real traffic and to test the bitumen in the range in which bitumen is expressing viscous properties rather than the elastic ones.

15.4.3.1 The calculation of the frequency in PWR and traffic

The sample of 100 mm diameter has the circumference equal to:

$2\pi r = 2 * 3{,}14 * 50 * 10 - 3 \text{ m} = 0{,}314 \text{ m}$

If three wheels are moving with the speed of v=5 km/h, that is v=1.388 m/s, then the distance covered by one wheel before the other one arrives in the same spot is equal to x = 0.314 / 3 = 0,1046 m.

The time between the wheels is then calculated from the velocity equation

$V = x/t$

Then $t = x/v = 0{,}1046 / 1{,}388 = 0{,}0754$ s.

In this case the frequency between the wheels is defined by

$f = 1/t = 13{,}2 \text{ 1/s} = 13{,}2 \text{ Hz}$.

So in fact the used explanation of 5 km/h testing the lower frequency is faulty, because the frequency between the wheels is higher than the typically applied 1,59 Hz to predict the influence of the truck moving with the speed of 60–65 km/h, with the distance between axels of around 10 m, on the elastic behavior of the bitumen.

If we consider the frequency of the wheels in the passenger car, of approximately 3 m distance on the wheelbase, the frequency at 65 km/h is approximately 6,02 Hz. Using similar assumptions, a relationship between speed of vehicle and the frequency of load is presented in Figure 215, where it should open up why testing bitumen using frequency sweeps could be useful for prediction of behavior at low and high speeds.

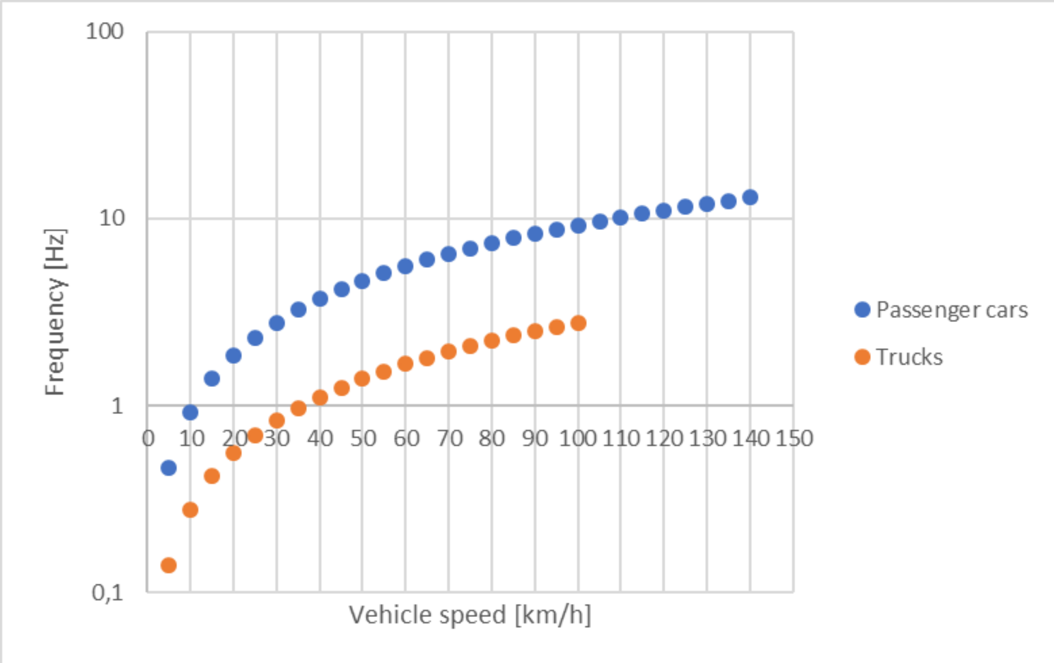


Figure 215. The estimated frequency of load impact between wheels of trucks (10 m) and passenger cars (3 m).

It is postulated hereby that the frequency used in PWR testing device is testing the bitumen at its most elastic state. The effect of speed in PWR was not discussed in ASTO final report. The average speed on the test roads within ASTO are also not provided.

The higher frequency could explain why the results of abrasion from SRK were higher than on the roads/test tracks (Kurki, Halttunen et al. 1993) (picture 2/3.4). The differences in elasticity at low temperature and high frequency could perhaps differentiate between bitumens in terms of their effect on the abrasion. Unfortunately, it is challenging to find any numerical values from the test tracks, as well as a description of the test in the references reports.

15.4.3.2 The influence of frequency on the properties of bitumen

Nevertheless, the explanation of the elastic behavior of the bitumen at higher frequencies and lower temperatures seems to be understandable. The theory implies that the lower the phase angle, the more elastic the behavior of the bitumen. The suggestion is that the dominant elastic behavior starts below the phase angle of 45°. The reference bitumens used in the ASTO project comprised B120 and B65. Comparing it to the data collected using the dynamic shear rheomether during the REMIX project between 2014–2018 using bitumen 70/100 and 160/220, we can see that indeed the temperature and frequency plays a role at the considered temperatures just above and below 0 °C (Figure 216). For example for 160/220 at 10 °C even the 10 Hz frequency applied to tested sample results in phase angle of 57°, while for the 70/100 this value is equal to 41°.

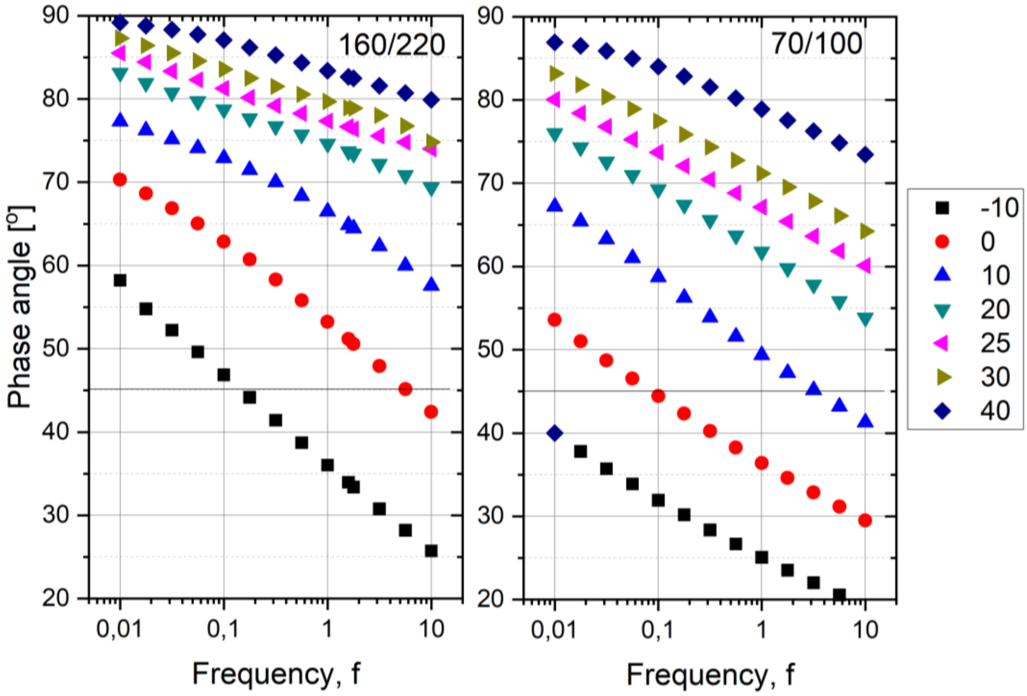


Figure 216. The relationship between temperature, frequency [Hz] and phase angle for bitumen 160/220 and 70/100 tested using dynamic shear rheomether during the REMIX project (LIVI database) (Pellinen & Makowska 2018).

Suggestion is to study which of the frequencies in bitumen actually influences the abrasion by studded tires. Perhaps an information from rheological measurements could provide an insight on the resistance of the binder to the studded tire wear. Combining it with the load of axle, could give us a response in the material.

It seems that 1 Hz is being reached by trucks at 30 km/h and passenger cars at 10 km/h. The frequency used 1.59 Hz is approximately 60 km/h. The 10 Hz is not reachable for trucks, assuming maximum speed of 100 km/h, but it is a reachable frequency for passenger cars travelling with speeds above 100 km/h. During the discussions between the stakeholders it was disclosed that due to the observations of real roads, the speeds during winter are kept below 100 km/h partially to prevent pronounced wear of the pavement, which stays in agreement with the above analysis.

Testing with 4 mm (Figure 217) of the 50/70 and a rather hard PmB 25/55–55 reveals that at very low temperatures (-20…-28 °C) their behavior in response to those applied frequencies is strongly elastic ($\delta < 10°$). If there is a certain threshold of phase angle which correlates with pronounced abrasion, certainly the bitumen PmB 25/55–55 would achieve it at higher temperatures. Similar type of analysis for binders used in Finland could prove useful.

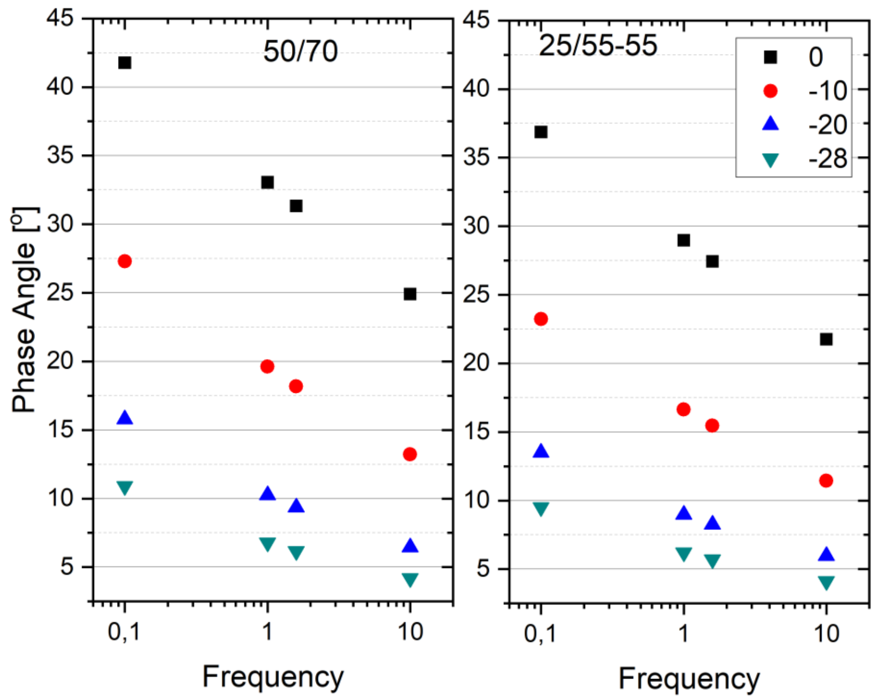


Figure 217. The phase angle in two binders tested in the Round Robin of the 4 mm plate plotted against the frequency [Hz].

15.4.4 Abrasion in the Tröger test

15.4.4.1 Principle

A core sample is cut in half and exposed to the abrasion by metal needles. The needles are moved by the compressed air (Pylkkänen & Kuula-Väisänen 1990). Similar as in Prall and PWR tests, the volume of the sample abraded is measured.

15.4.4.2 ASTO experience

During the project, the two types of aggregates (granite and granodiorite) were tested for the resistance to abrasion in dry, moist and wet conditions. Each of the mixtures had another variable, namely the composition of the filler. When the filler in the mixture came from the aggregate's own fines (reference), or from hydrated lime the difference between dry and wet behavior was minuscular. **However, reference mixtures consistently resulted in lower abrasion values, while the mixtures with hydrated lime expressed highest abrasion.** Interestingly, the use of limestone filler resulted in fairly similar abrasion level in dry conditions to the reference mixture, but in the wet conditions the abrasion for those mixtures was increasing by about 70% (Pylkkänen & Kuula-Väisänen 1990).

The aspect of filler role on the moisture resistance and resistance to abrasion by studded tires in wet environment should be evaluated in more detail.

15.5 The effect of various parameters on the abrasion due to studded tyres

15.5.1 The relation between SRK and Tröger test

The two tests corelate well between each other. Therefore, justified assumption is that the results distinguishing between materials can be similar in both equipment. The correlation between abrasion observed in field was slightly higher for PWR and Nordic Ball Mill abrasion test (Kurki, Halttunen et al. 1993), and for this reason they have been chosen as methods for use in specifications.

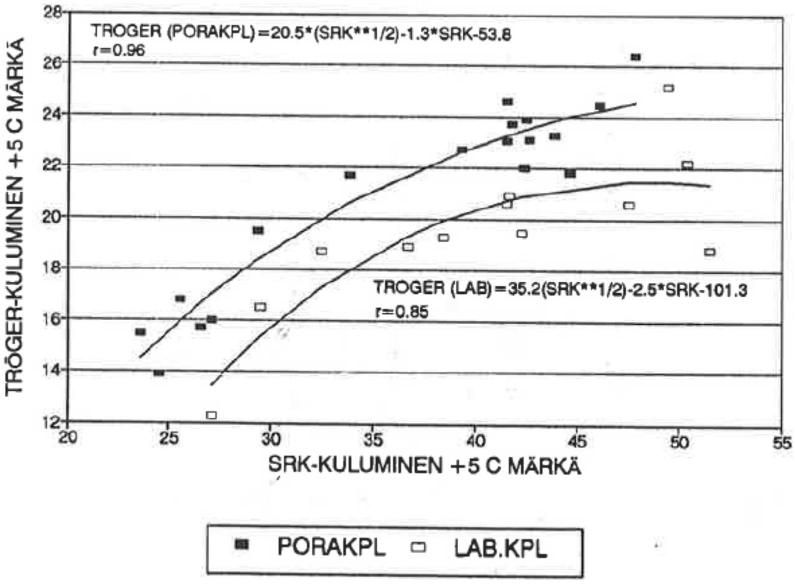


Figure 218. The relationship between PWR (fin. SRK) and Tröger test results from laboratory prepared samples and cores from test roads (Kurki, Halttunen et al. 1993)

15.5.2 The effect of filler on the abrasion

Apparently, the hydrated lime was reducing the resistance to the studded tires by about 6%. Therefore, using it as an adhesion promotor, we may cause the lower resistance to studded tires. The aggregate fines were also reducing the resistance to abrasion of the mixtures. Mixing carbon black, limestone and asphalt fines resulted in the mixture least resistant to abrasion (Kurki, Halttunen et al. 1993). As can be seen on the figures reprinted from the study (Figure 219 and Figure 220), the resistance to abrasion is higher in the mixtures with lower strength and marshal stability (Pylkkänen 1991). The addition of hydrated lime improves resistance to moisture, but decreases strength and resistance to abrasion (Kurki, Halttunen et al. 1993). The observation of stiffness of binder or its elasticity having an influence on pronounced abrasion, would be consistent with the changes in mastic influencing higher softening points, causing higher strength of mixture and leading to more brittle response during PWR.

Perhaps optimization of the speed of the wheels on the sample to represent conditions in field would actually help in predicting the true behavior. Currently the sample is exposed to the worst plausible conditions in order to inflict the most damage.

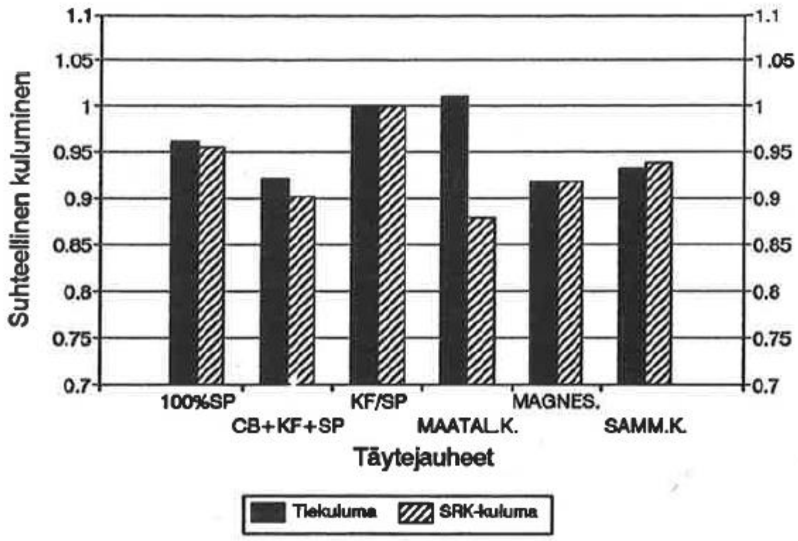


Figure 219. The influence of filler on abrassion by studded tyres as witnessed in field (tiekuluma) and by PWR test (SRK-kuluma). Samm.K – hydrated lime, KF/SP – limestone and aggregate fines, SP – baghouse fines, bitumen not specified (Kurki, Halttunen et al. 1993).

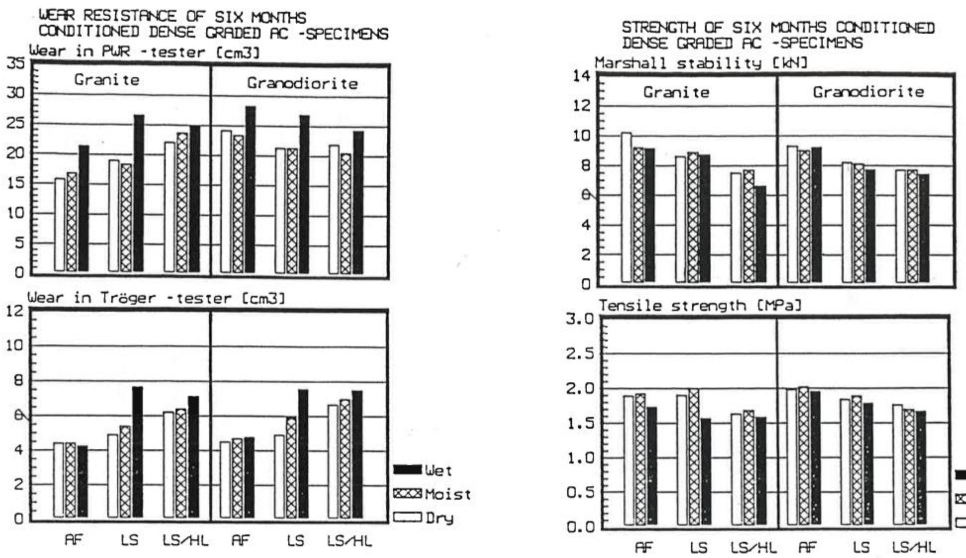


Figure 220. The wear resistance of long-term conditioned specimens (left side) alongside their Marshall strength and stability (right frames), where AF= aggregate fines, LS=limestone and LS/HL is limestone/hydrated lime (Pylkkänen 1991).

15.5.3 The effect of the binder on abrassion

It was noticed that the AB20 mixture had the best resistance to the wear by studded tires. This was a dense graded mixture. Two gap graded mixtures were compared to it, namely ABE20 (Asfalttibetoni epäjatkuva) and SMA16. The ABE20 mixture was tested using varying binders, where B80 LAG and B80AH were used as a reference.

Using 200AH would increase the abrasion by approximately 15%, and using the PmB3, would decrease it by about 15% as well. It is logical that the use of softer binder increases the rutting due to studded tires, thus comparing B80 to B120 and B200 we see a growing trend. The effect of the choice of the binder grade can result in **approximately 25% difference in abrasion.**

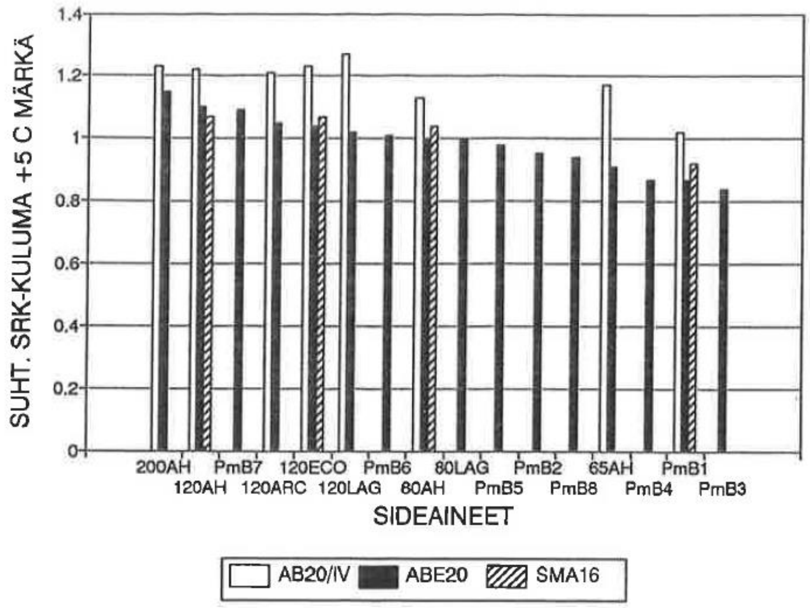


Figure 221. The effect of binder type on the abrasion by studded tires in PWR at +5 °C and in presence of moisture for different types of asphalt mixtures.

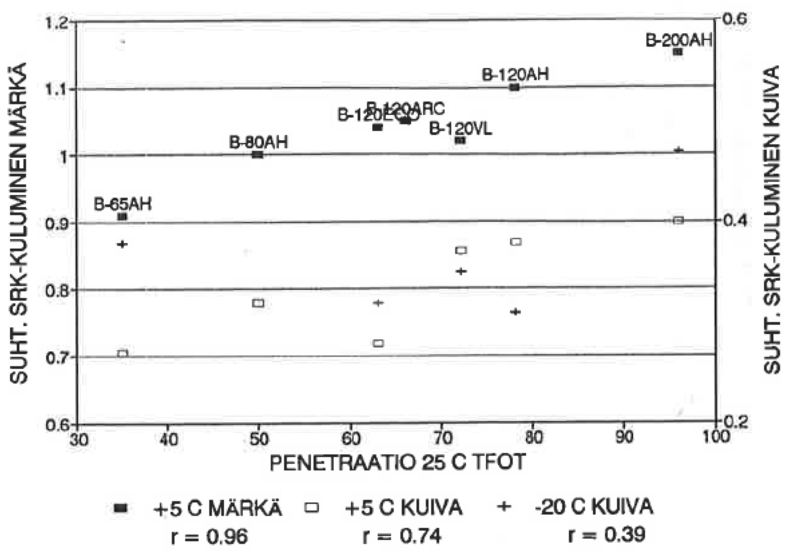


Figure 222. The relationship of Penetration of bitumen source AH and abrassion in PWR (Kurki, Halttunen et al. 1993).

When the wear was measured at low temperatures, that is -20 °C the wear of B65AH sample increased significantly and was higher than the wear in the B120AH and B200AH measured at that temperature. This was explained by the B65AH being below its Fraass breaking point, i.e. in the temperature region of a fully elastic response of bitumen. This binder was postulated to be simply expressing brittle behavior at that temperature.

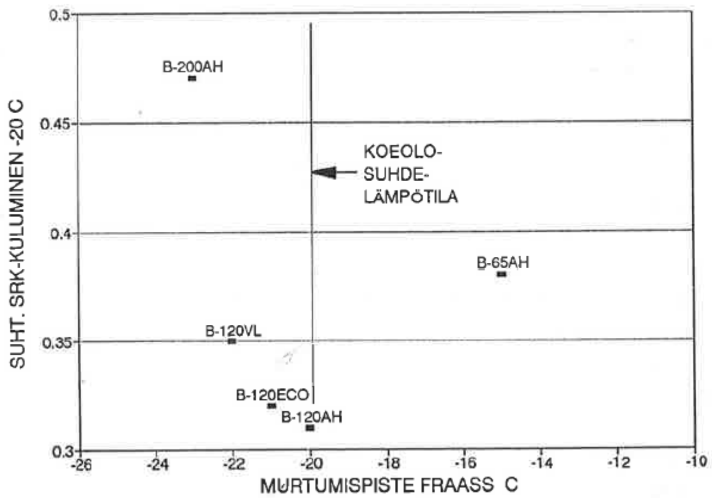


Figure 223. The PWR abrassion value measured at -20 °C plotted against the Fraass breaking point of corresponding bitumens (Kurki, Halttunen et al. 1993).

With PmB the analysis was not that simple. The 7 used binders apparently behaved differently, and in this case the result depended on the product used. Even using ductility at -10 to compare the PmB binders no clear answer as to the effect on the abrasion was achieved. Since then, the description of ductile behavior can be linked to a certain phase angle and complex modulus values (P. Kandhal 1977; Anderson et al. 2011; Rowe 2011), which also indicates that there could be a link between viscoelastic behavior of binders and their resistance to studded tyre wear.

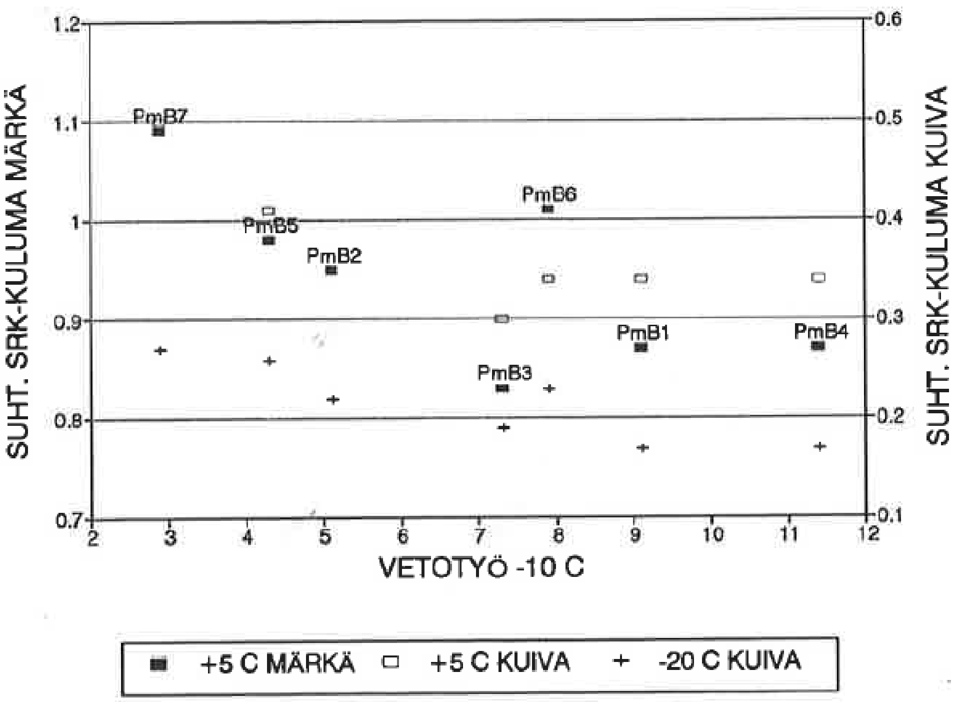


Figure 224. The relationship between ductility at -10 °C of PmB and wear in PWR (Kurki, Halttunen et al. 1993).

It was concluded that the effect of binder is very small. However, if we consider comparing the effect to 32–33 ml of abraded material in PWR (applicable for class II), the 15% lowered abrasion would result in material of abrasion on the level of 28 ml (applicable for class I). As presented in Figure 225, bitumen type has an effect on the abrasion both in wet conditions at 5 °C, as well as in the dry conditions at -20 °C. The use of PmB could result in the pavements more resistant to abrasion by studded tyres as witnessed by the PWR test results (Kurki, Halttunen et al. 1993). Again, it seems that stiffer grades like B65 and B80 had higher abrasion at low temperatures than the softer grades (B120 and PmBs created on their base). On the other hand, the observation of changes in field were not as apparent, therefore no clear suggestion was formed to prefer this type of binder over the non-modified one.

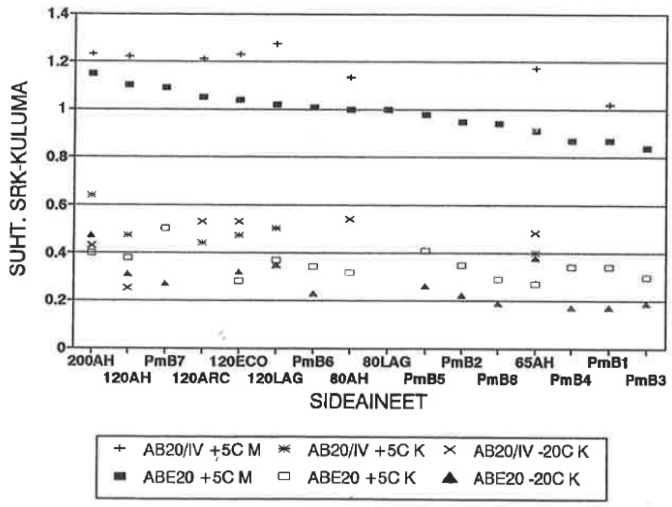


Figure 225. The relative abrasion of asphalt pavement samples at various temperature conditions in PWR test device.

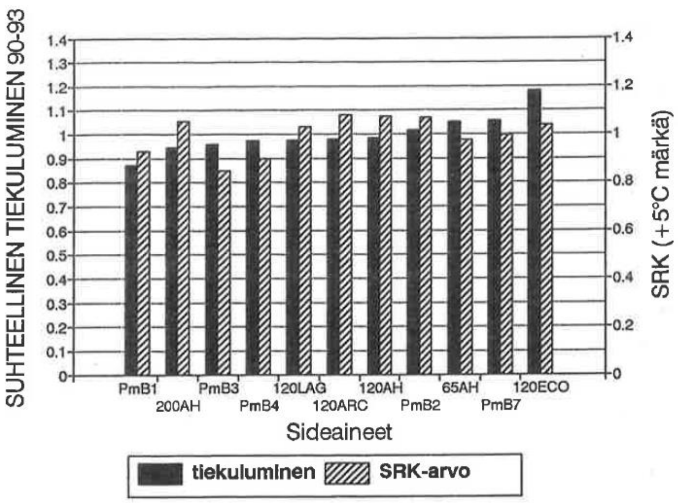


Figure 226. The result of the analysis of abrasion in field (tiekuluminen) and using PWR (SRK-arvo) for the various binders (Kurki, Halttunen et al. 1993).

The expression of results for bitumens and aggregates is somewhat misleading. The aggregates are plotted in the absolute values of abrasion, that is ml. While the bitumen analysis is presented in form of relation to reference. If we consider that the 1 stands for 35 ml, then 0,8 represents 28 ml, and 1,1 represents 38,5 ml. The difference is of a one wear category according to the FAS (Finnish Pavement Technology Advisory Council 2017).

Another aspect worth pondering about, is the susceptibility to moisture damage of the mixture and its effect on the wear. As presented in the reprinted Figure 227, the ratio of wear recorded in dry and moist conditions is differing between approximately 0,25–0,45 for different binders. It would be fair to assume that abrasion in mixtures with binders characterized by lower ratio are more susceptible to the effect of moisture on the abrasion. The analysis of the numerical results (Table 29) from the PWR would indicate that the same asphalt mixture AB20/IV with Teisko granodiorite could be classified as Abr_{37} or Abr_{55}, depending on the binder used. It is therefore not apparent why the conclusion that the binder has no effect on abrasion was made.

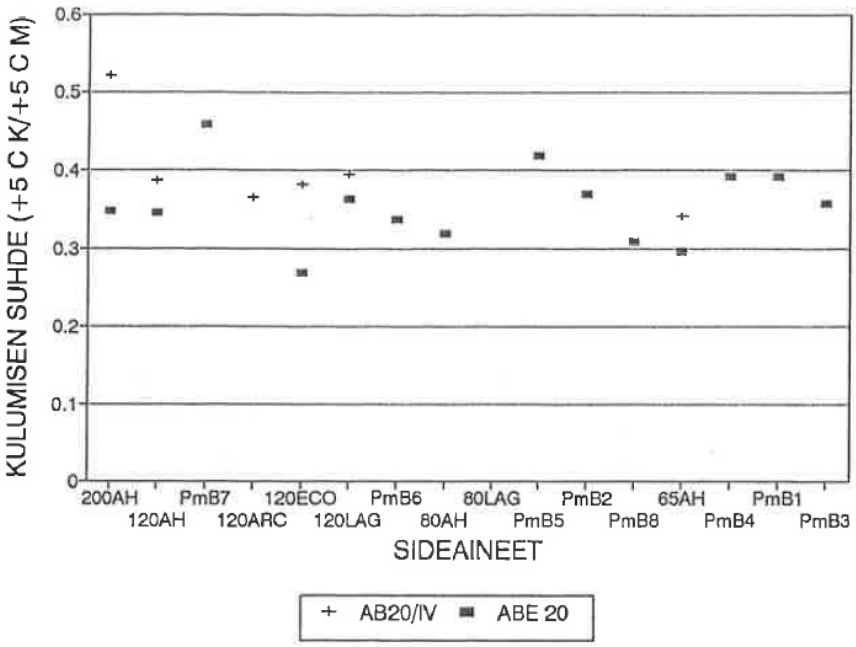


Figure 227. The ratio of wear obtained by PWR device without moisture and with moisture in the system at 5 °C performed on the reference mixtures (granodiorite aggregate) using various bitumen samples as binders.

The results obtained by Prall test were discussed as not reliable for polymer modified binders and it was proposed that the metal balls in Prall testing device are potentially bouncing off the surface of asphalt with PmBs. Therefore, the PWR tester was suggested for the use when testing the PmB containing asphalt mixtures. However, since ASTO, other researchers have reported similar observations indicating that the use of polymer modified binders is a potential for reduction of wear of pavements (Jackobson 1997).

Because of the above provided analysis of the results from ASTO, it would be worthwhile to challenge the statement prevailing in the industry that "the influence of the binder properties on asphalt wear is very small" (Blomberg 1991). The suggestion is to look back into the relationship between elasticity of the binder at traffic frequencies and to see if the link with abrasion is observed. The non-modified binder 80 AH seems to contribute to 3 times more abrasion than PmB at -20 °C in dry and frozen conditions. The current advances in bitumen testing using Dynamic Shear Rheometer allow us to perform the frequency sweeps at temperatures ranging from -30 °C to 120 °C if needed. Such possibility was not available at the time of ASTO program in Finland.

Table 29. Chosen numerical result of the PWR test performed on the reference mixture ABIV with Teisko Granodiorite and different binders at similar binder content, as well as investigating the influence of under and overfill of bitumen on abrasion (Kurki, Halttunen et al. 1993).

Type of mixture	Binder type	P_B	Wet wear 5 °C	STD	Dry wear 5 °C	STD
AB20/IV with aggregate 8	120AH	5,9	51,5	3,5	13,0	1,6
AB20/IV with aggregate 8	120A	5,9	48,2	5,7	16,7	2,0
AB20/IV with aggregate 8	120VL	5,9	49,9	3,4	17,9	0,8
AB20/IV with aggregate 8	120AA	5,9	43,0	0,7	15,5	0,9
AB20/IV with aggregate 8	65AH	6,1	41,5	3,2	14,2	0,9
AB20/IV with aggregate 8	200AH	5,8	45,3	1,7	22,9	0,8
AB20/IV with aggregate 8	120AH	6,7	53,0	3,8	12,8	1,0
AB20/IV with aggregate 8	120AH	5,5	47,0	1,3	16,1	1,5
AB20/IV with aggregate 8	PMB1	5,9	35.9	0,6	1,2	2,7

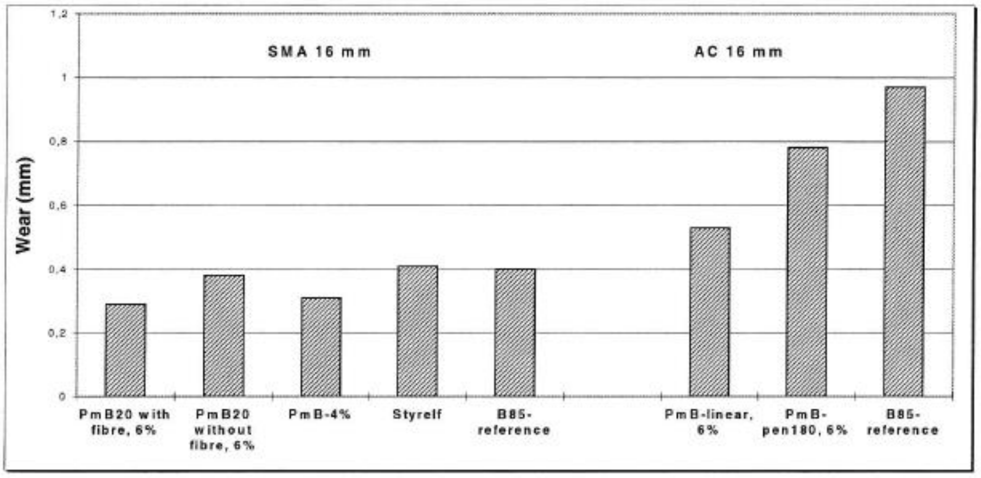


Figure 228. The influence of the modified binders on the wear resistance in road tests using quartzite aggregate (Jackobson 1997).

15.5.4 The follow-up of the ASTO test sections

In 1998 a review of the rutting progress since ASTO project was summarized (Kurki 1998). The 14 test roads were analyzed to investigate the effect of various parameters, of which the most crucial for hereby report are presented in this section.

The follow-up method comprised of the transverse profile measurements during the spring and during the autumn. The assumption proposed was that the hindered recovery (creep) would be observable between spring and autumn, whereas the effect coming from the studded tyre abrasion would be observable between the measurements in autumn and spring. The assumption for the winter season and its lack of effect to permanent deformation was in the increased resistance to the permanent deformation of the binder at lower temperatures.

It was found that the SMA type mixtures were resisting rutting due to studded tyre abrasion better than AB20 mixtures. The larger the D of the SMA the higher the abrasive resistance of the mixture. This has further supported the choices for the mixtures of the wearing courses in Finland and stays in agreement with research reported in Sweden (Kurki 1998; Jackobson 1997).

The comparison between the dry weather conditions of the eastern Finland with the western Finland and the high rain precipitation in that region indicated that the effect of the moisture may add up to 30%. In combination with the decreased abrasion on roads with adhesion promotors, Kurki (1998) concluded that the adhesion between binder and aggregate and resistance to moisture must be playing a role in the resistance to studded tyre abrasion. The suggestion was to expand the study of the subject.

Recently, already 22 years after this initial attempt at modelling the progression of the rutting speeds in Finland, during the works focused on development of the rutting predictive models, Dettenborn et al. (2020) suggested that the effect of the weather and binder rheology should be included in the future predictive models as well. Currently, data related to the binder type and origin is not as available as for example the transverse profile measurements for any road sections in Finland.

15.5.4.1 The effect of adhesion promotors

One of the test sections (ASTO 10) allowed for comparison of the mixtures with and without adhesion promotor (Raisamine, polyamine), using two binders – LAG and ECO (Russian economical), as well as two types of aggregates – diabase (code 15 in ASTO) and vulcanite (code 35 in ASTO). The mixtures were following the AB20/IV mixture design described in the report (Kurki, Halttunen et al. 1993).

The ASTO 9 test section used Teisko granodiorite, bitumen from the Arabian Heavy crude oil source and the diamine as the adhesion promotor.

The results of the resistance to studded tyre wear, as well as to deformation are presented in Figure 229 and Figure 230. Kurki (1998) concluded that the use of the adhesion promoting additives decreases the studded tyre abrasion on the level of 14%, but on the other hand increases susceptibility to permanent deformation by up to 30%.

Knowing that, one should retain caution during the mixture design. If the addition of amines decreases the viscosity of the binder, the permanent deformation may increase. The optimal approach would be to use the adhesion promotor but retain the temperature susceptibility of the binder and for that an optimization is suggested.

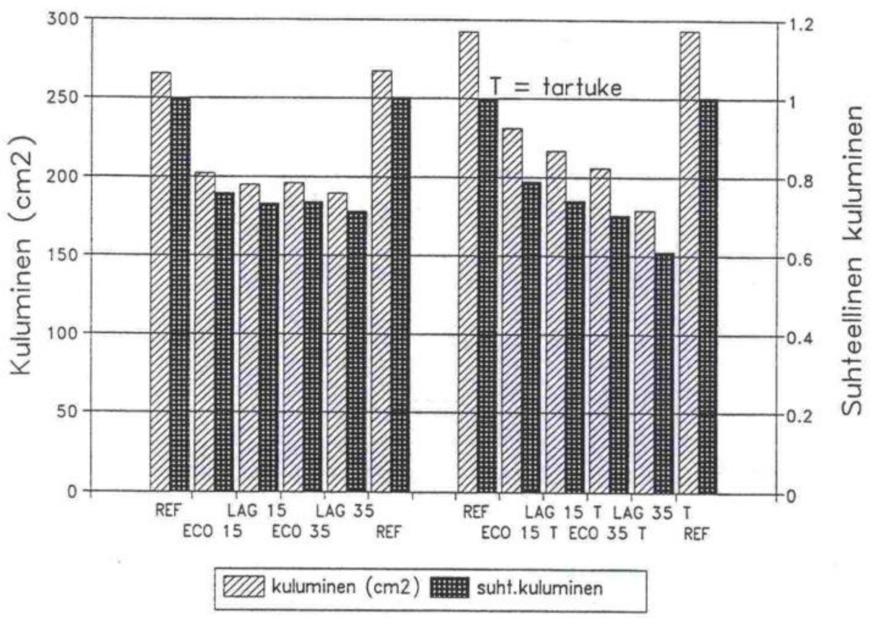


Figure 229. The abrasion caused by studded tyres – based on the analysis of the road VT5, test section ASTO 10, with the adhesion promotor – Raisamin, where in the AB20/IV mixtures the following raw materials were used: LAG – laguna binder, ECO – the Russian economical binder, 15 – diabase ASTO aggregate code 15 and 35 – vulcanite ASTO aggregate code 35.

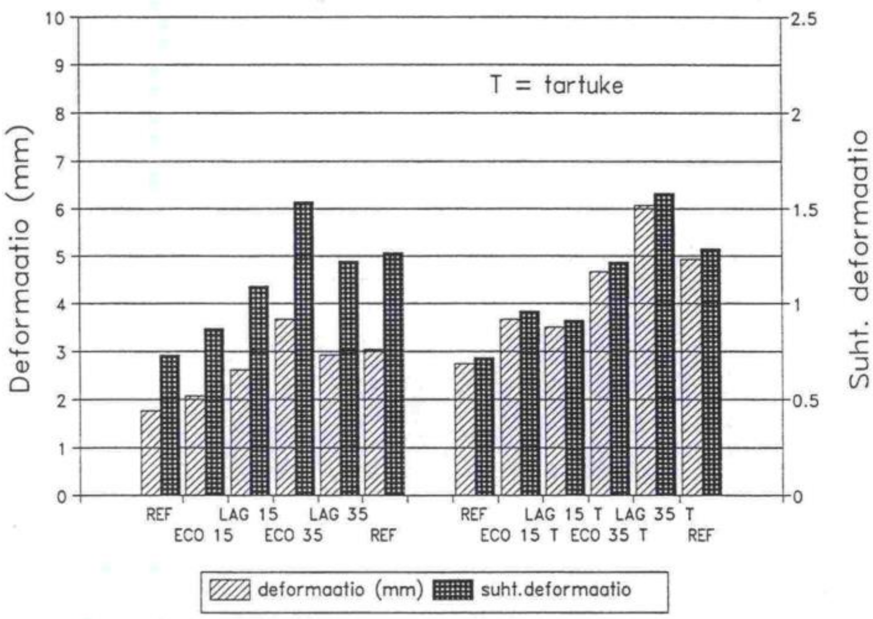


Figure 230. The effect of the adhesion promotors on the permanent deformation – based on the analysis of the road VT5, test section ASTO 10, with the adhesion promotor – Raisamin, where in the AB20/IV mixtures the following raw materials were used: LAG – laguna binder, ECO – the Russian economical binder, 15 – diabase ASTO aggregate code 15 and 35 – vulcanite ASTO aggregate code 35.

15.5.4.2 The binder effect on the abrasion

When the unmodified bitumen was used, the differences between deformation in AB20 and SMA16 were in the range of 24%. When the PmB1 was used, the differences between the permanent deformation of the mixtures decreased and were on the level of 7% difference, yet the PmB binders in general were characterized by higher permanent deformation in field and higher wear (Kurki 1998).

Kurki (1998) concluded that the quality of binders seems to affect the wearing, but the data set was too small to find suitable quality properties of the binder to correlate with performance. Nevertheless, the PmB1 improves wearing by 10%, while decreasing deformation by 30% in comparison with B-120 type binders.

The differences between the binders were small, but the 120-ECO (Russian economical) was performing worse of them all, on average 7% worse than the reference mixture. Author expressed a concern that since the ASTO the source of the binder stopped being reported, and further analysis of the effect of the binder source will be troublesome.

To truly understand the relationship between binder properties, permanent deformation and abrasion by studded tyres, it is suggested to start following the properties of the binders used on the pavements constructed. If not all of them, at least few construction sites per year should be chosen randomly for characterization of the raw materials to advance the database creation for the future generations.

16 The upcoming test methods

16.1 The use of other existing standard methods

16.1.1 Cantabro test

In order to evaluate the susceptibility for raveling, the test applied for porous asphalt could be investigated. The so-called Cantabro test provides the researchers with the value of the percent particles loss from the prepared asphalt mixture (12697-17:2017 Particle loss of porous asphalt specimens).

The test principle is simple and uses equipment available in the laboratories performing Loss Angeles abrasion tests. The sample of fixed size is placed into the Loss Angeles abrasion machine without the steel balls, and rolled for a fixed number of cycles. The mass before and after are measured, and a particle loss is evaluated based on those mass measurements.

The ITSR test, provides us with the relative value of dry to wet strength, but not with the numerical value of the strength measured from standardized sample. The Cantabro test result seems to be increasing, with the decreasing Indirect Tensile Strength of the specimen (Pasquini, Giacomello et al. 2020). The weaker the material the more likely it is to crumble during the Cantabro test. Moisture conditioning of samples prior to the Cantabro test provides a possibility to investigate the increase in the particle loss due to the exposure to water.

The AC and SMA mixtures with unmodified and modified bitumen were studied using the ITSR as well as Cantabro with moisture conditioning (Pasquini, Canestrari & Santagata 1997). In Table 30 an example of the results for the ITSR and Cantabro test is provided, demonstrating how the ITSR value (percent) may not in fact differentiate between the two samples resistance to raveling or loss of particles (Pasquini, Canestrari & Santagata 1997). Similar relationship was provided for asphalt concrete produced using slag aggregates (Pasquini, Giacomello, et al. 2020).

Table 30. Results from the study by Pasquini et al. (1997) for gap graded asphalt concrete with crumb rubber modified bitumen (ARAC) and typical dense graded asphalt concrete mixture (DGAC), comparing ITSR and particle loss (PL) in Cantabro test.

Mixture	ITS dry		ITS wet		ITSR [%]	Cantabro dry		Cantabro wet		Increase in PL due to moisture [%]
	V_a [%]	ITS (kPa)	V_a [%]	ITS (kPa)		V_a [%]	PL%	V_a [%]	PL%	
ARAC	5,9	993	5,7	957	96,4	6,6	1,8	6,7	2,1	13,5
DGAC	5,7	804	5,7	794	98,7	5,7	10,2	5,7	13,3	30,9

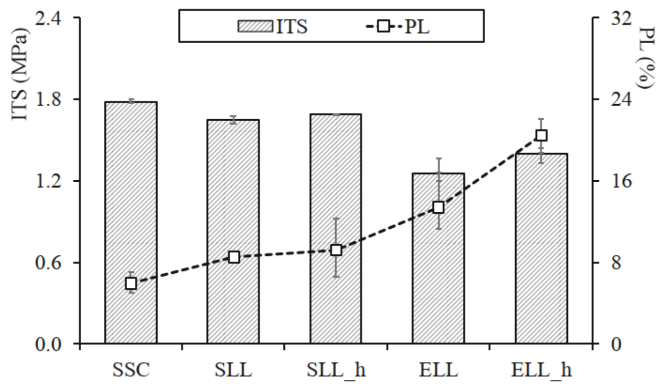


Figure 231. Basic mechanical properties of mixtures composed using slag where ITS stands for Inderect Tensile Strength, PL for particle loss, and in the sample names L stands for ladle furnace slag, E for electric furnace slag and S for silice (First letter in sample name stands for coarse aggregate type second for fine aggregate and third letter for filler material) (Pasquini, Giacomello et al. 2020).

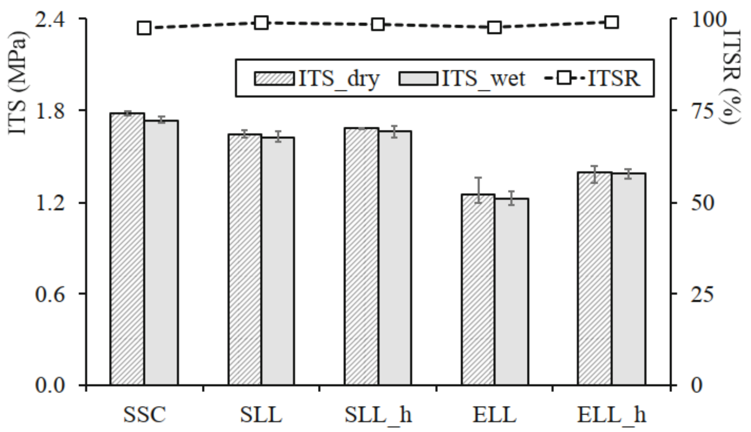


Figure 232. Water resistance (ITSR) of the tested mixtures using slag, in the sample names L stands for ladle furnace slag, E for electric furnace slag and S for silice (First letter in sample name stands for coarse aggregate type second for fine aggregate and third letter for filler material) (Pasquini, Giacomello et al. 2020)

In China (Li & Li 2016) the resistance of AC mixtures to moisture damage with increasing RAP content was evaluated with ITSR and Cantabro, and as well it was found that ITSR did not allow for a good discrimination between the mixtures in terms of resistance to moisture. Using the Cantabro test, the authors were able to conclude that with RAP content changing between 0 and 60%, the optimal (minimal) loss of particles is achieved in mixtures containing 30% RAP.

Alvarez et al. (Alvarez et al. 2008) suggest that at least for the porous asphalt, the results from the Cantabro test correlate well with air voids accessible to water. It was suggested as a simple and quick test for screening and selecting material combinations. Anastasio (S. Anastasio 2015) investigated the use of Cantabro test after the freeze-thawing cycles and observed that with increased water absorption into aggregate, the increased number of microcracks was present in the aggregate and that resulted in higher particle loss as witnessed with Cantabro test.

16.1.2 The Resistance to Scuffing

The resistance to scuffing or raveling is defined as the loss of particles due to the action of tires. The test methods standardized for the evaluation of raveling propensity are described in SFS-EN 12697-50.

The principle of the test is exposure of the slab of asphalt concrete to the action of normal load and shear load. This is to simulate the action of tyre moving forward as well as during turning of the car.

16.1.3 Peel test

The peel test under 90° angle is a test used for the characterization of adhesives. It has been adapted for the purpose of testing bitumen by Blackman et al. (2013). The lower plate is prepared from the aggregate on which a layer of binder is applied. The aluminium alloy peel arm is then positioned on top of the binder layer. The tensile pull is applied to the arm of the alloy and a peel force is being recorded.

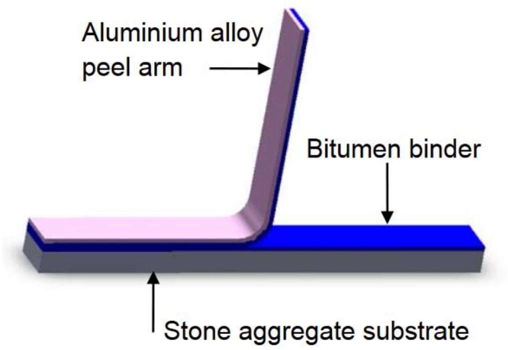


Figure 233 Schematic diagram of a 90° peel test (Blackman, et al. 2013)

The analysis of samples in the one combination of aggregate-binder after conditioning in water baths and without conditioning suggests only 11% of the strength retains after the conditioning. It was postulated that the test has a potential to predict the likelihood of susceptibility to moisture damage in considered materials.

16.2 Friction After Polishing (FAP)

The equipment used for the evaluation of the Friction after polishing (FAP) was postulated to provide the information about the adhesion between bitumen and aggregate in the environment of moisture. The method is currently standardized and described in SFS-EN 12697-49 (Blazejowski, Wojcik-Wisniewska & Baranowska 2018).

The principle of the method is a measurement performed in two chambers. In the first chamber a set of three rubber heads is polishing the sample of asphalt while the water and silicon oxide powder is continuously supplied to the system. This is expected to simulate the effect of tyres on the surface. In the second chamber, the friction coefficient is determined during the breaking of the disc with three rubber sliders. The disc is sped to the 100 km/h. Before the disc touches the surface the water of the temperature 12 °C at 20 l/min is applied. After the disc is lowered onto the sample, the friction coefficient is measured when the disc speed lowers to 60 km/h (Blazejowski, Wojcik-Wisniewska & Baranowska 2018).

The results indicate that highly polymer modified bitumen resulted in lower friction coefficients. Analysis of the results under the microscope revealed that the highly modified bitumen had much better adhesion to the grains and was simply not polished off during the rubber head polishing stage. It was postulated that this indicates a possibility to study the adhesion between binders and aggregates using the FAP method (Blazejowski, Wojcik-Wisniewska & Baranowska 2018).

Based on this information, it can be postulated that the measurements of the evolving friction in field could be indicative of the progress of stripping during the initial phase of exploitation. In Finland the excessive polishing will occur during the winter season, after the change to the winter tyres. However, the monitoring in the initial few months could be a non-destructive test to follow the pavement performance in terms of progress of the initial moisture damage.

17 The methods not standardized in EU

17.1.1 Pneumatic Adhesion Tensile Testing Instrument test (PATTI®-test)

The Pneumatic Adhesion Tensile Testing Instrument test is evaluating the pull off force required for detaching binder from the aggregate. The test was developed as a simple method plausible for adaptation in laboratories (Santagata, et al. 2009).

The principle of the test is presented in the Figure 234. On a polished surface of aggregate the pull-stub is attached by means of a bitumen film. The binder outside of the pull-stub diameter is removed by means of a heated knife (similar as trimming during DSR measurements). Then the structure of the tester is attached to the pull-stub. The pneumatic system lifting the lid connected to the pull-stub causes under pressure, which keeps the system on the aggregate surface by suction. The pressure of air necessary to detach the pull-stub from aggregate is recorded digitally and recalculated into tensile force necessary for failure of bond between bitumen and aggregate (Zhang et al. 2015; S. Anastasio 2015).

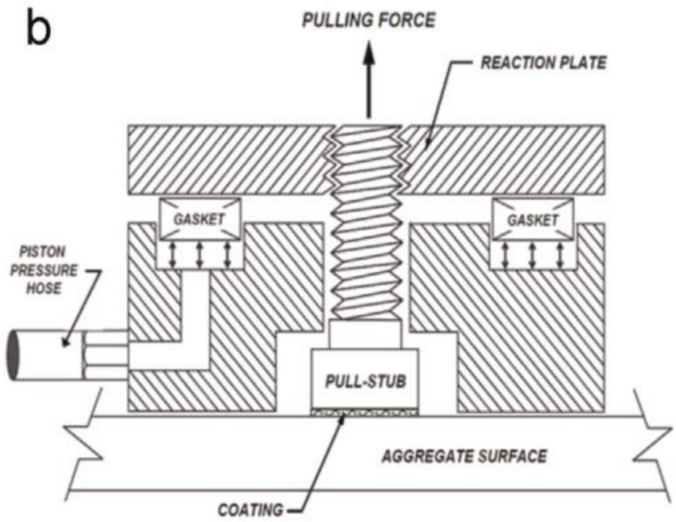


Figure 234. The schematic of the test set-up for PATTI-test (Zhang et al. 2015).

The PATTI® test (ASHTO TP 91) was evaluated by RILEM community (International Union of Laboratories and Experts in Construction Materials, Systems and Structures) in Round Robin. The recommendation of the community underlined that the test classified the aggregates differently than any other evaluated method (Rolling Bottle Test, Boiling Water test, and contact angle measurements), therefore the caution is advised. For that reason, there was no recommendation in regards of PATTI®. It was underlined, that this test is performed using the aggregate in form of a large bulk piece, unlike in other tests. For the moisture to reach to the interface between bitumen and aggregate, the diffusion of water through the rock mass is necessary. As a result, the diffusion affecting parameters probably play a role in the test and attention should be placed on establishing them (Porot, Soenen et al. 2018).

17.1.2 Pull-off test

The principle in pull-off test is the same as in PATTI test. Essentially the tensile force required to break the bond between two aggregate plates, glued with the binder, is measured (Zhang et al. 2015). However, in this case the measurement can be conducted in special dedicated equipment, or in the Dynamic Shear Rheometer (DSR) equipment adapted for the purpose (Figure 235). The presence of DSR in many asphalt laboratories reduces the threshold of the uptake of the method into use.

Additional bonus from using DSR for testing of the tensile force, is the ability to control the thickness of the binder layer. This is not as easily achievable during sample preparation in PATTI test. The use of a conditioning chamber in combination with DSR allows us to condition the sample as is required and to test the system at multiple temperatures, e.g. lower than room temperature. This is not achievable in case of PATTI test.

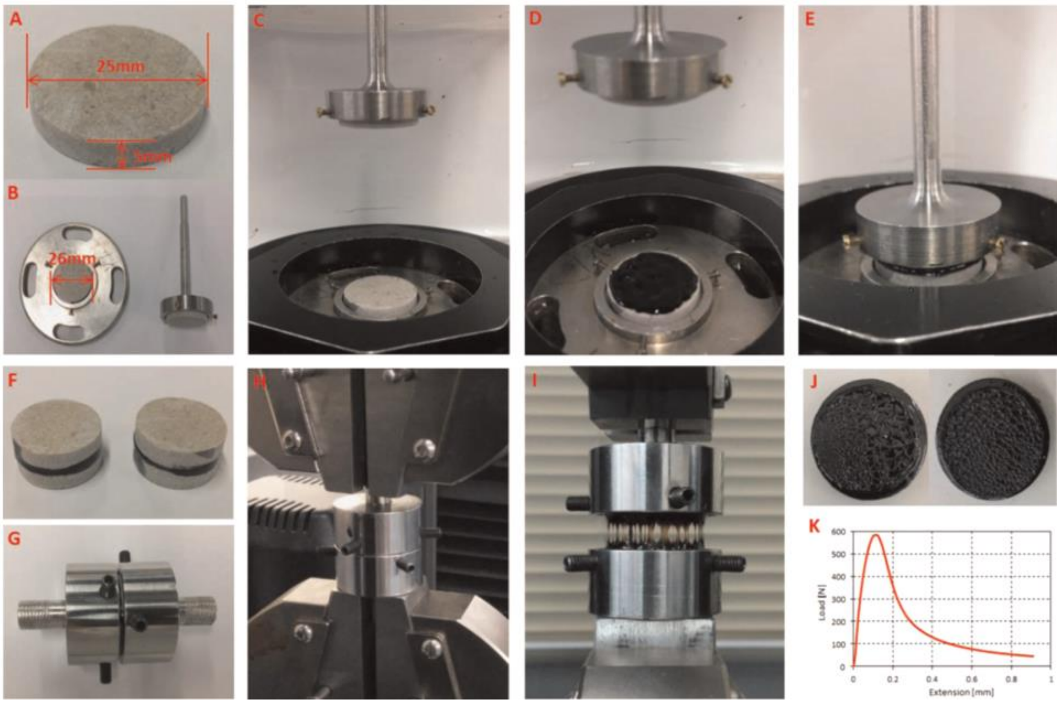


Figure 235. Different Pull-off tests, where set up presented in A–E is a modification of the DSR equipment (Zhang et al. 2015).

Unfortunately, both peel-off test, pull-off test and PATTI test result in a cohesive failure rather than adhesive one, regardless if the sample was dry or wet (Figure 236) (Zhang et al. 2015; S. Anastasio 2015).

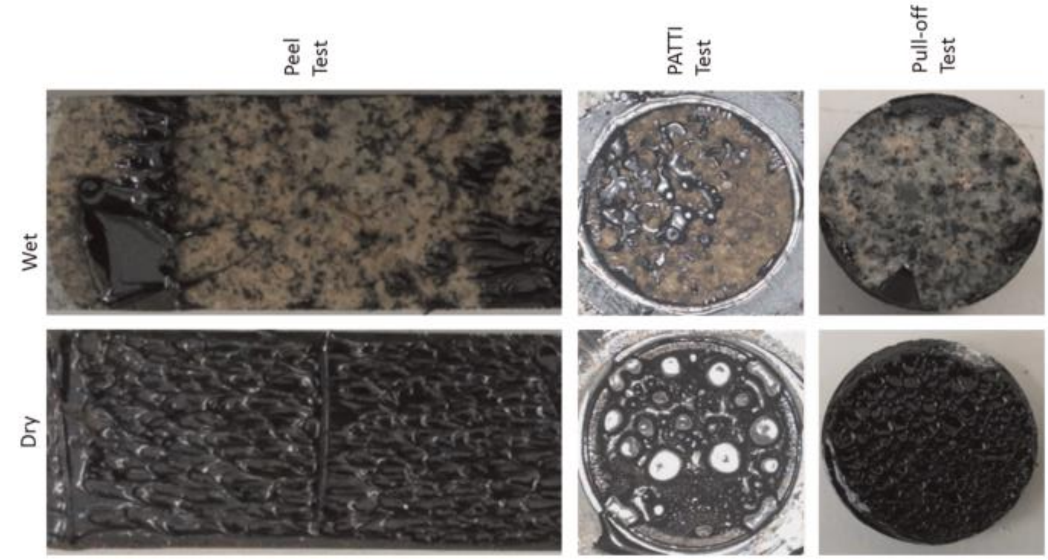


Figure 236. The structure of the aggregate surface after the peel test, pull-off test and PATTI test when tested in dry and after moisture conditioning (Zhang et al. 2015).

17.1.3 Diffusion of moisture through mastic

The effect of the mastic composition on the uptake of moisture into the mastic was studied by Kringos et al. (Kringos, et al. 2011). In the initially optimized sample geometry four types of mastics were investigated in terms of moisture uptake over the course of time (see Table 31 and Figure 237).

Table 31. Composition of studied mastic specimens (Kringos et al. 2011)

Component	Mastic 1	Mastic 2	Mastic 3	Mastic 4
Bitumen 70/100	25% wt.	25% wt.		
Cariphalte XS			25% wt.	
Sealoflex 5-50(PA)				25% wt.
Lime	25% wt.			
Hydrated Lime		25% wt.	25% wt.	25% wt.
Norwegian crushed sand	50% wt.	50% wt.	50% wt.	50% wt.

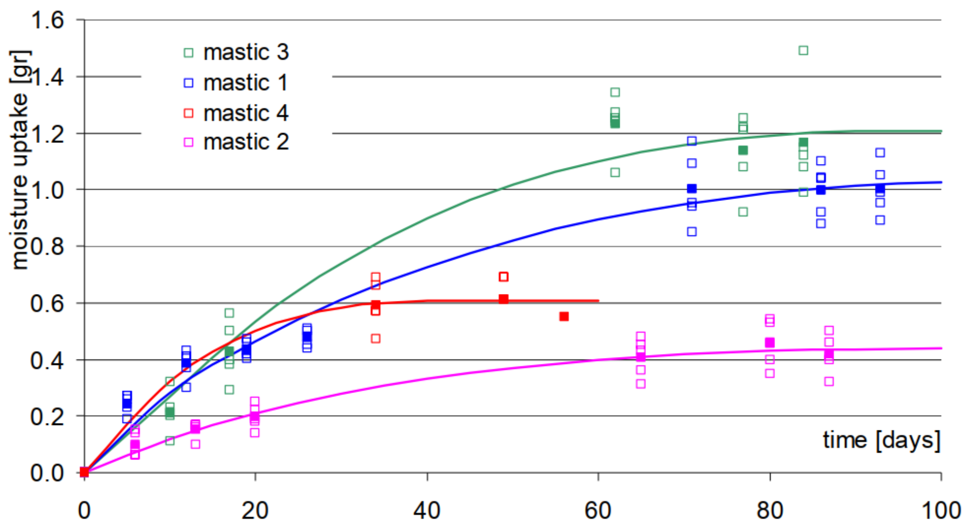


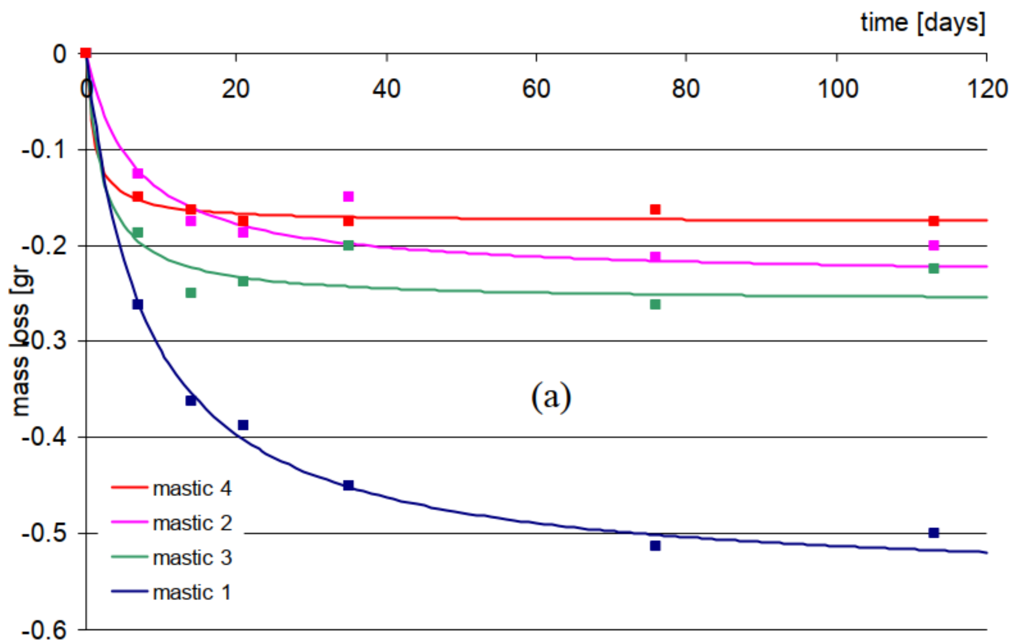
Figure 237. The time dependence of moisture uptake by each studied mastic (Kringos et al. 2011)

This type of test demonstrated, that the effect to freeze-thawing behavior may be coming from the composition of mastic. If the water trapped in the mastic freezes, the material is more likely to develop microcracks. The more trapped unbound water, the more likely the effect is stronger. Of course, in this case, we do not know if the water is bound or unbound in mineral fraction, and no evaluation of the repeatability is provided.

However, one should consider that the moisture transfer through the asphalt concrete may be related to properties of bitumen and filler, i.e. mastic. Interestingly, the effect of binder on the uptake of the moisture into the mastic is underlined (Figure 237). Additionally, the research comprised of the study of drying of the mastic (Figure 238), from which it is indicated that the diffusion of moisture through mastic during drying as well depends on the composition of binder as well.

It should be postulated that the resistance to the freeze-thawing of the asphalt concrete should be best achieved in the asphalt concrete with the mastic composition assuring the best hydrophobicity. The uptake of the water into the structure should be minimal by the material itself, be it binder or filler or other aggregate fraction. At the same time the drying rate of the water trapped in the structure should be high. As was discussed in the review related to the effect of air voids, the permeability of the water through the structure may play a role in this respect. The aggregate gradation affects the compactibility and distribution of air voids. Too fine of air voids inflicted by bad design of mastic composition (e.g. micropores) may lead to the capillary suction, and as a result in low drying rates.

The suggestion would be to focus on the conscious material choice and optimal mix design, considering the hydrophobicity of the mastic, drainage and permeability of the asphalt concrete.



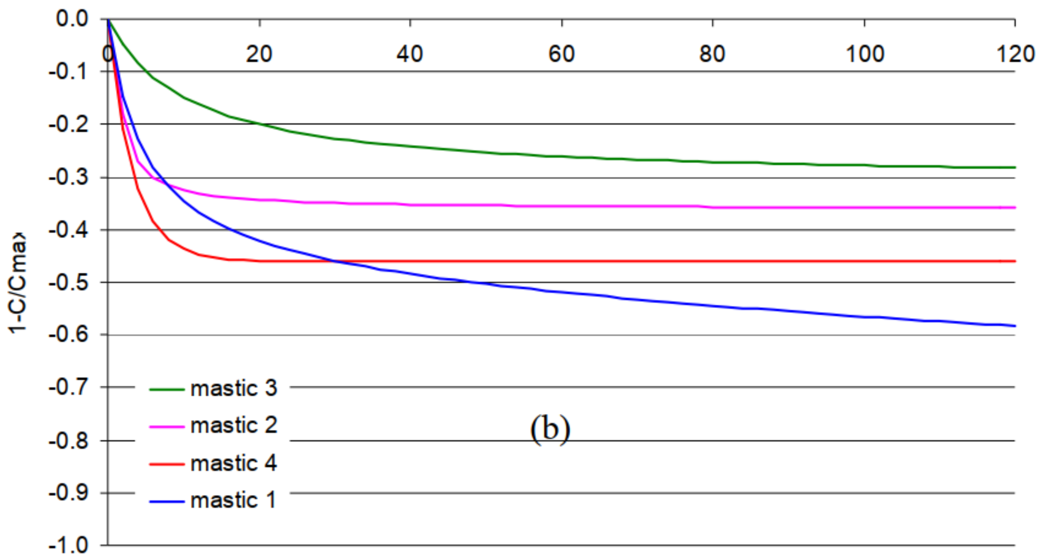


Figure 238. The loss of moisture from mastic – actual (a) and relative (b) (Kringos et al. 2011).

17.1.4 The compression using water pressure (ASFADUR)

As an alternative to Indirect Tensile Strength Ratio, the ASFADUR project (Laukkanen, Halonen & Pyy 2012) evaluated the applicability of a water pressure impact test. In principle, the test investigates how the samples after the initial compaction due to creep (3.5 mm) respond to 3000 cycles of strong water pressure. This test was intended to simulate the pressure of water in the pores due to the compression inflicted by the car tyres. The pressure was adjusted depending on the testing temperature

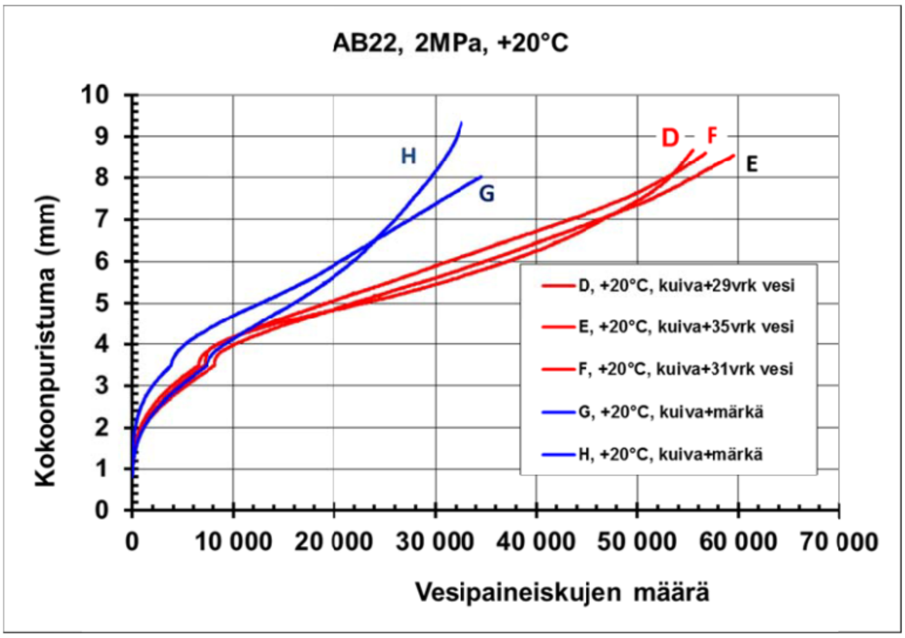
- 1000 and 2000 kPa at 20 °C
- 7000 kPa at 5 °C.

The AB22 samples were tested in various ways:

- All the samples were pre-creeped to 3.5 mm.
  a. Portion 1 was saturated under vacuum and kept for 28 days in water bath.
  b. Portion 2 was saturated with water under vacuum conditions but without the water bath conditioning afterwards.
  c. Portion 3 was kept dry.

The best resistance to water was observed in samples with air voids filled with water. The air voids in this study were kept exceptionally high, to simulate the potential situation observed in the joints. The air void level was targeted at 10% by dimensions.

The conclusion was that the preliminary creeping of the sample decreased the air void content and has improved the resistance of samples to the water pressure. The freeze-thawing cycles weakened the sample's resistance to water. If the samples were 100% saturated with water, they resisted the combined action of water and pressure better than in the undersaturated state (Figure 239).



Näyte		A	B	D	E	F	G	H	K	L
Tyhjätila IPK	%	6,1	6,5	6,4	6,6	6,5	6,8	6,6	6,2	6,4
Tiheys	kg/m^3	2303	2293	2295	2290	2294	2286	2292	2300	2297

Figure 239. The results from the water pressure impact test on vacuum saturated (H, G) and water bath saturated samples (D, F, E) depicting that first set deformed at faster speed than the water bath saturated ones (Laukkanen, Halonen & Pyy 2012).

The suggestion was that the water filling the voids is acting as a component of the composite and is also able to take the load. Hence, both in ITSR and compressive strength tests, the fully saturated sample is expressing the higher strength than the dry sample. This is proposed to be originating only in replacing the air by water. The observation was that if the result suggests 100% or over retained strength, this means that apparent strength was increased by the water in the filled voids by more than the weakening of the adhesion between binder and aggregate due to the exposure to moisture.

In this cyclic compression testing, the adhesion additive had a positive effect. The speed of creeping was reduced, tough it was not specified which promotor was considered for this particular evaluation.

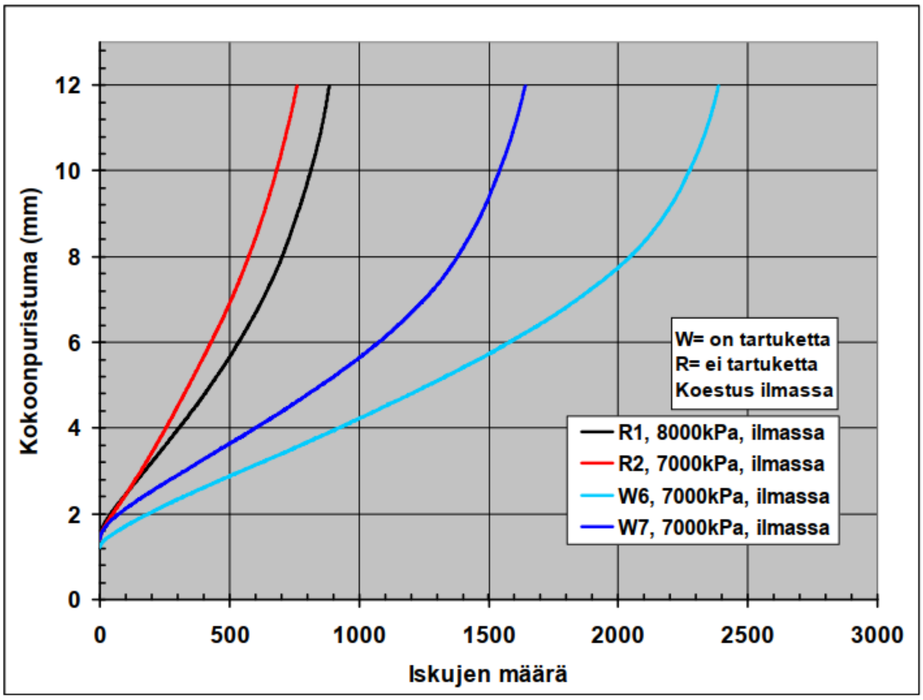


Figure 240. The effect of the adhesion promotor on the resistance to compressions using water pressure at 5 °C (W – no promotor, R – with promotor).

17.1.5 Tecnico accelerated ageing (TEAGE)

One of the aspects avoided in the research on the resistance to moisture, is in fact the aging of the bitumen. The moisture damage resistance is understood as a compatibility between raw materials. However, the moisture damage, loss of bitumen and raveling can happen as a result of pronounced aging.

The typical bitumen aging tests (such as Rolling Tin Film Oven Test or Pressure Aging Vessel) applied to the raw materials, test for its resistance to oxygen and heat. However, in the field situation the asphalt concrete pavements are exposed to sun, water, oxygen and heat. One of the more simple solutions, using essentially a box periodically filled with water and a source of UV light, is the Tecnico Accelerated Aging procedure presented schematically in Figure 241 (Crucho et al. 2020).

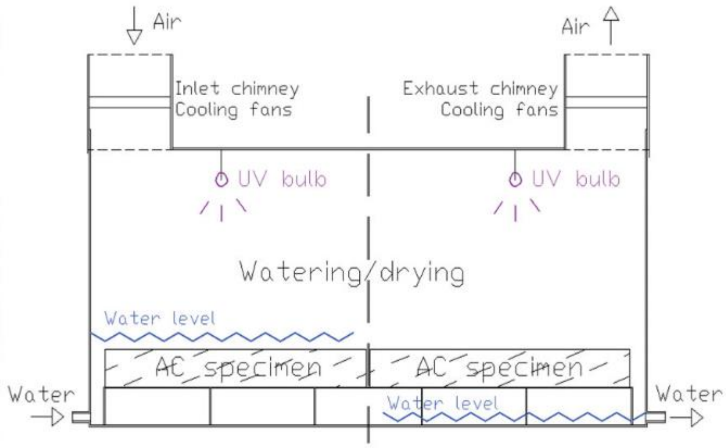


Figure 241. Schematic of a prototype of the TEAGE container (Crucho et al. 2020).

It was observed, by testing the water drained from the container, that during this type of aging there is a loss of bitumen from the specimens. Oxidation products become water soluble. The longer the period of aging, the more bitumen loss occurs, leading to the decrease of the fatigue life (Crucho et al. 2020).

This procedure was developed and adapted to simulate the aging in Lisbon conditions for the period of 7 years, by comparison with field observations (Crucho et al. 2020). For that reason, the direct applicability for the Finnish environment is perhaps not advised. On the other hand, the correlation with sun exposure in Finland as compared to Lisbon, should result in shorter aging time. Additionally, the alteration of the procedure to include the changes of the temperature above and below the 0 °C mark seems possible.

17.1.6 TEAGE + ASFADUR?

Perhaps, TEAGE procedure can be combined with the freeze-thawing procedure proposed during the ASFADUR project for a full evaluation of asphalt behavior due to weathering (Laukkanen, Halonen & Pyy 2012) and a laboratory simulation of field behavior to study the behavior of bitumen and additives.

After such combined conditioning procedure, just like any oxidative aging of bitumen, the stiffness of the binder is envisioned to increase, which should decrease the permanent deformation issues. However, the resistance to studded tyre abrasion (Prall), tensile strength and fatigue life should be evaluated afterwards for the treated mixtures. If the Cantabro test is considered as a predictor of particle loss and raveling resistance of the mixture, the test should be conducted on the aged samples as well.

Utilizing such modified test, could also answer the question on the behavior of the adhesion promoting additives during the life of the pavement. The amines, as water soluble particles could be expected to transfer into the water over the course of weathering. Studying the time necessary to fully drain and dry the sample, could answer about the water adsorption into the asphalt mixture. This methodology of aging could be a stepping stone towards the laboratory simulation of the multiple recycling.

18 The survey of officials and contractors – PART 5

In connection with project BITU2020 a decision was made to conduct the survey of professionals in Finland on the subjects covered in the literature research conducted within other work packages.

Due to the fact, that Finland is a relatively small market with a limited number of professionals dealing with the covered subjects, the decision was made to anonymize all of the answers. Because of that the questions were rather turned into multiple choice questions than opened ones.

The goal of the survey and analysis of the responses is to identify the current state of the practice related to the quality control and the quality assurance of all raw materials, ready asphalt mixtures and compacted asphalt pavements, with a specific focus on adherence to moisture damage prevention. Aalto University acted on the behalf of Finnish Transport Infrastructure Agency and INFRA organization in evaluation of the understanding and adherence to the existing specifications and guidelines.

The output of the project is a suggestion on the path for the development of the Finnish Asphalt Specifications in fluctuating market conditions. The point is to identify, if the currently existing guidelines and specifications are clear and understandable by both producers and quality controllers.

19 Survey organization

19.1 Goal

The goal of the survey was to:

- Map the magnitude of the problem of the moisture damage in Finland.
- Determine the level of understanding of the specifications related to moisture content in Finnish Asphalt Specifications (Asfalttinormit 2017, later referred to as AN2017) among professionals purchasing and producing asphalt concrete.
- Investigate the quality control procedures related to
  - aggregate
  - bitumen
  - adhesion promotor content choice
  - moisture damage related prediction tests.
- Map the currently used bituminous products.
- Map known and used modifiers in the industry.

19.2 Methods

A list of 50 questions was prepared by a steering group committee to be sent out to professionals in the Finnish asphalt concrete industry. The goal was to map the current understanding of the specifications, European standards, as well as to understand which products are already used on the market.

19.2.1 Steering group

The survey was initiated by the BITU2020 steering group, which comprised of:

- Katri Eskola, Väylävirasto
- Heikki Jämsä, INFRA ry
- Jussi Tuominen, NCC Industry Oy
- Antti Lyytinen, Skanska Industrial Solutions Oy
- Nina Orttenvuori, YIT Suomi Oy / PEAB Industri Oy
- Michalina Makowska, Aalto University
- Leena Korkiala-Tanttu, Aalto University.

19.2.2 Question development

The subject of the questionnaire was discussed already in the preparative state of the project. However, during the first two meetings with the steering group an additional discussion and review of interesting aspects among the interested parties was conducted. Based on those discussions, the manager of the project, namely Michalina Makowska, prepared a first draft of the survey. The list of questions proposed was distributed among the steering group members and a possibility to add interesting questions was provided.

At that stage additional questions were proposed by steering group members and incorporated into the questionnaire. The questions were translated from

English into Finnish by Michalina Makowska with the help of Heli Nikiforow. The final proofing comments were provided by other members of the steering group.

19.2.3 Tools

In order to assure anonymity of participants a WebproSurvey 3.0 tool provided by survey.aalto.fi was utilized. This tool allows an organization of the fully anonymized survey.

Two options to invite participants into the survey exist, one being personal link and the other a general web link from which a number of answers can be provided.

However, due to the fact of predictably low number of participants and a possibility of one person answering through the weblink multiple times, we opted for personal invitations. This way a number of participants was limited, and their actual connection to the industry assured.

19.2.4 Invited participants

Based on the discussions and the goal of the survey the steering group identified two groups, namely:

- **Officials**, that is people responsible for tender announcements, review acceptance and inspection of the documentation after the construction
- **Quality control professionals (QCP)**, namely people involved in preparation of the documentation related to quality control of asphalt concrete.

Based on that division two groups were invited.

The nine official workers on the behalf of Centre for Economic Development, Transport and the Environment (fin. ELY-keskus) from around Finland were invited as officials.

Representatives of companies were invited to participate in the survey, so that each company was asked how many workers within their company would be eligible to be considered a specialist on the subject. The companies contacted during interview comprised Skanska Industrial Solutions Oy, NCC Industry Oy, YIT Suomi Oy (towards end of project PEAB Industri Oy), Asfalttikallio Oy, GRK Road Oy, Mitta Oy. In total 31 professionals were invited into the survey from within of those companies.

19.2.5 List of questions

List of questions with available answers is provided in Appendix 1 in English, while in Appendix 2 in Finnish and with the print out of collected answers.

19.3 Responders

The total number of respondents amounted to 12 participants, of which two did not agree to take part in the survey. The split between officials and QCPs was equal and five responses was received from the side of officials and five from the side of QCP.

20 Analysis of the responses provided by the officials

20.1 The aim behind the question set prepared for the officials

Questions 3–22 were displayed for the participants who self-reported to be on the side of officials. The aim of the questions was to investigate the true magnitude of the moisture related issues in Finland based on the observations of people directly responsible for the maintenance of the infrastructure.

The aim of the survey was also to investigate if

- the evaluation of the distresses is based on used monitoring techniques or personal perception
- are the quality assurance documents of the raw materials such as slag aggregates according to the current specifications (Finnish Pavement Technology Advisory Council 2017)
- what is the understanding by the officials of the rules published in Finnish Asphalt Specifications related to moisture damage prevention
- what is the understanding of the standard 13108-20:2016 "Bituminous mixtures. Material specifications. Part 20: Type Testing" during the evaluation of the provided documentation.

20.2 Analysis of the answers

The answers were provided by relatively small group of people, but considering invitation of 9 participants on the side of officials, the participation of 5 assured 55,5% response rate.

20.2.1 Moisture damage related group of questions

Among the participants, 100% admitted to the observation of surprising moisture related damages within the last 5 years (Appendix 1, question 3). In terms of occurrence of such damages 60% of respondents observed less than 10 cases, and 40% of respondents observed 10–50 cases (Appendix 1, question 4). However, in question 5, only 60% of respondents admit to clearly observe an increase in such occurrences within the last 5 years.

This suggests that problem indeed exists in Finland but is not extremely pronounced. What is more, because of the fact that respondents are responsible for different geographical areas in Finland, this may suggest that some regions are more affected by moisture damage than the others.

In order to evaluate if the observation was just perception based and to investigate if the performance following tools are utilized by officials, a series of questions related to the subject was provided within question 6. A 100% of respondents admits to have been going to perform field evaluation of moisture damaged areas, of which 20% admits to perform those sometimes, and 80% quite often.

The confidence in the answers provided about the observations is therefore high.

In order to investigate if some of the non-destructive performance evaluating methods performed cyclically on the pavements in Finland are considered of use for this particular problem, the questionnaire asked about incorporation of chosen methods into the moisture damage evaluation. The chosen methods included:

- International Roughness Index (IRI)
- Transverse profile measurements – profile shapes
- Transverse profile measurement derived rut depth
- Surface roughness determined from the laser transverse profile measurements
- Ground Penetrating Radar measurements
- Virtual visual evaluations.

Theoretically, each of those methods could be helpful in the long-term follow-up of the performance related to the moisture damage, but it was interesting to see which of those methods are used in practice.

The International Roughness Index relates to all kind of distresses of the surface and their influence of the smoothness of ride. Therefore, IRI alone cannot be used to evaluate just the moisture damage, as the value will include both moisture damage, fatigue and thermal cracks. However, in combination with visual analysis, e.g. videos and pictures from the road, it is relatively easy to exclude the increasing influence of the thermal cracking onto the reading. The currently existing Road Picture database (Tiekuva Kanta 2020) is accessible for interested parties and the set of pictures collected during different years is available for comparison. Arguably, some areas are pictured more frequently than others which may affect to its usability. In principle the moisture damage demonstrates itself in loss of adhesion between the grains. This leads to the loss of grains and increase of the surface roughness, as well to the microcracking and pot-holes.

The formed potholes should be visible on the profile measurements. The sudden local change in the profile over the stretch of few meters may be indicative of local damage. On the other hand, as was discussed in the review of the literature related to the moisture damage, the loss of strength of the material results in increased cracking and failures leading to pronounced rutting, e.g. effect witnessed with Hamburg Wheel Tracking device. The effect of the moisture damage is expected to be most observable during the summer season. It is envisioned, that in areas in which the failure is material related, rather than localized due to the construction time related issues, the damage would demonstrate itself rather in increased rutting speed as well as in increasing surface roughness.

The recent development in the road surface monitoring techniques resulted in the development of the surface roughness evaluation tool based on the transverse profile measurements (Virtala, Alanaatu & Huuskonen-Snicker 2019). Currently, the technique is used to study bleeding and segregation after construction (Virtala, Alanaatu & Huuskonen-Snicker 2019). It is suggested hereby, that in the country with the abundantly used polishing inflicting studded tires, the evolution of the surface towards the increased roughness over time should be indicative of moisture damage.

As was reported previously, the permittivity measured from the pavement depends on the permittivity of the aggregate, binder and air voids as well as content of water inside of the pavement (Saarenketo 2006). This fact was utilized in the studies of moisture saturation of unbound materials (Saarenketo 2006). It was recently proposed that the following of the permittivity changes in the surface layers of the pavement during dry and wet seasons, would be in fact indicative of pavements ability to saturate with moisture, which could be recalculated into the air voids (Pellinen, Eskelinen & Hartikainen 2018). During the moisture damage, the adhesion between binder and aggregate is lost and additional voids and cracks are introduced into the pavement. As a result of that more moisture can infiltrate into the structure. Progressing moisture damage should be observable by increasing permittivity in the surface layers of the pavement over the course of time during rain season, see the explanation of the principle in the Figure 242.

Interestingly, 100% of respondent indicated that they do not use the IRI in evaluation of identification of potential moisture damaged areas. Only 20% of the respondents admits to occasionally use the rut depth data for evaluation of moisture damage, when 80% does not.

Only 20% of respondents admitted to occasional use of the surface roughness parameters developed from the transverse road profiles for the evaluation of moisture damage, and only 20% also admitted occasionally using the GPR data for the same purpose. However, 40% of the respondents use the data from laser scans and GPR scans to evaluate likelihood of moisture damage within the pavement. The remaining respondents split between the answers "no, never" and "no, but I am aware it would be plausible".

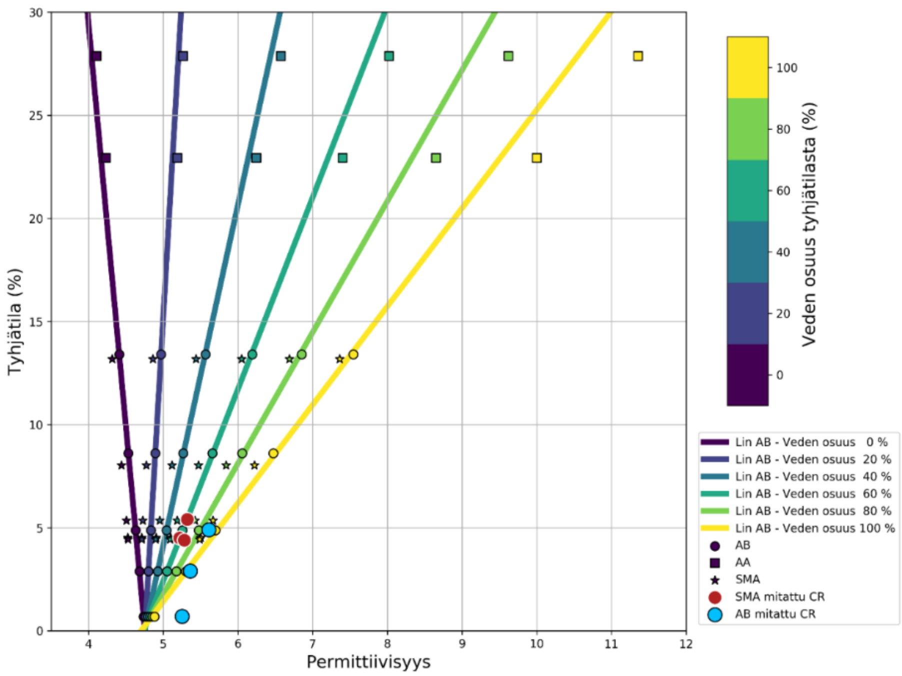


Figure 242. The relationship between permittivity, air voids content and moisture saturation level in the pavements proposed. Reprinted after Pellinen, Eskelinen & Hartikainen (2018).

The answers indicate that the potential of the set of tools available on the market for performance monitoring are transferring into the practice of official workers at slow pace. It is understandable that their role is not in the development of the monitoring techniques based just on the existence of monitoring equipment. However, it is suggested based on this literature review and answers from the officials to increase the education about the monitoring techniques and their potential application for evaluations. Ideally investing in software development, which easily transfers this sort of data from the field measurements into the decision making, is suggested. Increasing education on subject of data analysis and training for the officials could be of use with the goal of supporting digitalization in mind.

Part of the questionnaire was focusing on the aspect of evaluation of the quality assurance data related to moisture damage resistance prediction of AC and SMA pavements. The current guidelines are based on the results of ITSR and MYR tests described in detail in separate work package of this project. The 40% of respondents notified that they receive the raw data from measurements of the ITSR, not only the final percentage. However, when asked if they are aware how often is the ITSR measurement performed nobody admitted to know precisely, the respondents answered in 40% that they are not aware of that, or in 60% that they believe it is defined in the type testing related standard.

The Finnish Asphalt Specifications also define the specific limitations of the aggregate mineralogy for it to be considered a good road material. The answers of the officials split almost equally between paying attention to this aspect and never investigating this issue.

Due to the fact that soft asphalt pavements (SA, or PAB in Finnish) are required to be characterized by different methodologies than AC and SMA pavements, a separate set of questions was prepared for those materials. When asked if the MYR test is performed and its result delivered for SA-V mixtures, with the information about choice of adhesion promotor amount, only 20% of respondents gave a strong positive answer, 20% admitted that it happens occasionally, and 60% admitted to never receive such documentation.

The 60% of respondents admitted that they do not receive the evaluation of PAB-B by means of ITSR results (question 15).

Interestingly, the question 16, investigating the adherence to InfraRYL guidelines of providing the MYR test results during the production, which is not covered in FAS, was reported as received by 60% of respondents. The 40% of respondents do not receive such documents.

The aspect of testing the adhesion properties between soft bitumen and aggregate by means of testing the aggregate with stiff bitumen 70/100 was asked about in questions 17 and 18. The 60% of respondents believe that testing aggregate with stiffer bitumen is in fact predicting moisture resistance of the soft asphalt (PAB-B). Also 60% of respondents believe that compatibility between aggregate and soft bitumen for use in PAB-V is not required. The 40% of the respondents have the opposite opinion.

When asked of the knowledge of the frequency of testing of moisture damage resistance of the SA mixtures, 100% of responses were negative, of which 20% was negative, but knowledgeable on required frequency (question 19).

20.2.2 The set of questions related to the quality of the pavement

In the question 7, the officials were asked if they observed exceptional decrease of quality of the pavements within the last 5 years. Unanimously, all agreed that the quality has decreased, but 60% of the respondents are convinced that the cause is in different resistance of materials to traffic and weather conditions, while 40% is of the opinion that the deterioration in pavement quality is due to the accumulated lack of maintenance and repairs due to low budget, so-called "korjausvelka" (fin.).

A series of questions presented in section 8 was investigating the opinions of the officials in respect of the reason behind the decreased quality of the pavements. The only strong positive answer among all respondents was towards the influence of the changing weather and rain conditions in Finland and its effect on deterioration. Second highest result among respondents indicates that bad compaction and increasing air voids were the reason behind the deteriorations. For the majority of the other answers the officials were agreeing on a strong "maybe" answer. This indicates that in the opinion of the officials with the changes in climatic conditions other changes occurred, namely changes of contractors, changes of mixture designs, changes of the origin of the aggregates, changes in the supplier of bitumen. Interestingly 60% of respondents believe that change of the bitumen grade had no effect on performance. Interestingly, only 20% of respondents admitted to know the source of bitumen in the supervised construction sites.

20.2.3 Slag aggregate related questions

One of the observations of the industry and professionals prior to the BITU2020 project was that upon a change of the specifications related to the use of slag aggregate with the update of FAS to version in 2017, the change related to this material may have remained unnoticed. This is in fact supported by the answers provided by the officials. When asked if the slag aggregate is required to meet any volume expansion criteria, 60% of respondents answered negatively. This still indicates that 40% of the respondents are knowledgeable of this change. However, when asked if the producer of this product informs about the volume expansion 100% answered "no". A 100% of respondents also answered that they do not know how often this value should be tested or is tested by the producer.

This set of questions underlines one of the problems with the asphalt industry. Upon the change of specifications in 2017, the changes in the document were communicated. However, if the specification is not known to the professionals which should operate with it three years after its publication, a fault in the publication and education system is probably the cause.

20.3 Conclusions related to the responses of officials

One of the biggest issues identified during this survey is in insufficient understanding of specifications, which should comprise a basis of quality control activities. The suggestion is to review the procedures related to publishing, teaching and assuring that the changes in the documentation are recognized. Development of courses and certification are highly encouraged.

The distribution of answers provided by respondents indicates that some professionals are knowledgeable in aspects related to their field, while some professionals are not upgrading their competences. Aalto University has recognized a lack of quality assurance and quality control of the level of professionals on the Finnish market. In comparison to other engineering fields, in Finland where FISE certification is applied for geoengineering, the asphalt industry seems to remain an outlier of the civil engineering in Finland.

There is a lack of courses on the market related to upgrade of competences for staff, quality control workers and decision makers. The upgrade of the competences is left to self-study and optional personal development. It would be highly suggested to follow either FISE type certification procedures, accreditation similar as in FINAS, or internally developed quality assurance for asphalt professionals, similar as road safety course (fin. "tieturvakortti") or system adapted in Finnish Geotechnical Society.

Taking an example from other field connected with road structures, we can get inspiration from the geotechnical field. The Finnish Geotechnical society's (SGY) Site Investigation committee have organized a system, where site investigators apply for their competence certification from the committee. For practical reasons it has been arranged so that the one person keep a small company, which looks through the applications and makes evaluation. A small fee is collected form this certification (Lepistö 2020).

Previously Finnish Transportation Infrastructure Agency (fin. Väylä) has required since the year 2019 in their contract documents that the site investigation companies must go through a training of one day in field to learn to do undisturbed sampling in a right way to assure comparable results. Before this change, the professionals in geotechnical field were supposed to read the 4–5 pages guidelines. The training was given by an experienced technician from Aalto University. The training was supposed to happen in Helsinki region by the end of July. Each company had to pay for technician's fee and travelling costs.

To get continuity to a FISE certification, each qualified person has to show that they have participated for at least 2 days training during last years. For example, you can take part to SGY's typically one day trainings, where you get a certification. FISE accepts also courses organized by universities.

Suggested aspects to cover within such certification for asphalt industry, would include courses on:

- existing specifications with explanation on the reasons behind the formulations,
- changes in specifications from previous years, with explanation of the importance, risks and reason behind them,
- the aspects related to the type testing and quality assurance
- novel developed techniques of monitoring, especially available in Finland
- tools available for the pavement engineers on the Finnish market, i.e., methodologies, available databases and softwares (practical workshop to test the softwares).

The full advantage of the investments related to research and development may be realized when the transfer of the results to practitioners is supported by education. Improvement of specifications and standards is bringing the assumed benefits, only if the people responsible of assuring adherence to those documents are knowledgeable about their content and the purpose of the requirements.

21 Analysis of the responses provided by the QCP

21.1.1 The aim behind the question set prepared for the officials

The questions directed towards QCP are between questions 24–50 in the survey. The first set of questions focuses on the quality control of the aggregates (24–32), which is followed by the quality control of binder (33–39), adhesion promotors and moisture damage resistance control (40–49). The last question was intended to investigate if there are already some preferences towards brands of modifiers within Finnish industry or interest in promising new materials on the market. The QCP were asked what the product brands are they are familiar with.

The questions related to the frequency of testing were intended on evaluating how the knowledge dispersed currently in multiple sources is taken up and understood in the industry. Currently, some production related quality control guidelines are provided in INFRA RYL (Rakennustietosäätiö RTS 2010), mixture design related guidelines in Finnish Asphalt Specifications (Finnish Pavement Technology Advisory Council 2017), while type testing and quality control during production is defined in European standards (SFS-EN 13108-20 and SFS-EN 13108-21). In the European standards, however, a freedom to self-define the frequency of testing within some allowed range is left for the producer. Due to this fact, different producers may be performing QC with a drastically different frequency.

Because of the results of a review of evolution of the ITSR technique and the potential sources of error, described in detail in Part 4 of BITU2020 – Phase, a series of parameters related to laboratory procedures was identified and asked about in questions 41–43.

21.1.2 Analysis of the answers by QCPs

The answers to question 24, indicate that Nordic Ball Mill abrasion test is the most respected property of the aggregate and the parameter with the highest control level in the industry. Unfortunately, this particular question in the survey had a technical error. The intention of the question was to be multiple answer question. However, the question was not allowing the respondent to pass to the next question unless all the rows were ticked. This was noticed and reported to the organizers by a second respondent, the issue was fixed and starting from second respondent the survey was working properly. The first respondent decided to tick Nordic Ball Mill abrasion answer for every row in order to pass to the next question. For that reason the answers from this question in the survey are considered indicative at this point and should not be used as official knowledge.

Nevertheless, it is clearly visible that freeze-thawing test of aggregate is never conducted by the participants. The quality control of the water absorption testing occurs typically when the supplier is changed, or the stockpiles are renewed. Interestingly, one response indicated that this test is never performed and one that test is performed on schedule once a year. In question 30, however, the supplementary questions about water absorption test were asked. In this case it seems that perhaps the test is not performed by all on the regular schedule, but majority is ready to perform it when requested by officials. Interestingly, the water absorption of aggregates with water absorption above the level of 1% is assured by only 20% of respondents, while 20% admits to never do it, and the remaining 60% admits to be ready to perform it when requested by officials. Of course, the negative answer may indicate that the QCP is not using the aggregates with water absorption levels of above 1% in their practice.

Question 31 and 32 indicate that the information about the mineralogy is coming mostly from the side of the aggregate producers, but 20% of respondents admits to be performing the quality assuring tests themselves.

In terms of assurance of filler properties, since the surface area of filler is linked to the moisture resistance of the mixtures, interesting was to evaluate if this parameter is reassured. Unfortunately, 80% of respondents admitted not to test for that property (question 28), while only 20% admitted performing this step.

The set of questions related to the properties of slag aggregates used for paving applications, indicate similar split between QCPs as in the case of officials. Only 40% of respondents was aware of the existing requirement related to the volume expansion in the Finnish Asphalt Specifications 2017. Of course, one of the issues is that the use of this material is rather localized currently in the Northern regions of Finland. Therefore, from practical point of view QCP and officials involved in construction works in northern parts of Finland are expected to be more knowledgeable of the subject than in other parts of Finland. Nevertheless, since the companies operate in different regions in different years, it was expected that the knowledge of this fact would be higher on the side of contractors than officials. Only one QCP (20%) respondent answered that the producer of the slag aggregates reports the volume expansion. However, the information about the frequency of testing of this property from the side of producer is not known by 100% of respondents.

The questions 34–38 were focusing on the quality control of the bitumen. Only one respondent admitted testing the traditional bitumen rheological characteristics such as Penetration, Softening Point and viscosity upon a change of the bitumen source. On the other hand, only one respondent admitted using different bitumen providers. The 60% of respondents admitted checking the properties when the smell indicates a significant change in the product, but 100% of respondents admits using the data provided by the producer of bitumen in their quality control reporting. Such measures are allowed in the Type Testing standard.

According to the answers, there is currently no defined frequency of quality control of received product, while the standard suggests at least one grade property evaluation per 300 tons of one grade or one class of binder. When asked if there have been changes in the quality of the bitumen, 60% of respondents said that it occurs from time to time, while 40% did not observe differences. When asked if the type of bitumen has changed within the 3 last years, one respondent again admitted using different providers and observing the change. Two of the respondents mentioned that there was no changes in type and 2 that it sometimes occurs. Interestingly only 60% admitted to be occasionally performing the quality control of received product. Neither of the respondents admitted using spectroscopy in their quality control activities.

The question 39 was intended to evaluate if the control of the adhesion promotor as asphalt component occurs in asphalt plants by the organoleptic means (e.g. color, smell). This is suggested to be performed with defined frequency in the factory production control standard EN 13108-21. One respondent admitted that this is conducted by means of smell, one that only visual evaluation is used, one that the evaluation is not conducted, and two respondents admitted performing the quality control of that component in some other way. The certain frequency to perform such evaluation of adhesion promotors is defined in the type testing standard, but similar as in the case of aggregate and binder the producer of asphalt concrete may relay on self-reported quality control data by the producer of component.

It is worrying that the only quality assurance between provider and factory happens on the site of component provider for binder and adhesion promotors. Even if in case of modifiers, their use is relatively low, the volume of binder used is quite high and the properties of the binder directly influence the performance of the asphalt pavement. More control, or quality assurance would be desired from factories for those components, especially considering that similar activities are reported for aggregates.

The responses related to the moisture damage prevention tests, reveal interesting split between QCPs. Only 40% of respondents is always testing the adhesion between the aggregate and binder during mix design using the procedure described in EN 12697-12 Method C (MYR-test) for PAB-V. It seems that 20% of respondents is using their previous experience and trusts that when the same source of aggregate is used the evaluation is unnecessary. While 40% of respondents is using different than required tests to evaluate the need of adhesion promotor. Not all of the mixtures need to be marked with the CE logo. However, we could assume the suggestion in the type testing standard as baseline. The MYR is required at least once per formulation of the mixture.

The question about PAB mixtures and the use of MYR test indicates also that the existence of multiple guidelines about the frequency of testing is misleading. 80% of respondents perform it at mix design stage (acceptable according to type testing suggestions), 80% performs it when the source of aggregate changes acceptable according to the type testing suggestion). However, only one respondent admitted to recheck the value upon change in source of bitumen or parameters in aggregate. It is worth mentioning that only one respondent admitted using different sources of bitumen. Two respondents admitted checking MYR of PAB mixtures upon the change of amount of adhesion promotor, which is essentially the reason why the test should be performed. The requirement of performing the test every 1000 tons, as suggested in INFRA RYL guidelines is reported only by two respondents. The suggestion in the same guidelines is to be performing the "bucket test" (fin. "sankokoe") during the production of PAB mixtures. Only 60% of respondents admits performing those, but mostly for own production control use.

When asked about the use of the method PANK 4301 (in which 70/100 bitumen with Open Asphalt gradation aggregate is used) for the PAB mixtures, majority admitted to use it during the mixture design and upon change in source of aggregate. This stays in line with the belief that the test evaluates effect on adhesion from the side of aggregate. Interestingly, one of the respondents answered that the test is repeated upon the change in adhesion promotor.

When asked how often the moisture resistance test is evaluated for the mixtures, in general question the 100% answer fell into the option “only during the design of the mixture”. In retrospective, there was no question “how often do you perform the design of the mixture”. Nevertheless, when the questions turned into more detailed aspects of the frequency for specific mixtures, such as AC and SMA, the answers varied a little. The question related to the frequency of testing is investigating what is producers’ own defined quality frequency, which in the type testing describing standard is defined as minimum values. This was a multiple answer question, as there is many circumstances in which the standard suggests that tests should be repeated. Nevertheless, 4 answers fell into “only during mix design”, one answer that this is performed at 1 test per 5 year and two answers that 1 test per 3 years. 80% of respondents admits performing the test when the source of aggregate changes, which is relatively adhering to type testing requirements. However, less than full number answers indicated that the water resistance testing is conducted upon changes in aggregates and fillers. Interestingly, the change in the source of RAP is not triggering the testing as answered by the respondents.

The suggestion from the type testing standard SFS-EN 13108-20:2016 is to demonstrate the adherence to the reported value upon the change in components, that is source of aggregate, grade or type of bitumen, content of RC addition of additives. However, the type testing standard accepts all bitumen sources as equal provided the grade is the same (or not negatively affecting the properties of the final product declared). However, upon the change of bitumen grade – type testing should happen. This was answered only by one respondent. Admittedly, when the change in the bitumen grade occurs the type of the mixture could be considered as different and perhaps the mixture is considered as different by the producer.

The context for the answers is provided below as a partial review of SFS-EN 13108-20:2016:

- Type Test report is valid for a single mix formulation as long as there is no changes in constituent materials, or in their properties which may influence the mix formulation, characteristics or performance (if performance is reduced in comparison to declared)
- Changes in raw materials considered a trigger for repeated Type Testing
    - Aggregate properties change: shape, percentage of crushed and broken surfaces, resistance to fragmentation, resistance to wear, resistance to abrasion by studded tyres, change in rock type as defined by EN 932-3, change in particle density greater than 0,05 Mg/m^3,
    - Aggregate or filler origin change
    - Bitumen grade or type change
    - Reclaimed asphalt properties result in a change to the performance levels or classes of the mix formulation as declared in the Type Test Report
    - Additive change to the extent that the performance levels of classes of the mix formulation will change from those declared
- Soft Asphalts require at least one MYR test during mix formulation
- Type Test is valid for 5 years.

Lastly, the list of questions focused on the differences between laboratory practices of performing ITSR indicates that the problems with repeatability and reproducibility may be related to the lack of control of certain important parameters discussed in previous parts of this report series.

- The use of freeze-thaw cycle for evaluation of SMA and AC mixtures is not performed, but is also not required. Nobody performs it voluntarily.
- Adherence to the certain height difference between samples is reported only by one respondent. 80% does not pay attention to it.
- One respondent controls the samples by fixed mass of sample before compaction and two respondents prepare the samples by applying certain amount of gyratory compactions/marshal blows. Since 100% of respondents answered that the mixtures are not comparable by air voids, the indication is that air void content is not the determining parameter in sample preparation. Question remains, what is it then if not height, air voids or number of applied compactions?
- The saturation level is not controlled for as indicated by 80% of responses.
- Controlling if binder content between dry and wet sets is similar was reported only by 40% respondents.

As indicated by the research reviewed in part 4 of the report, the method of compaction has an effect on the results. The analysis of responses in question 42 is problematic. Seemingly all three methods of compaction are used in Finland. Indeed, the question was not specific enough to ask which compaction method was used with which mixture. It is suggested to evaluate if the results reported by different companies are comparable, based on the possible differences in sample preparation methodology.

21.1.3 Conclusions related to the responses of QCPs

Similarly, as in the case of officials, QCPs answers were not uniform. The differences between companies exist in terms of understanding and applying the standards, regulations, specifications, and guidelines. The system would benefit from simplification and unification.

The problems with water resistance could very well originate in the low frequency of testing of this parameter on the producer side. Unification of the methodology of performing the test could be useful for comparison between mixtures from various providers.

22 Suggestions

22.1 The future bitumen quality criteria based on database

In the light of the changes in legislation, it is suggested that Finland should follow the product information and data collected in the databases by the officials in the countries of potential future exporters (e.g. Zofka & Błażejowski 2018; Radenberg et al. 2016) or information from the countries, which at this point are importing the products (Dasek et al. 2017).

However, it is not sufficient to rely on the data available for the past years and decades, as the market situation fluctuates and is expected to dramatically change in 2020 and after 2 transition years from it. As an example, the Plock refinery announced the investment into the visbreaking unit, which should be operational by the end of 2020 (Reuters 2018). Such changes potentially create the lower production of the bitumen and higher likelihood of the use of non-traditional vacuum residue derived bitumen types.

It will become strategically important to be able to identify the bitumen type and composition in a quick manner. The ability to identify vis-broken residue, HFSO, deasphalted oils with fluxes and to understand the effect of the composition on the future performance will be of high importance. In the case of absence of quality assurance system, the market may be opened to the influx of products of lower quality rejected by buyers elsewhere.

Because the database of performance of those future products may in fact not exist at the moment, the importance of the increased quality control an assurance in the transition period is underlined. **Some of the products on the market may in fact be released for the first time in the following years. The increased performance testing of the mixtures prepared with the new bitumens is advised.**

Current European bitumen grading system focuses on fresh bitumen properties and its behavior during short-term aging. However, the signals indicate that it is the long-term performance (equivalent to three years in service) that may be affected the most by the differences in the products (Dasek et al. 2017).

Finland should revisit its bitumen quality criteria during future years and identify the performance predicting parameters for the novel products emerging on the market. Relying on the knowledge developed in 1993 during the ASTO program (the basis of the current asphalt specifications (Finnish Pavement Technology Advisory Council 2017) may be insufficient in the future (Kurki, Halttunen et al. 1993).

22.2 Suggestions for moisture damage studies – The most promising path

In case if the ITSR test is still of interest, the effort should be placed to unify the testing procedure in Finland, as well as to prepare calibration tests using various binder sources which are more in line with the binders currently on the market. In order to improve the repeatability of the Strength test, suggested is a consideration of semi-circular specimen testing. The inclusion of freeze-thawing cycle to predict the behavior in field is still advised.

The additives reviewed in combination with the review of the different tests seem to pose a risk of overdose or underdose, and it is strongly underlined here that each time an additive would be added into the mixture, a detailed mixture design would need to occur testing the performance in terms of moisture susceptibility. For this reason it is of most importance to focus on either tweaking the currently used method using some of the parameters discussed in section 14.1 or to switch to a method which allows a good predictability of performance in field. With the switch in mind – some of the most differentiating tests between asphalt concrete mixtures found for asphalt were Hamburg Wheel test and Cantabro. The HWT is testing the creep and moisture sensitivity, while applying the load in the form which is inflicted by the traffic and thus expected to simulate the road conditions in most reliable way.

During the review it was underlined, however, that addition of adhesion promotors may influence the permanent deformation or resistance to abrasion. The designers of the mixtures should be aware and considerate of this.

The effect of bitumen on the abrasion and resistance to moisture was studied in Finland in worldwide, and it is important to remember that different source of binder may inflict different performance in field. This simple fact may have been forgotten in the Finnish industry in the relatively simple market between 1995–2014. With the increasing influx of bitumens from different sources the mixture designers should evaluate the influence of the binder on the mixture properties in order to predict the plausible risk coming from the use of different source. However, currently the source of the binder is not disclosed upon purchase and it may be relatively complicated to distinguish between binders. For this reason, the PART 2 of BITU2020 literature review series discusses the possibilities available for the mixture designers for the distinguishing among products.

22.2.1 Caution in focusing on only moisture related damage

The set of reports produced for the purpose of Part 4 in BITU2020 project focuses on describing performance aspects related to the presence of moisture. However, there are many types of failures in asphalt concrete pavements. The development of the pot-holes follows seepage of water through cracks and standing of the water inside of the such distresses as increased local raveling, cracking inflicted as a result of fatigue damage, low temperature cracking (Dore & Zubeck 2009).

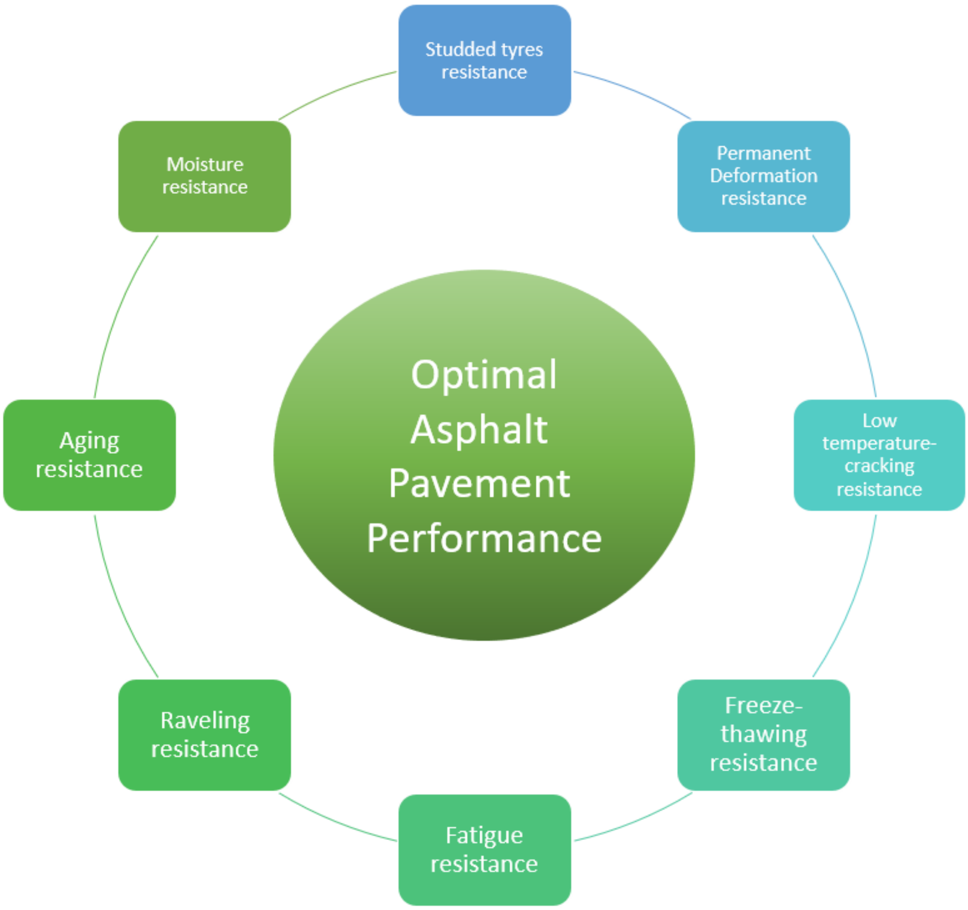


Figure 243. The optimal characteristic of well performing asphalt concrete pavement.

For a long period of time, the Finnish Asphalt Specifications were tunneled towards optimization of resistance to abrasion by studded tyres and permanent deformation. The testing of the mixtures covered only three of the aspects leading to the deterioration of asphalt pavements.

The ideal way forward would be to incorporate multiple performance evaluators for the best material. If the material is having high moisture resistance, but is likely to age prematurely, the likelihood of cracking and raveling is high. This on the other hand would lead to moisture damage. The fatigue, thermal stresses and aging are all inflicting cracks into pavement, creating new path of water entry into the structure. However, maximizing the resistance to permanent deformation and fatigue, may result in higher susceptibility to studded tyre inflicted abrasion and higher occurrences of thermal cracking.

During the literature review it became apparent that any modification of the asphalt towards improvement in one of the mechanisms of damage, has a power to lower the resistance of pavement to damage through different failure mode.

The optimization of moisture damage should be accompanied by the studies on:

- Behavior at low temperatures
- Permanent deformation

- Fatigue resistance
- Aging resistance.

22.2.2 Low Temperature cracking

A significant part of this report was based on the experiences from Poland (Zofka, Maliszewska, Horodecka et al. 2013), Sweden (Olsson, Krona & Nordgren 2010) and Finland (Kurki, Halttunen, et al. 1993). What is worth mentioning, is that during those research programs the resistance to low temperature cracking was evaluated as well in different configurations.

The Finnish mixes (Kurki, Halttunen, et al. 1993) were tested during the ASTO project by Thermal Stress Restrained Specimen Test (TSRST) in limited combination range of two mixtures AC12 and AC22 with two bitumens, B120-AH unmodified and airblown. It was pointed out that the mixture change did not affect the cracking temperature significantly. However, the bitumen characteristics seemed to play a role.

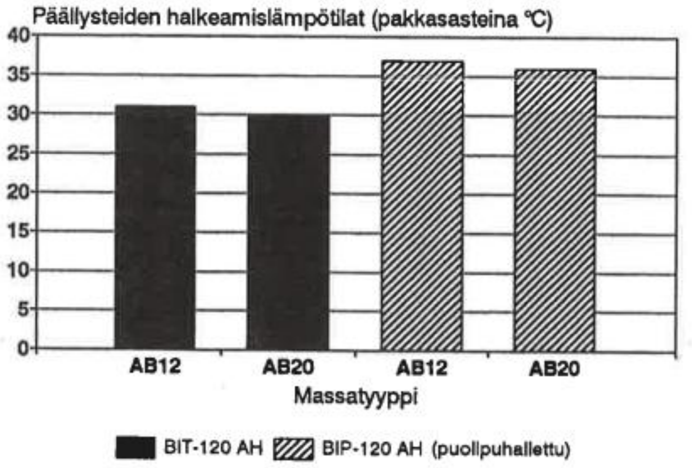


Figure 244. The results of the TSRST test from ASTO (Kurki, Halttunen et al. 1993).

In the Swedish study (Olsson, Krona & Nordgren 2010), the differences in TSRST result (cracking temperature) were deemed insignificant between samples, or rather not differentiating between the samples. The values recorded from each sample varied between -20…-25 °C for all seven tested bitumen. The observation was made that the stiffer the bitumen, the faster the cracking occurs during the test.

The Polish study (Zofka, Maliszewska, Horodecka et al. 2013) evaluated the influence of additives on the low temperature cracking, using the TSRST as well. However, as for the HWT (Table 27) only chosen mixtures were taken into testing. Because of that, only the quartzite mixtures had an unmodified binder reference and two adhesion promotors, namely AD-here and hydrated lime. The average of the results for those mixtures differed between -19.64…-20.88 °C, which can be considered as insignificant influence. Granted, the applied experimental matrix allows only for comparison of two adhesion promotors on one type of mixture and one type of bitumen.

Considering the above, it would be advisable to include the TSRST testing in the optimization effort of the mixtures in Finland, especially in case of decision making between different products. Alternatively, the binder related tests focusing on describing its low temperature properties could be of more interest in the future (Part 2 of the report). The good resistance to low temperature cracking assures lower occurrence of the cracks and lowers chances for moisture infiltration into the pavement. It is not directly a test indicating the resistance to moisture damage, and yet it is indicative of the performance of the mixture.

22.3 Envisioned-long term future

Of course, the BITU2020 project is focused on the characterization of the bitumen, thus one of the conclusions should be to follow the quality of the bitumen. However, as was presented in the part 1 of the report, the sources of crude oil are limited and depleting. The source of crude oil is unstable in case the political situation becomes unstable. Any conflicts in the countries known to be providing good quality bitumen will influence the market, as we have seen for example within last years in the case of Venezuela.

As the sources of crude oil deplete, struggles are envisioned. The effect to the bitumen production may be more drastic or beneficial, and it is hard to predict. However, it means that the emergence of the non-bituminous binders is envisioned in the scenario in which crude oil availability is limited due to political conflicts. As a result, the tests required for characterization of the bitumen, which are developed based on decades of experience and correlation of field behavior to the composition of bitumen, may become obsolete.

Novel bio-based materials may behave completely different in the tests designed for bitumen, for example Rolling Thin Film Oven test (do Nascimento Camargo, Bernucci and Vasconcelos 2019). But in fact, their behavior in field may be good or similar. Therefore, looking at a long-term future it would be beneficial to focus on the performance of the composite, the asphalt concrete itself, once it is prepared.

In the chase of the development of the products we should have a database of criteria met by our pavements currently, such as resistance of asphalt concrete to deformation, fatigue, moisture damage etc., will provide us in the future the aim to which the novel materials will try to live up to.

Therefore, fast discriminative tests for bitumen in order to distinguish its source and to determine its compatibility with aggregate and modifiers, is definitely an advantage to current mix design and mixture testing procedure. However, the mechanical tests of the ready mixtures should not be abandoned in terms of the baseline of the status quo.

22.4 Machine learning and bitumen quality

The importance of the correctly build database for the development of the future artificial neural networks is underlined here. The studies reported to use MLAs use the limited sample sets, typically locally sourced.

The suggestion is to keep collecting the infrared data from fresh, RTFOT and PAV aged samples, as well as from those extracted from cores and RAP material before recycling – in combination with knowledge of the source of the original binder in RAP.

The role of digitalization and data storage for the future purposes is huge in this case. A data point without metadata and descriptors is useless for future developments, as will be discussed in the machine learning section of this report. Therefore, it is suggested that testing of the binders and data collection related to the materials available locally in field starts as soon as possible and is linked to the pavements, as well as their performance markers.

Designing the database – which spectra is expected to be indicative of what? How much data is necessary to distinguish between multiple parameters and at what point do we encounter the data redundancy? Is adding additional parameters improving the categorization? How to collect data in the best way?

Some of the issues to consider during the research while starting to analyze the samples:

- Developing the database collection protocol – sample naming, list of tests, procedures.
- Performing supervised learning knowing the link between IR and rheology and performance.
- Performing the unsupervised learning to investigate yet unapparent patterns between bitumen parameters.
- Adding more dimensions of data to differentiate between samples not only in x-y coordinate system, but also in XYZ coordinate system – not only spectra – but development of spectra over time and possibly gases.
- Testing the redundancy levels for bitumen applied in the field.

22.5 Research questions as a result of review of the mechanism of the moisture damage

1. What is the capacity of bitumen to absorb moisture in form of vapour? The potential higher sodium levels in some bitumens may cause differences in osmotic transfer of water through bitumen films. Or is this on average the same and rather dependent on the RH%?
2. Can the mixture design influence the moisture suction and storage in our case? Is the mastic in fact the governing parameter, which we can manipulate? Is drying a parameter which we can affect by a choice of materials?
3. Apparently, different rolling compaction may inflict different distribution of air voids in the structure. This will influence not only the air voids content, but the permeability, diffusion of vapour and capillary suction within the pavement. Should we investigate also the most optimal compaction method for the optimal resistance to moisture damage?
4. If calcium hydroxide absorbs the products of aging, should this be the additive for most oxidative aging prone bitumens?
5. Is there one modifier, which can be applied to all bitumens? What are the rules? Do we loose some recyclability with the additives?

6. Could the maintenance technique such as application of deicers decrease the observed moisture damage? Decreasing the surface tension of the water on top of the pavement during spring (period of time of low RH and higher temperatures) could slow down the progress of the damage.

22.6 Suggested paths for moisture resistance testing

As a result of the literature review a suggestion is to investigate applicability of:

- acidity and basicity of the binder, and the adhesives
- the rheological studies of binders with consideration of the high frequency measurements at low temperatures to predict the studded tyre wear due to the binder
- Hamburg Wheel tracking device – to study the resistance to creep but also the stripping point
- semi-circular bending test in place of indirect tensile strength test in ITSR test
- the use of well heated and overheated aggregate in laboratory evaluations if such procedure is planned in plant production
- the Hallberg test for evaluation of the stripping work which compares well to the existing data from ASTO
- the Cantabro test as a measure of resistance to raveling.

Improving control of existing methods:

- water adsorption in fines – linked with Hallberg test
- conducting the MYR test for SA mixtures.
- collecting the “sankokoe” test results and specifying the procedure for it
- ITSR – improvement of instruction, linking the result to the field performance
  a. the effect of binder source on the result
  b. the effect of RAP addition
  c. current additives
  d. superheating of the aggregate.

The suggestion would be also to focus on the conscious material choice and optimal mix design, considering the hydrophobicity of the mastic, drainage, and permeability of the asphalt concrete. This implies:

- control of the air voids and
- mastic composition as a moisture absorbing material, as well as
- control of diffusion of moisture through binder.

22.7 Suggestions based on the results of the survey

The question related to bitumen quality indicate that no differences in products were observed. Interestingly only 60% admitted occasionally performing the quality control of received product. The question remains if it is plausible to notice differences in substrate which quality is not evaluated. It would be encouraged that an operating quality level was set within the company, and that definition of the baseline of used component existed, in order to evaluate if any deviation from norm occurs.

In terms of potential adopting of use of adhesion promotors, a good definition of their acceptance quality control limit should be provided or defined in the country before the large scale use can be started.

The Type Testing standard allows the use of the quality control reports of components from the producer of component. However, the responsibility to assure conformity of the final product upon changes (non-conformities) in the component relays on the side of the asphalt concrete producer, as it is the producer who assures the declared performance. It is understandable that performing of the quality control of each component would require significant stress on the laboratory. However, complete negligence to investigate if component received is a component ordered creates a space for plausible loss of quality of the final mixture.

When not performing the quality assurance and random sampling, the low ITSR testing frequency is worrying. The type testing frequency of one per 5 years, is allowed in the type testing standard as the minimum. From this minimum, the producer is expected to derive the frequency based on statistically processed data which demonstrates what is in fact acceptable testing frequency to assure no negative effect to the performance.

However, it is postulated here that during the fluctuations of the components on the market such extremely low frequency is not sufficient to catch the deviations from norm or changes on the market. Despite the fact, that the standard unifies the bitumen of the same grade as the same product, the practitioners are advised to test for adherence to ITSR results upon the change of the bitumen provider. The constitution of bitumen may affect the adhesion as discussed in other documents disclosed within this project (BITU2020 – Part 4).

Additionally, the fact that requirements about frequency of testing are dispersed in various sources, should be considered as risk factor in the Finnish asphalt industry. Suggested is collection of all the requirements in one, clear document for use in asphalt industry.

It is suggested to evaluate if the results of ITSR reported by different companies are comparable, based on the possible differences in sample preparation methodologies. A clear guideline would be desired. Of course, some parameters may be coming from the client and their own requirements. However, if the products operate on the same market and are to be considered exchangeable, an ability to compare the results is of high value and based on the set of the responses the comparability may be questionable.

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Glossary

Asphal-tene	Highest molecular weight fraction of bitumen insoluble in n-heptan; having the highest content of sulfur (Hunter, Self & Read 2015; McKenna, Marshall & Rodgers 2013)
Aromatics	A fraction of bitumen characterized by a certain solubility, composed of aromatic compounds
Resins	A higher molecular weight fraction of bitumen characterized by a certain solubility, composed of aromatic and more polar compounds
Saturates	A fraction of bitumen characterized by a certain solubility, composed at large of alifatic compounds
Maltenes	The non-asphaltene fraction of bitumen, soluble in n-heptane (McKenna, Marshall & Rodgers 2013; Hunter, Self & Read 2015)
API	The American Petroleum Institute gravity; petroleum gravity compared to that of water (Hunter, Self & Read 2015)
S	Sulphur also sulfur. In the context of feedstock can be in the form of heteroatom in the large organic molecule or in crystalline form after separation from feedstock
AR	Atmospheric residue
VR	Vacuum residue
HSFO	High sulfur fuel oil, contains less than 3.5% sulfur by weight
MFO	Marine fuel oil, contains less than 0.5% sulfur by weight
VDU	Vacuum distillation unit
VBR	Visbroken residue
VBU	Visbreaking unit
CO_2	Carbon dioxide
H_2S	Hydrogen sulfide
SO_x	Sulfur oxides
IMO	International Maritime Organization

Project BITU 2020

1. Information for Research Participants in Project BITU2020

You have been invited to participate in a research study. Participation in this study is voluntary. You can discontinue your participation in the study at any time. Should you discontinue your participation, you will not be subject to any negative consequences, but information gathered from you up until the point of cessation of your participation may be used in the study according to this Privacy Notice and the applicable data protection legislation.

Privacy Notice for Project BITU 2020

1. The subject, goal and the possible funder of the research study:

The project focuses on reviewing the changes in refining industry due to the IMO2020 regulations and their impact on the bitumen supply. At the core focus of the project is a gain in understanding of the possible fluctuations on the bitumen market, potential changes to bitumen composition, as well as the impact those regulations may have on the performance of the asphalt pavements past the transition time of 2020-2022.

Within the project, a special focus is added to the differentiation between bitumen and bituminous mixtures composition. The link between chemical composition of bituminous mixtures and their performance, especially in terms of resistance to moisture damage and low-temperature inflicted damage, will be evaluated. The project reviews the current state of the practice in Finland in relation to the prevention of moisture damage in asphalt pavements.

Project is co-sponsored by the Finish Transport Infrastructure Agency and INFRA organization.

2. The concrete method by with personal data is collected; for example that data is collected by recording interviews or from the registries of public officials, how much of the participant's time is needed for research study participation, where the study will be conducted:

The survey is conducted through questionnaire, which can be assisted by the conversation with organizer. The conversation is to happen via skype or in person and will be recorded and stored as research data. Time required for answering: about 15 minutes.

3. Other possible:

The answers to the survey questions will comprise a material used during the development of the future Finnish Asphalt Specifications and production guidelines, as well as the input into future research needs statement. The goal is to gather honest answers, which will either lead to the clarification of the guidelines or further development of thereof.

4. What personal data is processed?

The personal data processed in the research project will include name of the participant, their e-mail address, skype ID, profession and job title, as well as date of the conversation.

5. Personal data has been collected from the following sources:

The participants in the survey are identified by the Finnish Transport Infrastructure Agency (FTIA) as collaborators, producers and quality controllers on behalf of the FTIA within the last 5 years. This data is vested in the representative of Aalto University responsible for organization of the survey. The FTIA shares the contact details to the contact person within the organization. The survey may be forwarded inside the organization to the person directly responsible for the activities covered in the survey. The information about the identity of the responsible person comes from the original contact person.

6. a) How is personal data processed in the study?

The concrete method by with personal data is collected and analysed.
The participants of the survey are suggested by the FTIA and their identity is known.
In the case of necessary assistance or question to survey organizer, the skype calls and conversations will be recorded. It is advised not to disclose the information which may allow matching of answers with the survey participant.
The link to the survey is personal and send to the e-mail account of the participant.
The survey is anonymous, but differentiates the subjects into two groups, namely quality controllers and producers. The results of the survey will be published in the form of summary of all responses along the number of participants. The identity of participants will not be disclosed for the data processing purposes.

b) What is the purpose of processing?

The purpose of this study is:
The goal of the survey and analysis of the responses is to identify the current state of practice related to quality control and quality assurance of all raw materials, ready asphalt mixtures and compacted asphalt pavements, with a specific focus on adherence to moisture damage prevention. Aalto University acts on the behalf of Finnish Transport Infrastructure Agency and INFRA organization in evaluation of the understanding and adherence to the existing specifications and guidelines.

The output of the project is a suggestion on the path for the development of the Finnish Asphalt Specifications in fluctuating market conditions. The point is to identify if the currently existing guidelines and specifications are clear and understandable by both producers and quality controllers.

c) Effects to data subjects
Currently in Finland there is a limited amount of people connected to the asphalt industry, which are connected to the production, quality control and quality assurance of asphalt pavements. This causes a situation in which such persons are easily identifiable by name and position. In order to protect their identity and assure honest feedback towards existing guidelines, this survey identifies participants by name, but anonymizes the responses and overall results.
The list of invited participants contains personal data and may be disclosed in the project to third parties.
The list of the actual participants in the survey is anonymized, thus will not be disclosed anywhere.
If the participant needs assistance with the survey and participates in a face-to-face meeting or skype conversation with the organizer, the identity of the participant is revealed to the survey organizer.
Information related to operation of other companies and other controllers should not be disclosed during the conversation.

d) Legal basis for processing personal data

The processing of personal data is required for the performance of a task carried out in the public interest, namely scientific research and for academic expression.

7. Sharing of Personal Data

Aalto will share research data with a third party Finnish Transportation Infrastructure Agency (FTIA, finnish: Väylävirasto) and INFRA organization.
FTIA identifies the survey subjects by name.
FTIA and INFRA ry will have access to the anonymized results of the survey.

8. Transfer of Personal Data to Non-EU/EEA countries

The university's policy is to take special care when transferring personal data to countries outside of the European Union and the European Economic Area, particularly where those countries do not provide data protection regulation according to the standards set by the GDPR. These transfers of personal data are conducted according to the GDPR utilizing for instance standard contractual clauses or other appropriate safeguards.

9. Storage period of your data and anonymisation

The data related to the invited participants will be stored for 10 years. The results of the survey after anonymization will be disclosed in the report of the project and available as an open source publication. Anonymised data is no longer personal data.

10. The rights of the study participant in a scientific study

According to the General Data Protection Regulation (GDPR), data subjects have the right

- to obtain information on the processing of their personal data
- of access to their data
- to request rectification of their data
- to request restrict the processing of their data
- to object to the processing of their data
- right to erasure, if research data have been unlawfully processed and processing is no longer necessary for archiving purposes in the public interest, scientific research purposes or statistical purposes and erasure of research data will not render impossible or seriously impair objectives of scientific research.

In the event, that the research study does not require, or no longer requires, the identification of a data subject, Aalto University shall not be obliged to acquire additional information in order to identify the data or the data subject for the sole purpose of fulfilling the rights of the data subject. If Aalto University cannot identify the data related to a data subject, the rights of access, rectification, objection and erasure shall not apply. However, if the data subject provides additional information enabling his or her identification and the identification of the research data, the rights are not affected.

Data is being processed for the purposes of scientific research, and the data is not used in decision-making related to the participant.

To exercise his or her rights, the data subject should contact the Data Protection Officer, or the research study contact person.

11. More information on the study and the exercising of your rights

The controller in this study is Aalto University.
The contact person in matters related to the research study:
Michalina Makowska, michalina.makowska@aalto.fi , +358 504318103
The research participant must contact Aalto University's data protection officer if they have questions or demands related to the processing of personal data.
Data Protection Officer Anni Tuomela
Phone number: 0947001
Email: tietosuojavastaava@aalto.fi
If the research participant sees that their data has been processed in violation of the General Data Protection Regulation or data protection legislation, the participant has the right to lodge a complaint with the supervisory authority, the Data Protection Ombudsman (see more: tietosuoja.fi).

*

- ○ Yes, I wish to participate in the study
- ○ No, I do not wish to participate

2. Are you working on the side of *

- contractor /asphalt producer or elsewhere (e.g. laboratory conducting quality control)
- official worker

3. Have you observed surprising damage related to moisture in the constructed roads within the last 5 years? *

- Yes, definetely
- No, definetely
- Not sure, I have no opinion
- Yes, but this is my personal perception

4. How many such cases did you observe in the last 5 years? *

- one
- less than 10
- 10-50
- 50-100
- more than 100 occurrences

5. Is there an increasing trend in the occurrence of moisture related damage within the last 5 years? *

- Yes, definetely
- No, definetely
- Not sure, I have no opinion
- Yes, but this is my personal perception
- Yes, but I think the increase in moisture damage is related to limited repair funds

6. Methods of following the moisture damage criteria in your practice *

	Yes, often	No, never	No, but I know that I can	Yes, sometimes.
Do you use IRI to identify the moisture damage?	○	○	○	○
Do you use rut depth to evaluate the moisture damage?	○	○	○	○

	Yes, often	No, never	No, but I know that I can	Yes, sometimes.
Do you define moisture damaged pavement based on the number of complaint from users?	◯	◯	◯	◯
Do you perform visual evaluation of the road surface from picture database?	◯	◯	◯	◯
Do you perform site visits on the damaged roads?	◯	◯	◯	◯
Do you use surface roughness as a parametyer in evaluation of the moisture damage? (from PTM measurements)	◯	◯	◯	◯
Do you use GPR data to evaluate propensity for moisture damage?	◯	◯	◯	◯
Do you use GPR data to evaluate the moisture damage in pavements?	◯	◯	◯	◯
Do you use transverse profile analysis for evaluation of moisture damage?	◯	◯	◯	◯

7. Have you observed the deviation in the quality of asphalt concrete pavements within the span of the last 5 years? *

- ◯ Yes, their resistance to wear due to traffic and weather conditions is lower
- ◯ No, the maintenance frequency has not increased
- ◯ Yes, but the issue is related to decreased funding for repair and accumulation of damages

8. Reasons behind decreased quality of asphalt pavements. *

	Definetely no	No, I don't think so	Maybe	Yes, perhaps	Yes, defintely
Was the decrease in quality caused by a change of contractor, but not the mixture design?	◯	◯	◯	◯	◯
Was the decrease in quality caused by a change of mix design but not the contractor?	◯	◯	◯	◯	◯
Was the change in quality caused by a change in aggregate type/source?	◯	◯	◯	◯	◯
Was the change in quality caused by a change in bitumen source?	◯	◯	◯	◯	◯
Was the change in quality caused by a change in bitumen grade?	◯	◯	◯	◯	◯
Was the change in quality caused by environmental factors, such as temeprature and precipitation?	◯	◯	◯	◯	◯

	Definetely no	No, I don't think so	Maybe	Yes, perhaps	Yes, defintely
Was the change in quality more related to the increased air voids content and hidnered compactibility?	◯	◯	◯	◯	◯
	◯	◯	◯	◯	◯

9. Do you receive the documentation related to type testing of AC mixtures, especially in the context of water sensitivity (eN 12697-12 method A)? (the actual test results of Indirect Tensile strength of dry and wet samples, not the "ITSR>80%") *

◯ Yes, I do

◯ No, I do not

10. Do you know what is the frequency of testing for water sensitivity (12967-12 method A) by the producer of the ordered AC mixtures? *

◯ Yes, I do precisely

◯ No, I do not.

◯ I trust CE marking and the frequency should be defined in standard EN 13108-21

11. Do you receive the documentation related to type testing of SMA mixtures, especially in the context of water sensitivity? (the actual test results of Indirect Tensile strength of dry and wet samples, not the "ITSR>80%") *

◯ Yes, I do

◯ No, I do not

12. Do you know what is the frequency of testing for water sensitivity (12697-12 method A) by the producer of the ordered SMA mixtures? *

◯ Yes, I do precisely

◯ No, I do not.

◯ I trust CE marking and the frequency should be defined in standard EN 13108-21

13.Do you receive results from the aggregate tests, in which it is written that they do not contain minerals badly influencing moisture resistance (e.g. results of the thin section tests PANK 2303 or petrographic analysis SFS-EN 12407)?

◯ Yes, always

◯ Yes, usually

◯ Sometimes

- Never

14. Do you receive the the results from the aggregate used in PAB-V or from the mixture on the basis of which the adhesion promotor amount is determined (e.g. PANK 2108, PANK 2401 or EN 12697-12, method C)?

- Yes, always
- Yes, usually
- Sometimes
- Never

15. Do you receive the documentation related to type testing of SA (PAB) mixtures, especially in the context of water sensitivity? (the actual test results of Indirect Tensile strength of dry and wet samples, not the "ITSR>60%") *

- Yes, I do
- No, I do not

16. Do you receive reports on water sensitivity testing during the production of PAB mixtures? (MYR [12697-12 method C] or sankokoe) *

- Yes, I do
- No, I do not

17. Is the ITSR test (12697-12 method A) on PAB-V aggregate in AA gradation and bitumen 70/100 enough of a proof for water damage resistance of PAB-V according to the Finnish Asphalt Specifications 2017? *

- Yes, it is
- No, it is not

18. Do you require PAB-V aggregate and V1500 binder to be tested for compatibility? *

- Yes, I do
- No, I do not

19. Do you know what is the frequency of testing for the water sensitivity by the producer of PAB mixtures? *

- Yes, I do
- No, I do not

- No, but I know what it should be

20. Is the slag aggregate for use in road construction and pavement required to be tested for volume expansion? *

- Yes, it is defined in Finnish Asphalt Specifications 2017
- No, it is not

21. Is the producer reporting the volume expansion of slag aggregate? *

- Yes, they do
- No, they do not

22. Do you know with what frequency does the producer of slag tests the volume expansion of their product? *

- Yes, I do
- No, I do not

23. Do you perform quality assurance on the aggregate related to performance indicators (water absorption, freeze-thaw resistance, density, flakiness index) after the delivery to the asphalt production plant? *

- Yes, with fixed frequency
- Yes, ocassionally
- No, never
- Yes, but not water absorbance and freeze-thawing resistance

24. How often do you perform the quality assurance of the aggregate performance markers delivered to the production plant?

	Nordic Ballmill abrassion	Water absorption	Freeze-thaw resistance
Never	☐	☐	☐
1 per year	☐	☐	☐
With change of supplier	☐	☐	☐
With change of stockpile	☐	☐	☐
Every 2000 tons of raw material	☐	☐	☐

	Nordic Ballmill abrassion	Water absorption	Freeze-thaw resistance
Upon deviation in gradation beyond that defined in EN 13108-21	☐	☐	☐
With reported change in one the other parameters by the provider, e.g. Flakiness index	☐	☐	☐
With organoleptically determined change in the raw material, e.g. color of aggregate change from black to red	☐	☐	☐

25. Is the slag aggregate for use in road construction and pavement required to be tested for volume expansion? *

- ◯ Yes, it is defined in Finnish Asphalt Specifications 2017
- ◯ No, it is not

26. Is the producer reporting the volume expansion of slag aggregate? *

- ◯ Yes, they do
- ◯ No, they do not

27. Do you know with what frequency does the producer of slag tests the volume expansion of their product? *

- ◯ Yes, I do
- ◯ No, I do not

28. Do you perform the quality assurance of volume expansion of the slag aggregate to test if it meets the requirement? *

- ◯ Yes, I do
- ◯ No, I do not

29. Do you assure the quality of the filler by testing its surface area 1 per year? *

- ◯ Yes, I do
- ◯ No, I do not. This is the responsibility of the provider

30. Do you assure the quality of aggregate by performing those specific tests *

	Yes, with fixed frequency	Yes, ocassionally	No, never	Yes, when requested by ELY
Water absorption according to 1097-6 of aggregate 8/16	◯	◯	◯	◯
Water absorption according to 1097-6 of aggregate 4/8 mm sieve	◯	◯	◯	◯
Water absorption according to 1097-6 of aggregate bigger than 16 mm	◯	◯	◯	◯
Do you perform the freeze-thawing resistance test on aggregates for which the water absorption is above 1%? (WA24>1%)	◯	◯	◯	◯

31. How do you assure, that the aggregate used for the construction in the asphalt concrete mixtures production does not contain too much of the minerals negatively affecting resistance to moisture damage?

◯ The aggregate producer delivered documents of thin section test results (PANK 2302)
◯ The aggregate producer delivered petrographic analysis test results (SFS-EN 12407)
◯ On the basis of the documents delivered during production of aggregate by their producer (SFS-EN 932-2)
◯ Based on the geologist’s statement
◯ I trust CE-marking

32. Do you perform the tests (mentioned in question 31) in which you assure that aggregate does not contain the minerals influencing negatively the moisture damage resistance?

◯ Yes
◯ No

33. Which information do you use when choosing the amount of adhesion promotor during design of the PAB-V mixtures?

◯ Aggregate is tested by us using the adhesion properties methodology PANK 2108 or/and 2401
◯ Aggregate is tested by using the adhesion properties methodology PANK 2108 or/and 2401, when PAB-V is designed using aggregate with which we do not have previous experience
◯ Using method described in EN 12697-12 (method C) always when designing mixture
◯ Using method described in EN 12697-12 (method C), when PAB-V is designed using aggregate with which we do not have previous experience
◯ We do not test anymore the aggregates which were used previously in PAB-V mixtures
◯ Adhesion promotor amount is chosen based on other knowledge than the above mentioned test results

34. How often do you test for rheological properties of the bidner (e.g. Penetration, Softening Point, Viscosity)? *

☐ With every new binder batch (tank)

- [ ] Once per year
- [ ] Less than every 2000 tons
- [ ] When the change in the origin of the binder is observed
- [ ] When the smell indicates the material deviating from the usual
- [ ] I use the producer reported values in my own quality control of the process

35. Have you observed the change in the bitumen quality/type/smell in the recent 3 years? *

- ◯ Yes, it is different
- ◯ No, it does not differ from the product before
- ◯ Yes, but I am using multiple providers of binder
- ◯ Yes, but it happens ocassionally that the product is different

36. Have you observed the change in the bitumen quality/type/smell in the recent 3 years? *

- ◯ Yes, it is different
- ◯ No, it does not differ from the product before
- ◯ Yes, but I am using multiple providers of binder
- ◯ Yes, but it happens ocassionally that the product is different

37. Have you observed the change in the bitumen quality/type/smell in the recent 3 years? *

- ◯ Yes, it is different
- ◯ No, it does not differ from the product before
- ◯ Yes, but I am using multiple providers of binder
- ◯ Yes, but it happens ocassionally that the product is different

38. Do you use any spectroscopic techniques to differentiate between the binders? *

- ◯ Yes, I do
- ◯ No, I do not

39. The standard 13108-21 suggests organoleptic investigation of additives. Do you perform it for adhesion promoters? *

- ◯ Yes, I use smell and sight to evaluate consistency

- ◯ Yes, I use smell only to evaluate consistency
- ◯ No, the smell is irritating for staff
- ◯ No, but we perform the quality assurance in other way
- ◯ Yes, I use sight only to evaluate consistency

40. How often do you perform the moisture resistance testing according to the 12697-12 method A *

- ◯ At mix formulation only
- ◯ 1 per year
- ◯ 1 per 2 year
- ◯ 1 per 3 year
- ◯ 1 per 4 year
- ◯ 1 per 5 year

41. Questions related to the procedure known as ITSR test (EN 12697-12 method A) *

	Yes, I do	No, I do not
Do you use freeze-thawing cycling procedure (PANK 4306) in your type testing for AC mixtures?	◯	◯
Do you use freeze-thawing cycling procedure (PANK 4306) during the mix design of AC mixtures?	◯	◯
Do you use freeze-thawing cycling procedure (PANK 4306) in your type testing for SMA mixtures?	◯	◯
Do you use freeze-thawing cycling procedure (PANK 4306) during the mix design of SMA mixtures?	◯	◯
Do you control the height difference between the specimen in dry and wet subsets to be within 5 mm?	◯	◯
When comparing two AC mixtures, e.g. using different aggregates, do you use the same height of the specimens in both cases?	◯	◯
Is the height of the specimen related to the maximum density of the tested mixture?	◯	◯
Do you control the water saturation level in the wet sample set?	◯	◯

	Yes, I do	No, I do not
Do you control that the binder content between dry and wet set are within set range?	○	○
Do you test the ITS at the same air void content in all your testing activities? (6.2.3a)	○	○
Do you compact the sampels using fixed number of blows/gyrations/etc. ? (6.2.3.b)	○	○

42. Sample preparation method for the use in ITSR tests *

	For use inside Finland	For use outside of Finland	For type testing	For both in and outside of Finland
Are the 100 mm samples prepared by the gyratory compactor?	○	○	○	○
Are the 150 mm samples prepared by the gyratory compactor?	○	○	○	○
Are the 100 mm samples prepared by the impact compactor (Marshal)?	○	○	○	○
Are the 150 mm samples prepared by the impact compactor (Marshal)?	○	○	○	○
Are the 100 mm samples prepared by the vibratory compaction?	○	○	○	○
Are the 150 mm samples prepared by the vibratory compaction?	○	○	○	○
Are the 100 mm samples prepared by the roller compactor?	○	○	○	○
Are the 150 mm samples prepared by the roller compactor?	○	○	○	○

43. Other questions related to the test EN 12697-12 method A (ITSR) *

	Yes, I do	No, I do not
Do you have problems with repeatability of the test?	○	○
Do you have problems with reproducibility of the test?	○	○
Do you have problems with meeting the requirements set in Finnish Asphalt Specifications (FAS) to ITSR test using the empirical designs provided in FAS 2017?	○	○

	Yes, I do	No, I do not
Do you sometimes use the adhesion promoters in AC mixtures with ITSR test?	○	○
Do you sometimes use the adhesion promoters in SMA mixtures with ITSR test?	○	○
Do you sometimes use the adhesion promoters in SMA mixtures with ITSR test?	○	○
Have you seen a significant positive influence of the adhesion promoter on the ITSR result?	○	○

44. How often do you perform water sensitivity testing according to 12697-12 method A for AC mixtures? *

- ☐ At mix formulation /design
- ☐ 1 per year scheduled
- ☐ 1 per 2 year scheduled
- ☐ 1 per 3 year scheduled
- ☐ 1 per 4 year scheduled
- ☐ 1 per 5 year scheduled
- ☐ At the change of aggregate source
- ☐ At deviation of aggregate parameter from stated in the original type testing report (e.g. change in flakiness index, gradation, water absorption)
- ☐ At every change of filler source
- ☐ At every change of bitumen source
- ☐ At every change of bitumen grade
- ☐ Every 1000 tons of bitumen of less
- ☐ Every 2000 tons of bitumen
- ☐ At every RAP source change, when RAP below 10% in mixture
- ☐ At every change of RAP content by 10-20% from reported in original type testing report
- ☐ At every RAP source change, when RAP below 50% in mixture

45. How often do you perform water sensitivity testing according to 12697-12 method A for SMA mixtures? *

- ☐ At mix formulation /design
- ☐ 1 per year scheduled

- [ ] 1 per 2 year scheduled
- [ ] 1 per 3 year scheduled
- [ ] 1 per 4 year scheduled
- [ ] 1 per 5 year scheduled
- [ ] At the change of aggregate source
- [ ] At deviation of aggregate parameter from stated in the original type testing report (e.g. change in flakiness index, gradation, water absorption)
- [ ] At every change of filler source
- [ ] At every change of bitumen source
- [ ] At every change of bitumen grade
- [ ] Every 1000 tons of bitumen of less
- [ ] Every 2000 tons of bitumen

46. How often do you perform water sensitivity testing according to 12697-12 method A for SA (PAB) mixtures using AA gradation and bitumen 70/100? *

- [ ] At mix formulation /design
- [ ] 1 per year scheduled
- [ ] 1 per 2 year scheduled
- [ ] 1 per 3 year scheduled
- [ ] 1 per 4 year scheduled
- [ ] 1 per 5 year scheduled
- [ ] At the change of aggregate source
- [ ] At deviation of aggregate parameter from stated in the original type testing report (e.g. change in flakiness index, gradation, water absorption)
- [ ] At every change of filler source
- [ ] At every change of bitumen source
- [ ] At every change of bitumen grade
- [ ] Every 1000 tons of bitumen or less
- [ ] Every 2000 tons of bitumen
- [ ] At every change of adhesion promotor type
- [ ] At every change of adhesion promotor amount

47. How often do you perform water sensitivity testing according to 12697-12 method C for SA (PAB)? *

- ☐ At mix formulation /design
- ☐ 1 per year scheduled
- ☐ 1 per 2 year scheduled
- ☐ 1 per 3 year scheduled
- ☐ 1 per 4 year scheduled
- ☐ 1 per 5 year scheduled
- ☐ At the change of aggregate source
- ☐ At deviation of aggregate parameter from stated in the original type testing report (e.g. change in flakiness index, gradation, water absorption)
- ☐ At every change of filler source
- ☐ At every change of bitumen source
- ☐ At every change of bitumen grade
- ☐ Every 1000 tons of bitumen or less
- ☐ Every 2000 tons of bitumen
- ☐ At every change of adhesion promotor type
- ☐ At every change of adhesion promotor amount
- ☐ Every 500 tons of asphalt mixture produced
- ☐ Every 1000 tons of asphalt mixture produced
- ☐ Every 2000 tons of asphalt mixture produced

48. Do you perform the bucket test (sankokoe) during production? *

- ○ Yes, I do
- ○ No, I do not

49. Do you report the results of the bucket test (sankokoe)? *

- ○ Yes, I do
- ○ No, they are for internal production evaluation

50. Which adhesion promotors and other asphalt modifiers are you familiar with(used in the past or have been in contact with producers)? If you do not know the chemical composition of the adhesion promoter, but you know just the brand or name of the product, please list it in "other". The goal of this question is to provide input into the future research design. You may skip the question if you consider it a sensitive information.
This question is voluntary.

Amine based, name brands: [ ]

polyphosphoric acid based, name brands: [ ]

calcium hydroxide, name brands/producers: [ ]

organosilane based, name brands/producers: [ ]

waxes, name brands/producers: [ ]

zeolites, name brands/producers: [ ]

polymer modifiers, name brands/producers: [ ]

rejuvenators, name brands/producers: [ ]

crumb rubber, name brands/producers: [ ]

polymerous fibers, name brands/producers: [ ]

other, name brands/product names [ ]

51. Which of the below bitumen suppliers have been providing the bitumen for you? The goal of this question is to provide input into the future research design. You may skip the question if you consider it a sensitive information.

- [ ] Nynas
- [ ] Total Bitumen
- [ ] Lotos Asfalt
- [ ] PKN Orlen
- [ ] Other [ ]

52. Participation Consent Project BITU2020

I have understood that participation is voluntary and at any point in the research study, I am at liberty to notify that I no longer wish to participate in the study, but all the information gathered up until that point is can be used as described in the privacy notice of the research study.
I have received sufficient information about the research study, I have had the possibility to have my questions answered, I have understood the information and I wish to participate in the research study.
Contact details: Michalina Makowska, +358504318103, michalina.makowska@aalto.fi Aalto-University *

- ( ) Yes, I wish to participate in the study
- ( ) No, I do not wish to participate

Project BITU 2020
Basic report
Project BITU 2020

Näytetään 12 vastaajaa kyselyn vastaajien kokonaismäärästä 12

1. **Tietoa tutkimukseen osallistuvalle** Project BITU2020Sinut on kutsuttu osallistumaan tutkimukseen.Osallistuminen tutkimukseen on vapaaehtoista. Voit keskeyttää osallistumisesi milloin tahansa tutkimuksen aikana. Jos vetäydyt tutkimuksesta, sinuun ei kohdistu mitään negatiivista seurausta, mutta sinulta siihen asti kerättyä aineistoa voidaan käyttää tutkimukseen soveltuvan tietosuojalainsäädännön mukaisesti. Tietosuojailmoitus Project BITU 2020
Tutkimuksen aihe, tavoite ja mahdollinen rahoittaja: The project focuses on reviewing the changes in refining industry due to the IMO2020 regulations and their impact on the bitumen supply. At the core focus of the project is an increase in understanding of the possible fluctuations on the bitumen market, potential changes to bitumen composition, as well as the impact those regulations may have on the performance of the asphalt pavements past the transition time of 2020-2022.Within the project, a special focus is added to the differentiation between bitumen and bituminous mixtures composition. The link between the chemical composition of bituminous mixtures and their performance, especially in terms of resistance to moisture damage and low-temperature inflicted damage, will be evaluated. The project reviews the current state of the practice in Finland in relation to the prevention of moisture damage in asphalt pavements. Project is co-sponsored by the Finish Transport Infrastructure Agency and INFRA organization.
Konkreettinen henkilötietojen keräämisen metodi; esimerkiksi henkilötiedot on kerätty haastatteluja videoimalla tai viranomaisten rekistereistä, kuinka paljon tutkimuksen osallistuvan aikaa tarvitaan tutkimukseen osallistumiseen, missä tutkimus suoritetaan:
The survey is conducted through a questionnaire, which can be assisted by the conversation with the organizer. The conversation is to happen via skype or in-person and will be recorded and stored as research data. The time required for answering: about 15-30 minutes.
Muu mahdollinen: The answers to the survey questions will comprise a material used during the development of the future Finnish Asphalt Specifications and production guidelines, as well as the input into future research needs statement. The goal is to gather honest answers, which will either lead to the clarification of the guidelines or further development of thereof.
Mitä henkilötietoja käsitellään tutkimuksessa?The personal data processed in the research project will include name of the participant, their e-mail address, skype ID, profession and job title, as well as date of the conversation.
Henkilötiedot on kerätty seuraavista lähteistä: The participants in the survey are identified by the Finnish Transport Infrastructure Agency (FTIA) as collaborators, producers and quality controllers on behalf of the FTIA within the last 5 years. This data is vested in the representative of Aalto University responsible for organization of the survey. The FTIA shares the contact details to the contact person within the organization. The survey may be forwarded inside the organization to the person directly responsible for the activities covered in the survey. The information about the identity of the responsible person comes from the original contact person.
a) Miten ja millä perusteella tutkimuksessa käsitellään henkilötietojasi?
The concrete method by with personal data is collected and analysed.The participants of the survey are suggested by the FTIA and their identity is known. In the case of necessary assistance or question to survey organizer, the skype calls and conversations will be recorded. It is advised not to disclose the information which may allow matching of answers with the survey participant. The link to the survey is personal and send to the e-mail account of the participant. The survey is anonymous, but differentiates the subjects into two groups, namely quality controllers and producers. The results of the survey will be published in the form of summary of all responses along the number of participants. The identity of participants will not be disclosed for the data processing purposes.
b) Tutkimuksen tarkoituksena on:
The goal of the survey and analysis of the responses is to identify the current state of practice related to quality control and quality assurance of all raw materials, ready asphalt mixtures and compacted asphalt pavements, with a specific focus on adherence to moisture damage prevention. Aalto University acts on the behalf of Finnish Transport Infrastructure Agency and INFRA organization in evaluation of the understanding and adherence to the existing specifications and guidelines. The output of the project is a suggestion on the path for the development of the Finnish Asphalt Specifications in fluctuating market conditions. The point is to identify if

the currently existing guidelines and specifications are clear and understandable by both producers and quality controllers.

c) Vaikutukset tutkimukseen osallistuville ja käsittelyperuste

Currently, in Finland, there is a limited amount of people connected to the asphalt industry, which are connected to the production, quality control and quality assurance of asphalt pavements. This causes a situation in which such persons are easily identifiable by name and position. In order to protect their identity and assure honest feedback towards existing guidelines, this survey identifies participants by name but anonymizes the responses and overall results.The list of invited participants contains personal data and may be disclosed in the project to third parties. The list of the actual participants in the survey is anonymized, thus will not be disclosed anywhere. If the participant needs assistance with the survey and participates in a face-to-face meeting or skype conversation with the organizer, the identity of the participant is revealed to the survey organizer. Information related to the operation of other companies and other controllers should not be disclosed during the conversation.

d) Legal basis for processing personal data

The processing of personal data is required for the performance of a task carried out in the public interest, namely scientific research and for academic expression.

Henkilötietojen jakaminen

Aallon saattaa olla tarpeen jakaa tutkimusdataa kolmannelle osapuolelle. Mahdollinen datan jakaminen suoritetaan yksityisyydensuojaa koskevaa lainsäädäntöä noudattaen. Aalto will share research data with a third party Finnish Transportation Infrastructure Agency (FTIA, finnish: Väylävirasto) and INFRA organization.FTIA identifies the survey subjects by name. FTIA and INFRA ry will have access to the anonymized results of the survey. Aalto huolehtii yksilöiden oikeuksien ja yksityisyyden suojaamisesta kolmansien osapuolten kanssa tehtävien sopimusten avulla.

Henkilötietojen siirto EU:n ja ETA:n ulkopuolisiin valtioihinYliopiston politiikka on noudattaa erityistä huolellisuutta, kun henkilötietoja siirretään Euroopan unionin ja Euroopan Talousalueen ulkopuolelle. Erityisesti, jos kyseisten maiden tietoturva ei täytä yleisen tietosuoja-asetuksen mukaisia vaatimuksia. Tällaiset henkilötietojen siirrot toteutetaan yleisen tietosuoja-asetuksen mukaisesti, esimerkiksi hyödyntämällä mallisopimuslausekkeita tai muita asianmukaisia suojakeinoja.

Säilytys ja anonymisointi

The data related to the invited participants will be stored for 10 years. The results of the survey after anonymization will be disclosed in the report of the project and available as an open source publication. Anonymisoitu data ei enää katsottavissa henkilötiedoiksi. Tutkimukseen osallistuvan oikeudet Yleisen tietosuoja-asetuksen (GDPR) mukaan rekisteröidyllä on oikeus: saada tietoa henkilötietojen käsittelystä, tarkastaa itseään koskevat tiedot, pyytää tietojensa oikaisua, pyytää tietojensa käsittelyn rajoittamista, vastustaa tietojensa käsittelyä.

Oikeus tietojen poistamiseen, jos tutkimusdataa on käsitelty laittomasti ja käsittely ei ole enää tarpeen yleisen edun mukaisten tavoitteiden saavuttamisessa, tieteellisen tutkimuksen tavoitteiden tai tilastollisten tavoitteiden täyttämiseksi, ja tutkimusdatan poistaminen ei tee tieteellisen tutkimuksen päämääriä mahdottomiksi tai aiheuta vakavaa haittaa tieteelliselle tutkimukselle. Siinä tapauksessa, että tutkimuksen tekeminen ei edellytä tai ei enää edellytä rekisteröidyn tunnistamista, Aalto-yliopisto ei ole velvoitettu hankkimaan lisää tietoja, jotta data tai rekisteröity voitaisiin tunnistaa vain sen vuoksi, että rekisteröity voisi käyttää oikeuksiaan. Jos Aalto-yliopisto ei tunnista dataa, joka on liitettävissä tiettyyn rekisteröityyn, niin rekisteröidyllä ei ole oikeutta tietojen tarkastamiseen, tietojen korjaamiseen, tietojen käsittelyn vastustamiseen eikä tietojen poistamiseen. Jos rekisteröity kuitenkin antaa lisätietoja, joiden avulla hänet voidaan tunnistaa tutkimusdatasta, niin oikeuksia ei rajoiteta. Dataa käytetään tieteellisen tutkimuksen tarkoituksiin, eikä sitä käytetä tutkimukseen osallistuvaan liittyvään päätöksentekoon.Rekisteröity voi käyttää oikeuksiaan ottamalla yhteyttä tutkimuksen yhteyshenkilöön tai tietosuojavastaavaan.

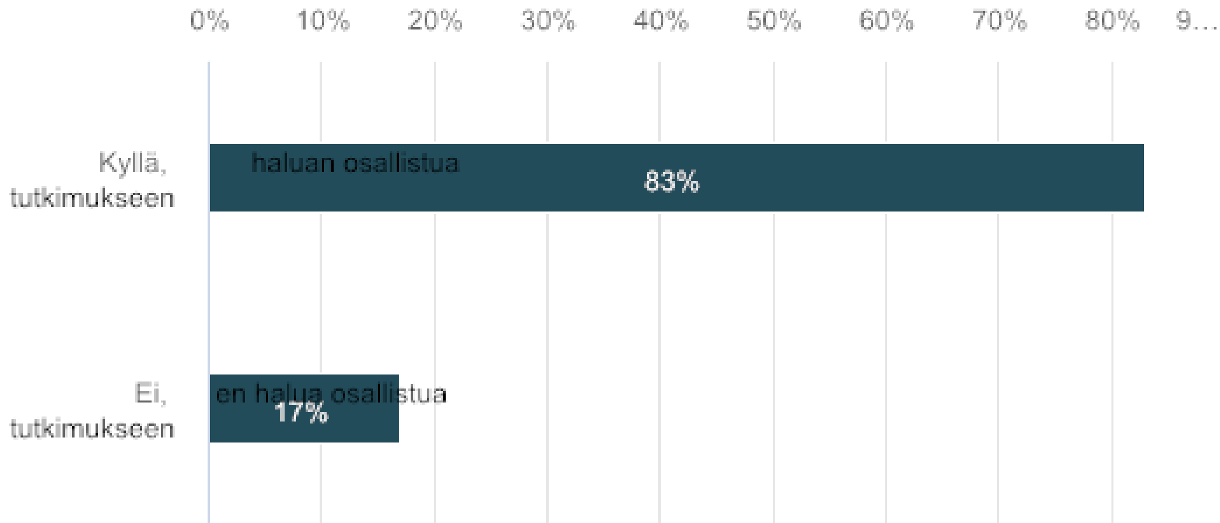
Lisätietoa tutkimuksesta ja oikeuksien käyttämisestä

Tässä tutkimuksessa rekisterinpitäjänä on Aalto-yliopisto. Yhteyshenkilö tutkimusta koskevissa asioissa on:Michalina Makowska, michalina.makowska (at) aalto.fi , +358 504318103

Tutkimukseen osallistuvan tulee ottaa yhteyttä Aalto-yliopiston tietosuojavastaavaan, jos hänellä on kysymyksiä tai vaatimuksia henkilötietojen käsittelyn osalta:Tietosuojavastaava:Anni Tuomela Puhelinnumero: 09 47001Sähköposti: tietosuojavastaava (at) aalto.fi

Jos tutkimukseen osallistuva kokee, että hänen henkilötietojaan on käsitelty tietosuojalainsäädännön vastaisesti, on osallistujalla oikeus tehdä valitus valvontaviranomaiselle, tietosuojavaltuutetulle (lue lisää: http://www.tietosuoja.fi).

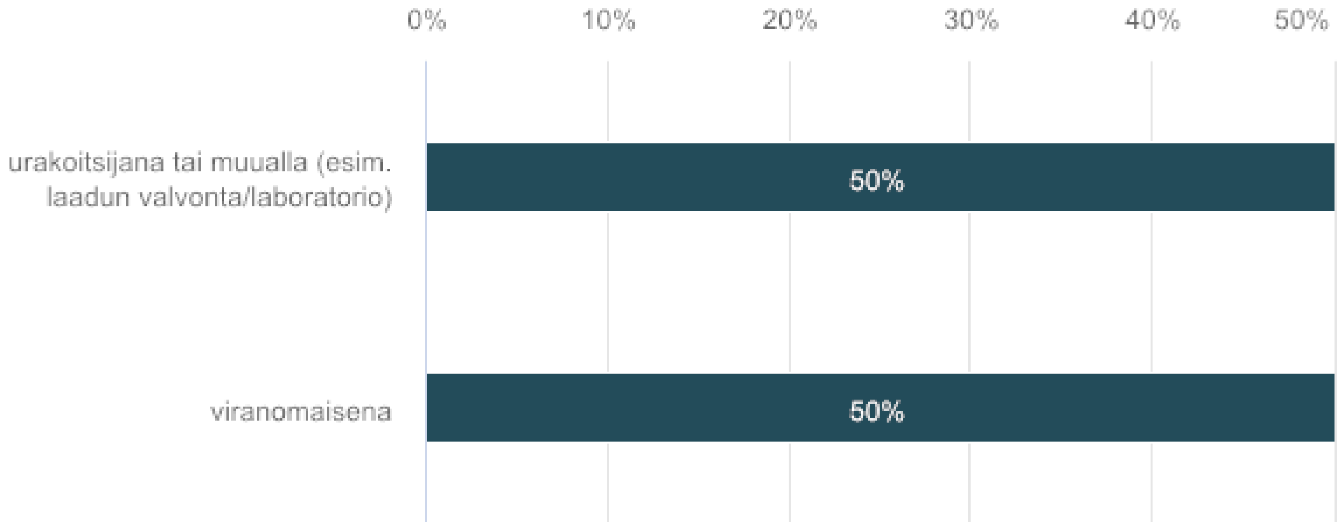
Vastaajien määrä: 12



	n	Prosentti
Kyllä, haluan osallistua tutkimukseen	10	83.33%
Ei, en halua osallistua tutkimukseen	2	16.67%

2. Työskenteletkö

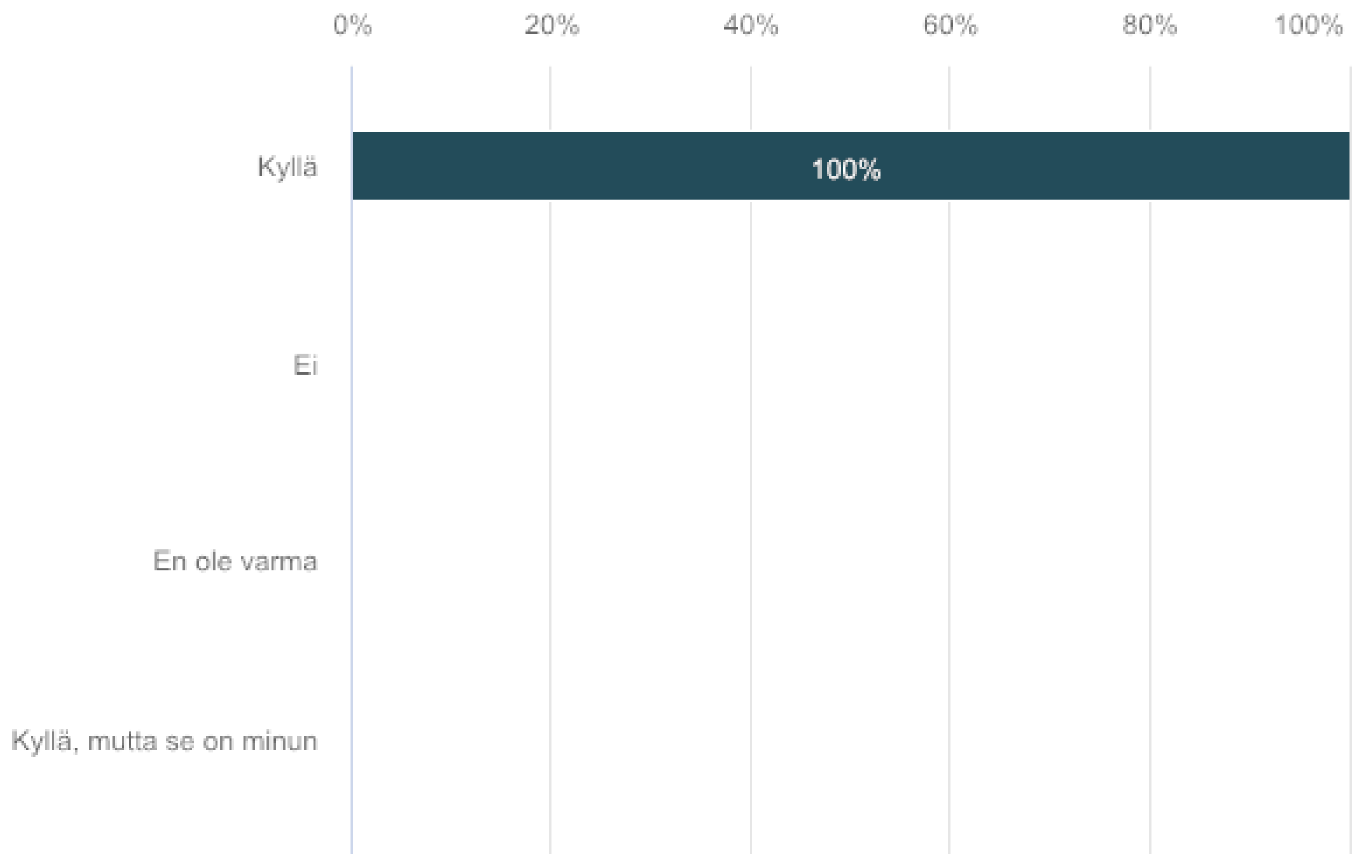
Vastaajien määrä: 10



	n	Prosentti
urakoitsijana tai muualla (esim. laadun valvonta/laboratorio)	5	50%
viranomaisena	5	50%

3. Oletko havainnut rakennettujen teiden yllättäviä kosteuteen/vesiin liittyviä vaurioita (alempana vedenkestävyys) viimeisen 5 vuoden aikana?

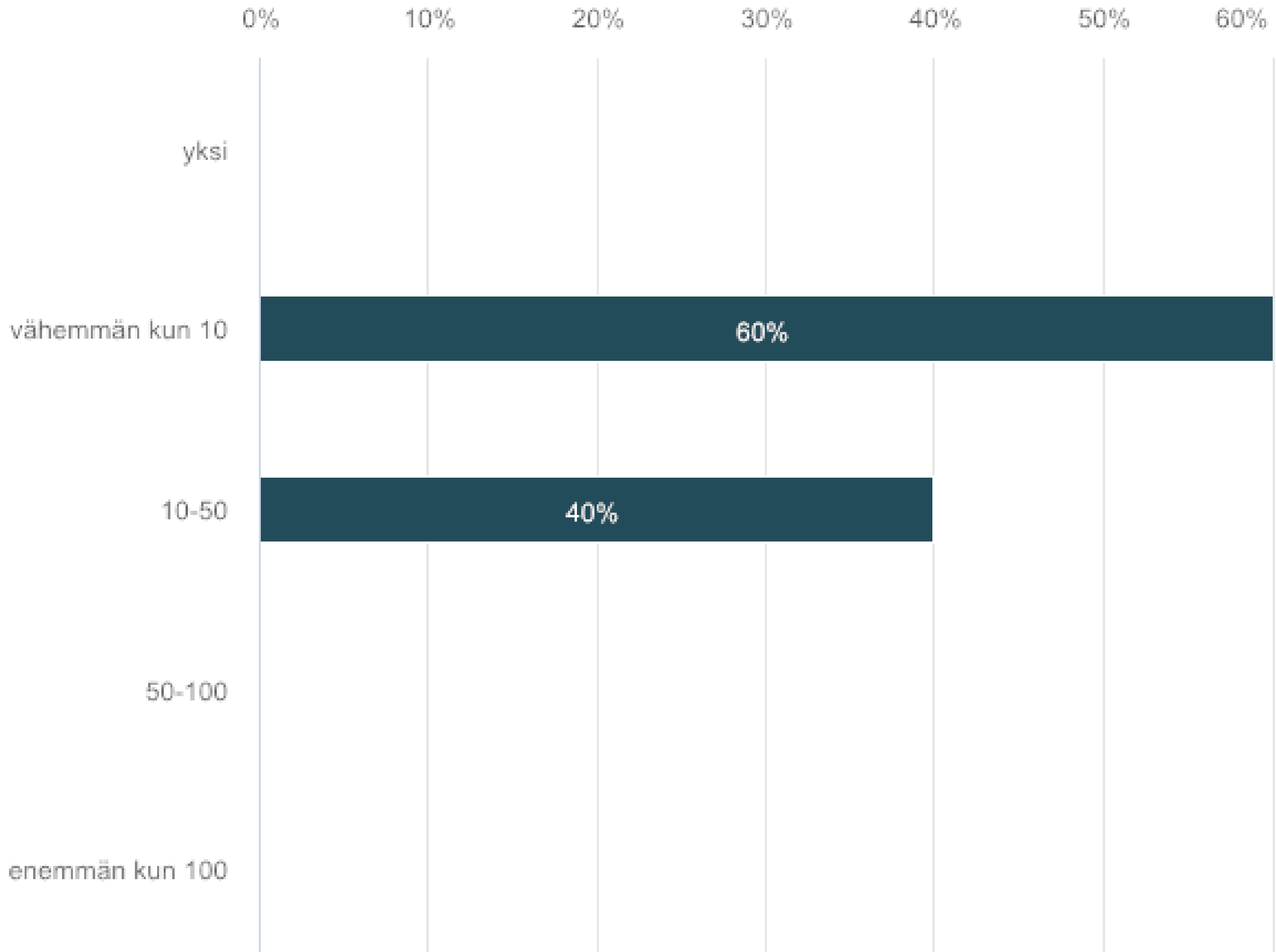
Vastaajien määrä: 5



	n	Prosentti
Kyllä	5	100%
Ei	0	0%
En ole varma	0	0%
Kyllä, mutta se on minun mielipide	0	0%

4. Kuinka monta tällaista tapausta olet havainnut viimeisen 5 vuoden aikana?

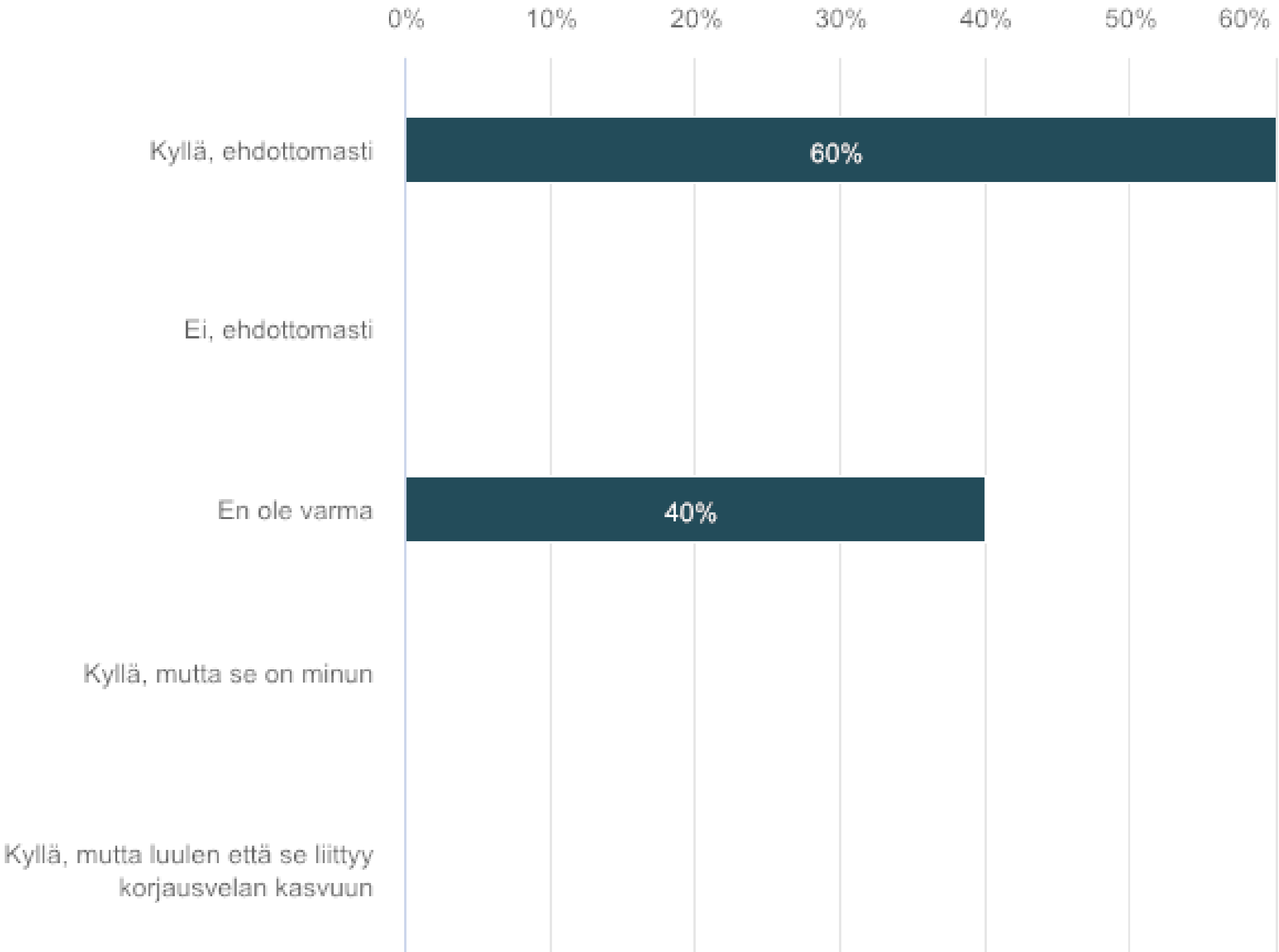
Vastaajien määrä: 5



	n	Prosentti
yksi	0	0%
vähemmän kun 10	3	60%
10-50	2	40%
50-100	0	0%
enemmän kun 100	0	0%

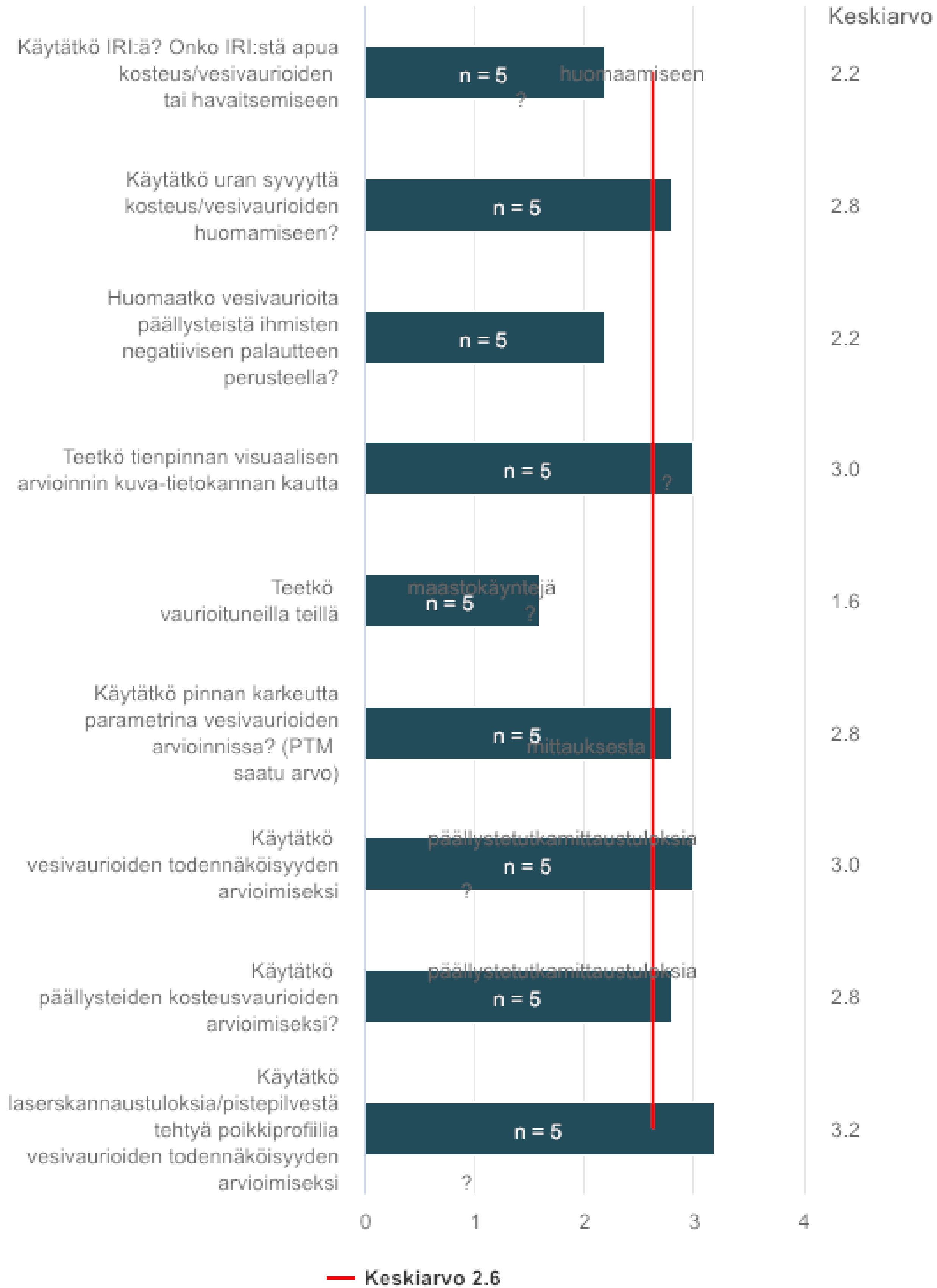
5. Oletko huomannut kosteuteen/veteen liittyvien vahinkojen lisääntyneen viimeisen viiden vuoden aikana?

Vastaajien määrä: 5



	n	Prosentti
Kyllä, ehdottomasti	3	60%
Ei, ehdottomasti	0	0%
En ole varma	2	40%
Kyllä, mutta se on minun mielipide	0	0%
Kyllä, mutta luulen että se liittyy korjausvelan kasvuun	0	0%

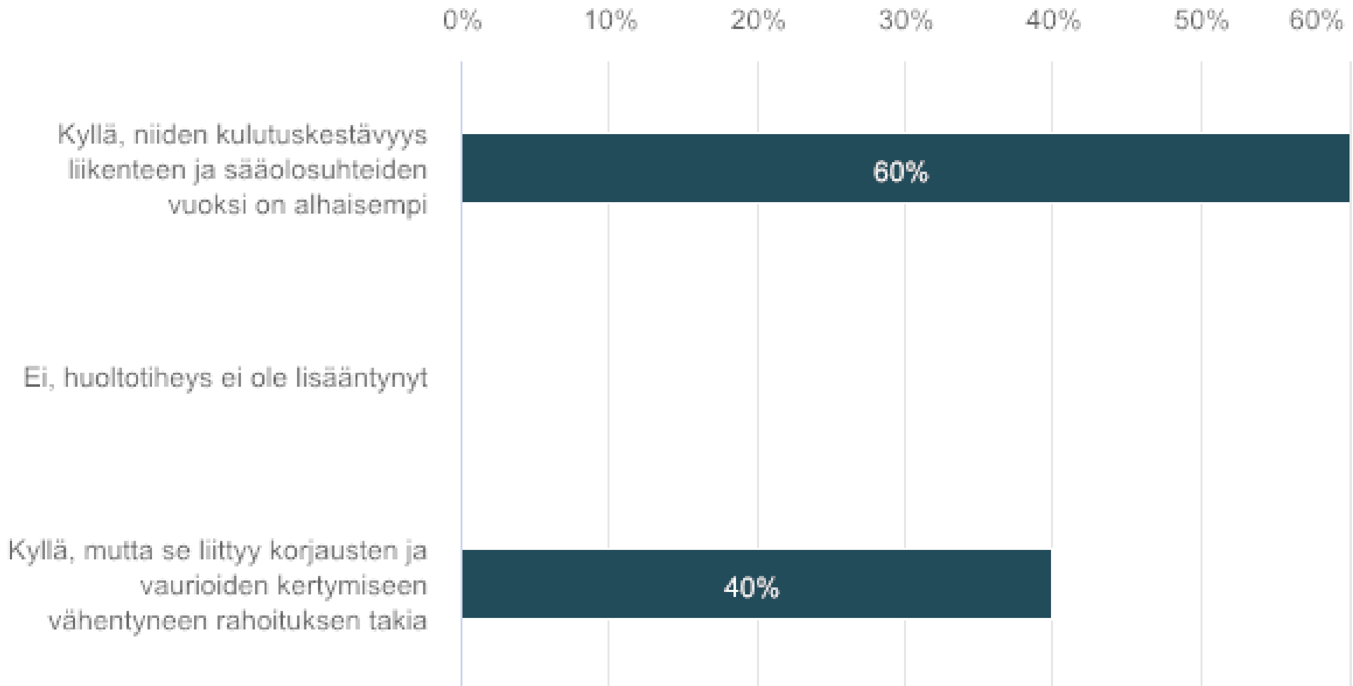
6. Menetelmät vesivauriokriteerien arvioimiseksi joita käytät
Vastaajien määrä: 5



	Kyllä, usein	Ei ikinä	Ei, mutta luulen että se on mahdollinen	Kyllä, joskus	Keskiarvo	Mediaani
Käytätkö IRI:ä? Onko IRI:stä apua kosteus/vesivaurioiden huomaamiseen tai havaitsemiseen?	0%	80%	20%	0%	2.2	2
Käytätkö uran syvyyttä kosteus/vesivaurioiden huomamiseen?	0%	40%	40%	20%	2.8	3
Huomaatko vesivaurioita päällysteistä ihmisten negatiivisen palautteen perusteella?	60%	0%	0%	40%	2.2	1
Teetkö tienpinnan visuaalisen arvioinnin kuva-tietokannan kautta?	20%	20%	0%	60%	3	4
Teetkö maastokäyntejä vaurioituneilla teillä ?	80%	0%	0%	20%	1.6	1
Käytätkö pinnan karkeutta parametrina vesivaurioiden arvioinnissa? (PTM mittauksesta saatu arvo)	0%	40%	40%	20%	2.8	3
Käytätkö päällystetutkamittaustuloksia vesivaurioiden todennäköisyyden arvioimiseksi?	0%	40%	20%	40%	3	3
Käytätkö päällystetutkamittaustuloksia päällysteiden kosteusvaurioiden arvioimiseksi?	0%	40%	40%	20%	2.8	3
Käytätkö laserskannaustuloksia/pistepilvestä tehtyä poikkiprofiilia vesivaurioiden todennäköisyyden arvioimiseksi?	0%	20%	40%	40%	3.2	3

7. Oletko havainnut asfalttipäällysteiden laadun poikkemia viimeisen 5 vuoden aikana?

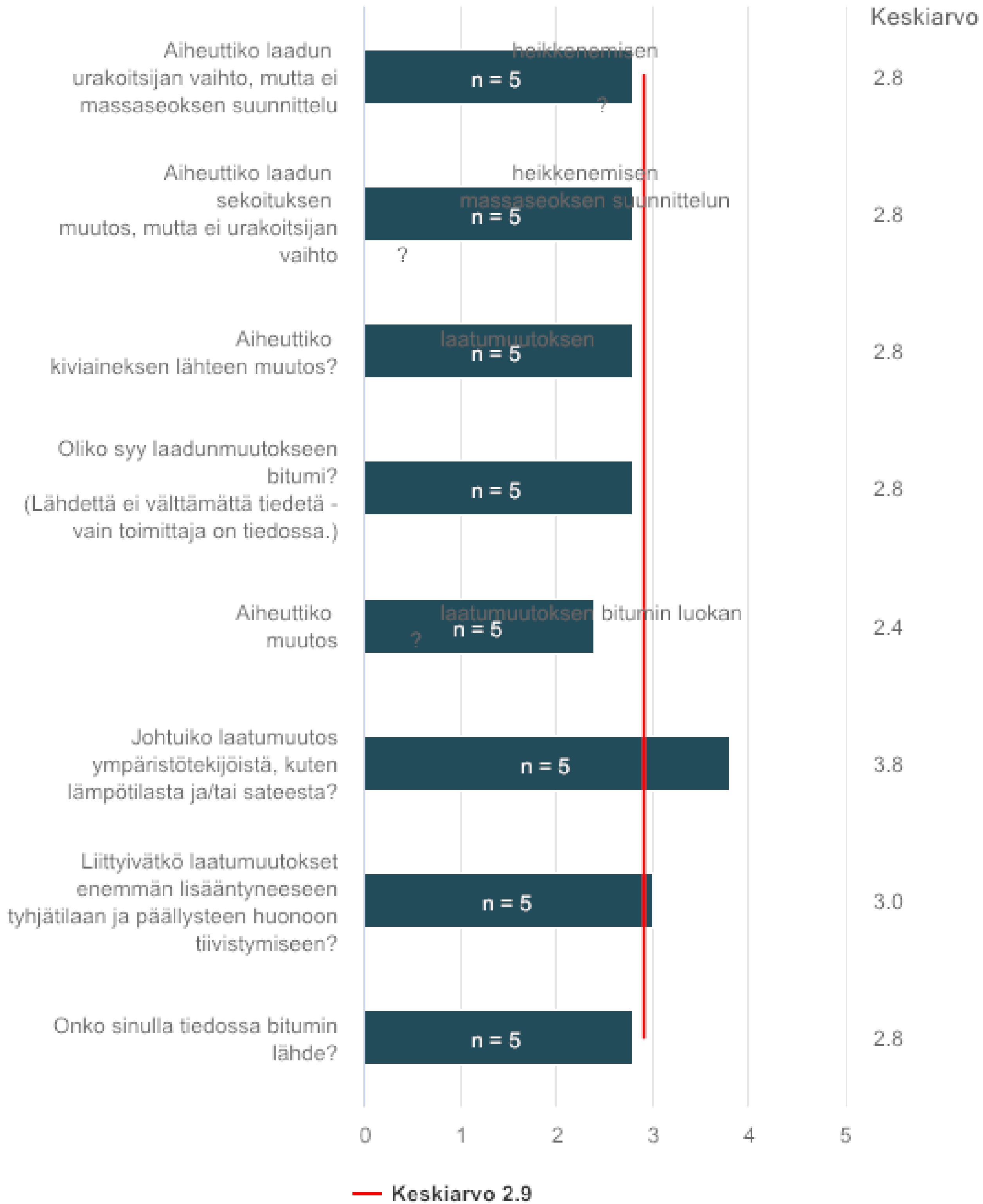
Vastaajien määrä: 5



	n	Prosentti
Kyllä, niiden kulutuskestävyys liikenteen ja sääolosuhteiden vuoksi on alhaisempi	3	60%
Ei, huoltotiheys ei ole lisääntynyt	0	0%
Kyllä, mutta se liittyy korjausten ja vaurioiden kertymiseen vähentyneen rahoituksen takia	2	40%

8. Syyt asfalttipäällysteiden heikentyneeseen laatuun.

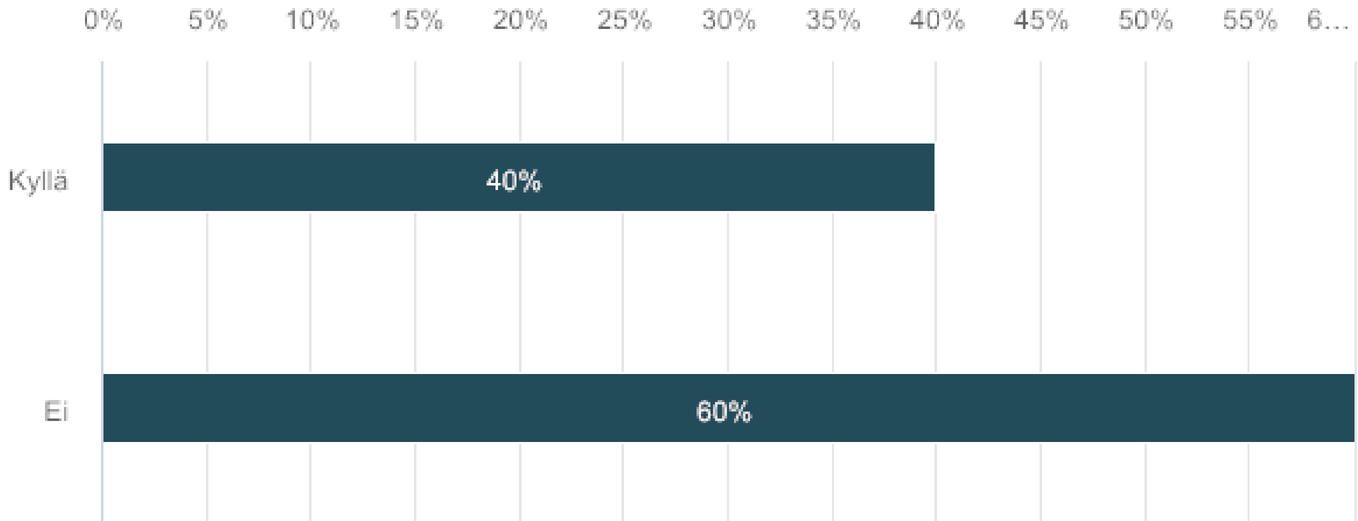
Vastaajien määrä: 5



	Ehdottomasti ei	Ei	Ehkä	Kyllä	Kyllä, ehdottomasti	Keskiarvo	Mediaani
Aiheuttiko laadun heikkenemisen urakoitsijan vaihto, mutta ei massaseoksen suunnittelu?	0%	20 %	80%	0%	0%	2.8	3
Aiheuttiko laadun heikkenemisen sekoituksen massaseoksen suunnittelun muutos, mutta ei urakoitsijan vaihto?	0%	20 %	80%	0%	0%	2.8	3
Aiheuttiko laatumuutoksen kiviaineksen lähteen muutos?	0%	20 %	80%	0%	0%	2.8	3
Oliko syy laadunmuutokseen bitumi?(Lähdettä ei välttämättä tiedetä -vain toimittaja on tiedossa.)	0%	20 %	80%	0%	0%	2.8	3
Aiheuttiko laatumuutoksen bitumin luokan muutos?	0%	60 %	40%	0%	0%	2.4	2
Johtuiko laatumuutos ympäristötekijöistä, kuten lämpötilasta ja/tai sateesta?	0%	0%	20%	80%	0%	3.8	4
Liittyivätkö laatumuutokset enemmän lisääntyneeseen tyhjätilaan ja päällysteen huonoon tiivistymiseen?	0%	20 %	60%	20%	0%	3	3
Onko sinulla tiedossa bitumin lähde?	0%	40 %	40%	20%	0%	2.8	3

9. Saatko AB-seosten tyyppitestaukseen liittyen vedenkestävyystestin EN 12697-12 menetelmän A mukaiset kuivien ja märkien näytteiden halkaisuvetolujuuden testitulokset etkä pelkästään tulosta "ITSR>80%"?

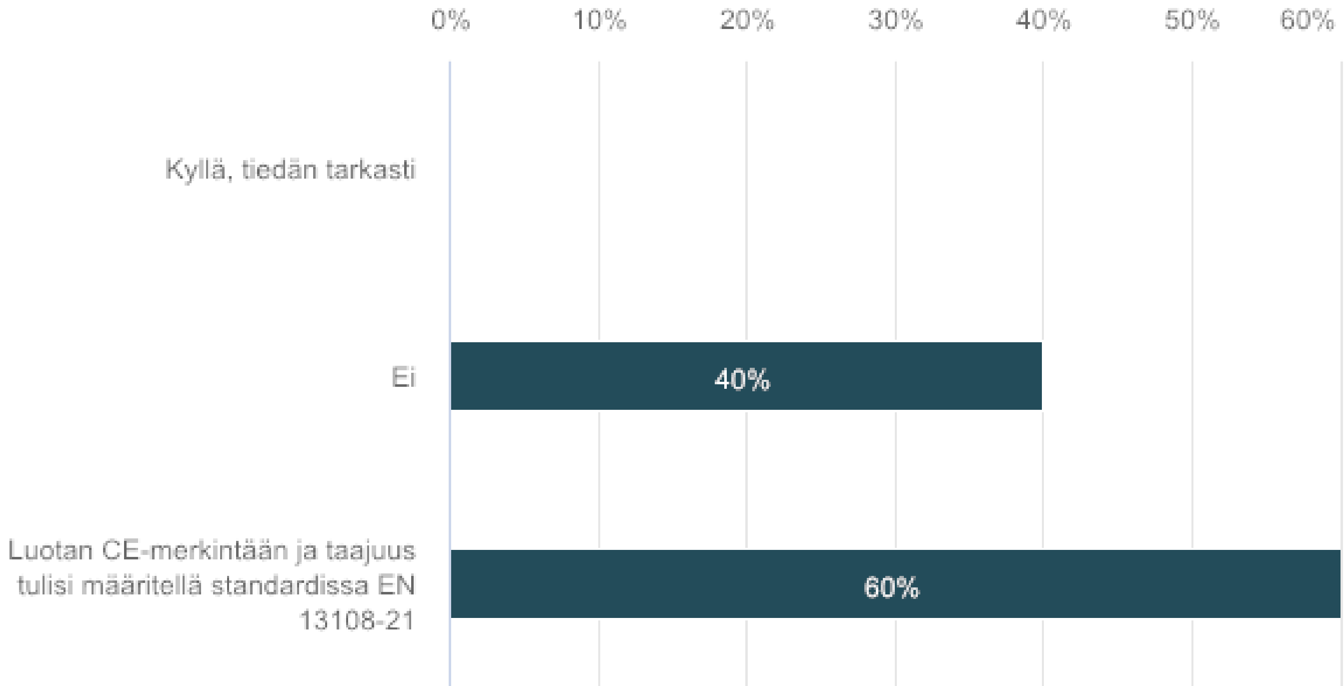
Vastaajien määrä: 5



	n	Prosentti
Kyllä	2	40%
Ei	3	60%

10. Tiedätkö, kuinka usein tilattujen AB-seosten valmistaja on testannut vedenkestävyyden (EN 12967-12 menetelmä A)?

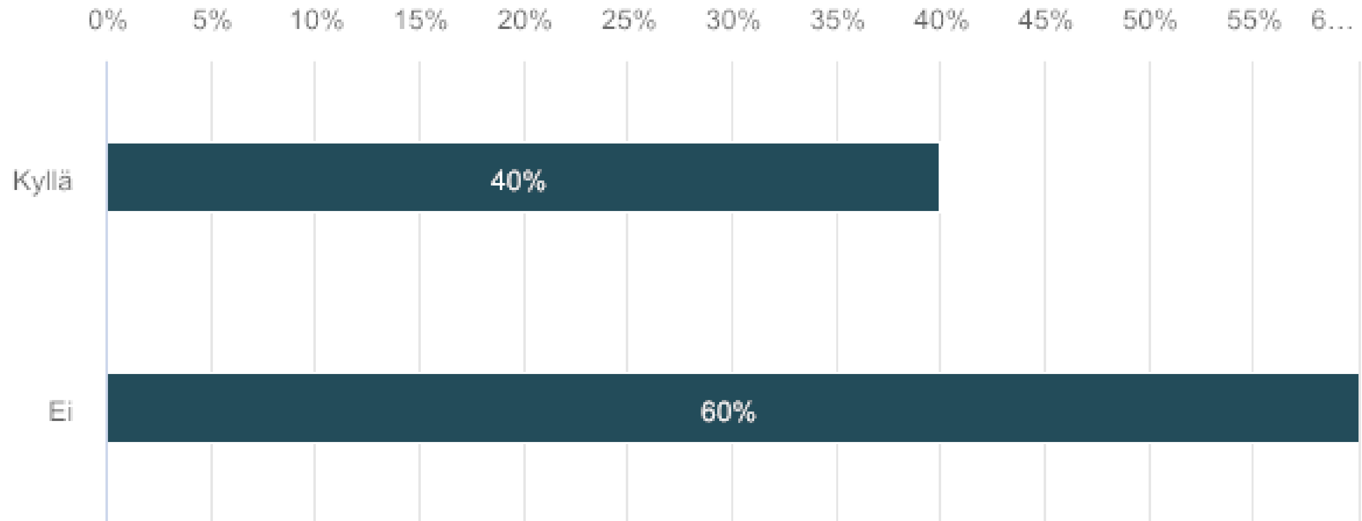
Vastaajien määrä: 5



	n	Prosentti
Kyllä, tiedän tarkasti	0	0%
Ei	2	40%
Luotan CE-merkintään ja taajuus tulisi määritellä standardissa EN 13108-21	3	60%

11. Saatko SMA-seosten tyyppitestaukseen liittyen vedenkestävyystestin EN 12697-12 menetelmän A mukaiset kuivien ja märkien näytteiden halkaisuvetolujuuden testitulokset etkä pelkästään tulosta "ITSR>80%?

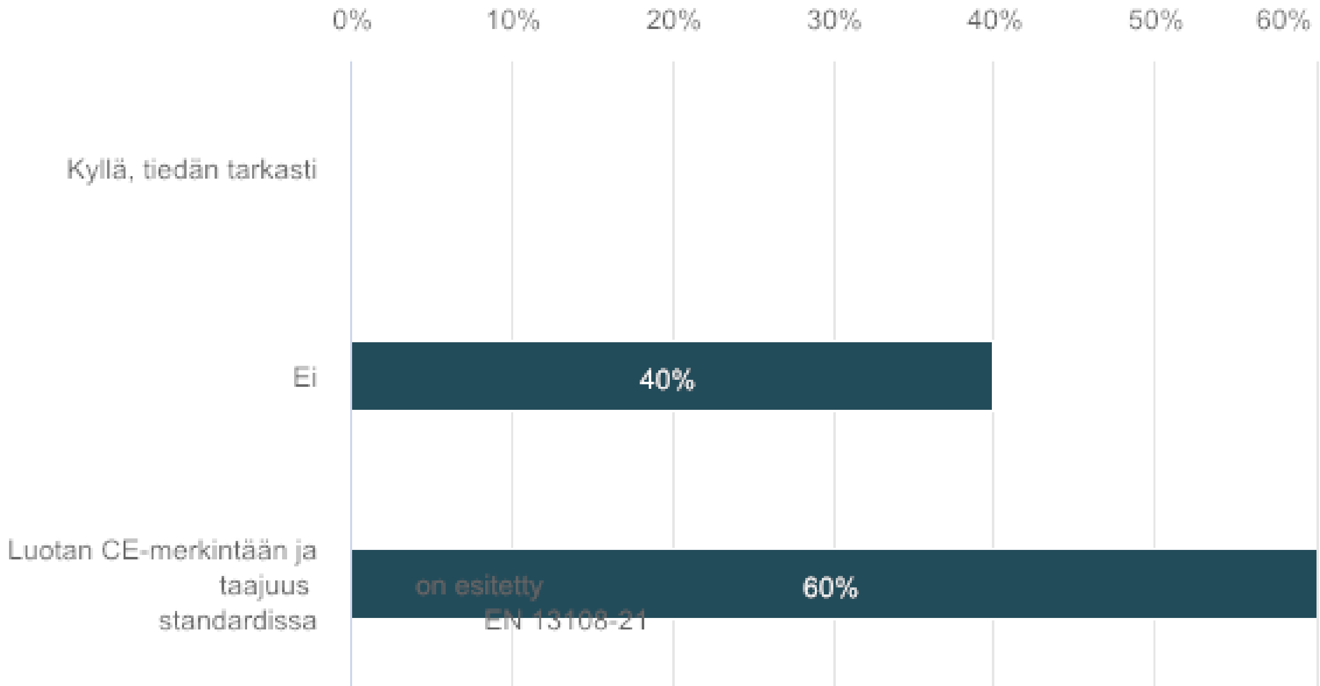
Vastaajien määrä: 5



	n	Prosentti
Kyllä	2	40%
Ei	3	60%

12. Tiedätkö, kuinka usein tilattujen SMA-seosten valmistaja on testannut vedenkestävyyden (12967-12 menetelmä A)?

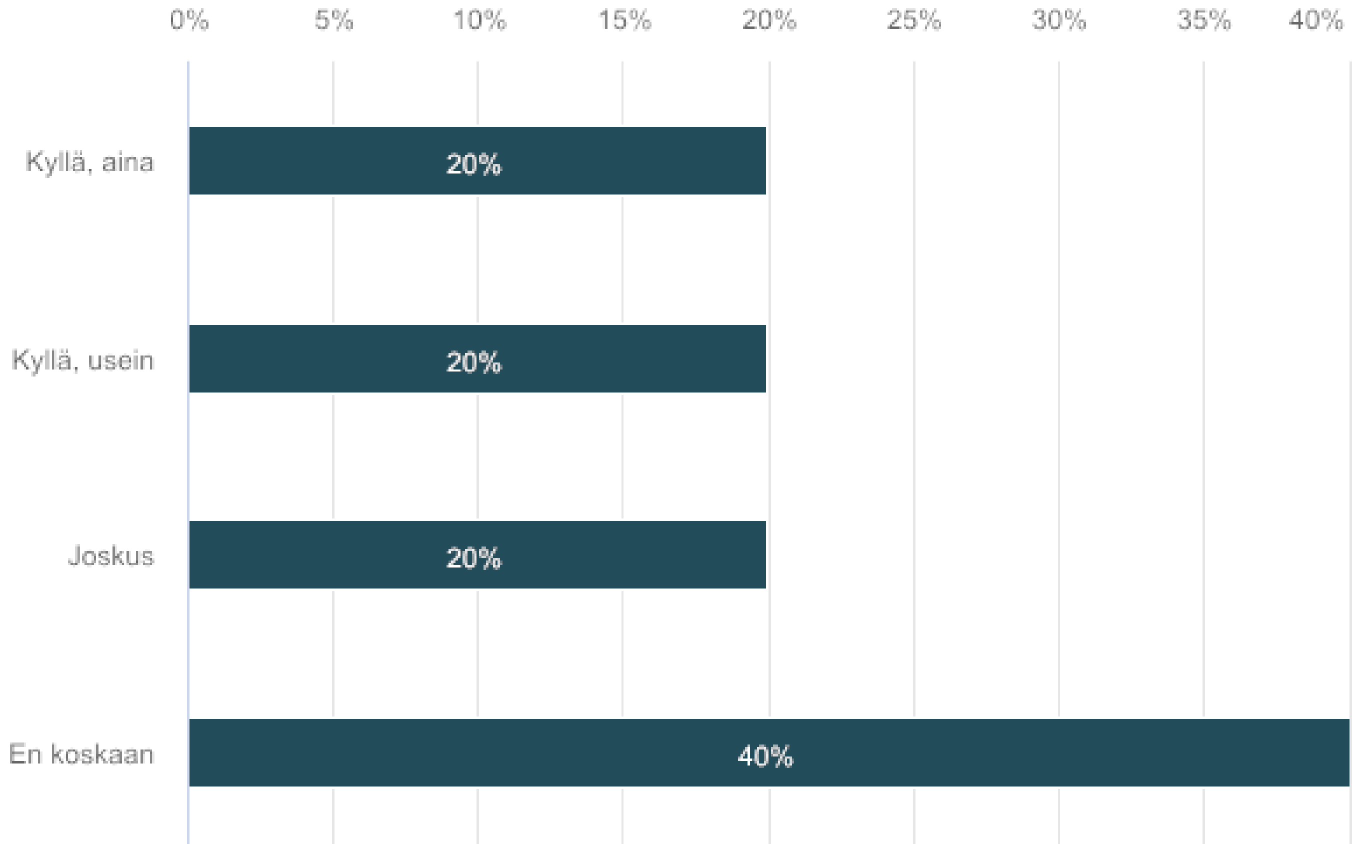
Vastaajien määrä: 5



	n	Prosentti
Kyllä, tiedän tarkasti	0	0%
Ei	2	40%
Luotan CE-merkintään ja taajuus on esitetty standardissa EN 13108-21	3	60%

13. Saatko päällystekiviaineksista testaustuloksia, joilla osoitetaan etteivät ne sisällä tarttuvuuden kannalta haitallisia mineraaleja (esim. ohuthietutkimuksen PANK 2302 tai petrografisen analyysin SFS-EN 12407 tuloksia)?

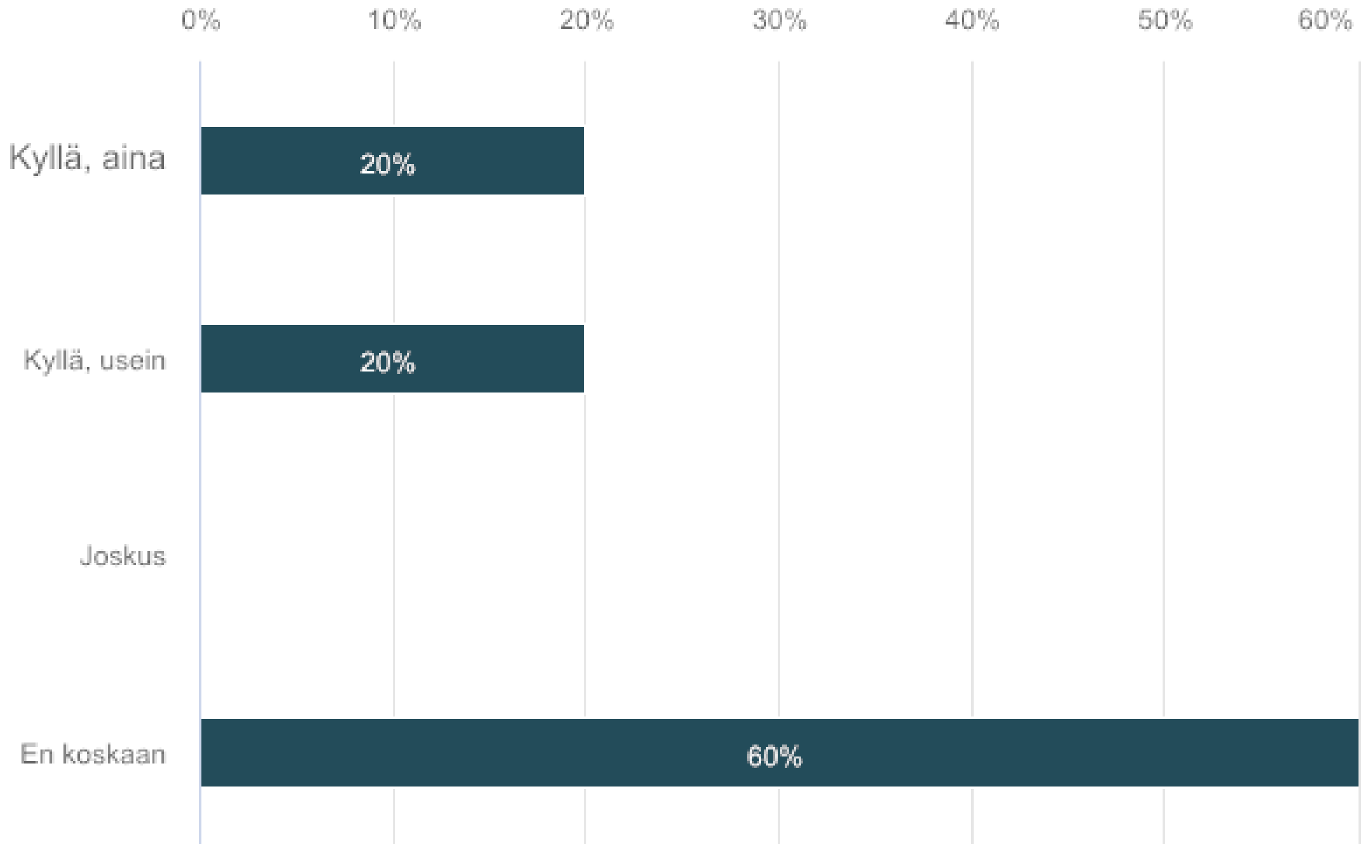
Vastaajien määrä: 5



	n	Prosentti
Kyllä, aina	1	20%
Kyllä, usein	1	20%
Joskus	1	20%
En koskaan	2	40%

14. Saatko PAB-V-kiviaineksista tai massoista testaustuloksia (esim. PANK 2108, PANK 2401 tai EN 12697-12, menetelmä C), joilla osoitetaan niille sopiva tartukepitoisuus?

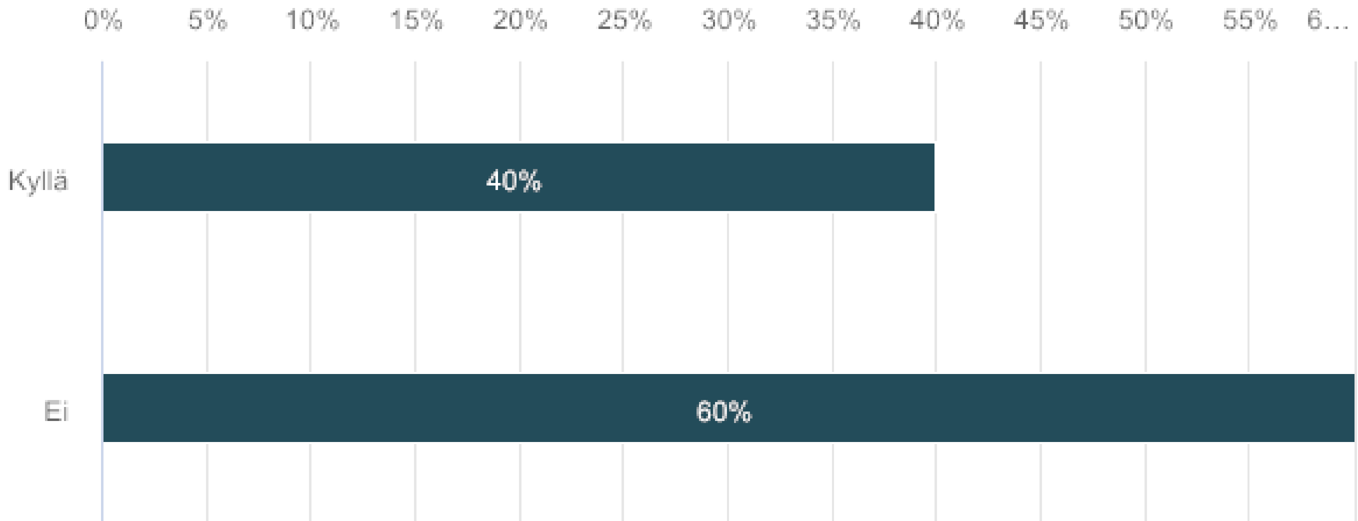
Vastaajien määrä: 5



	n	Prosentti
Kyllä, aina	1	20%
Kyllä, usein	1	20%
Joskus	0	0%
En koskaan	3	60%

15. Saatko PAB-seosten tyyppitestaukseen liittyen vedenkestävyystestin EN 12697-12 menetelmän A mukaiset kuivien ja märkien näytteiden halkaisuvetolujuuden testitulokset etkä pelkästään tulosta "ITSR>60%?

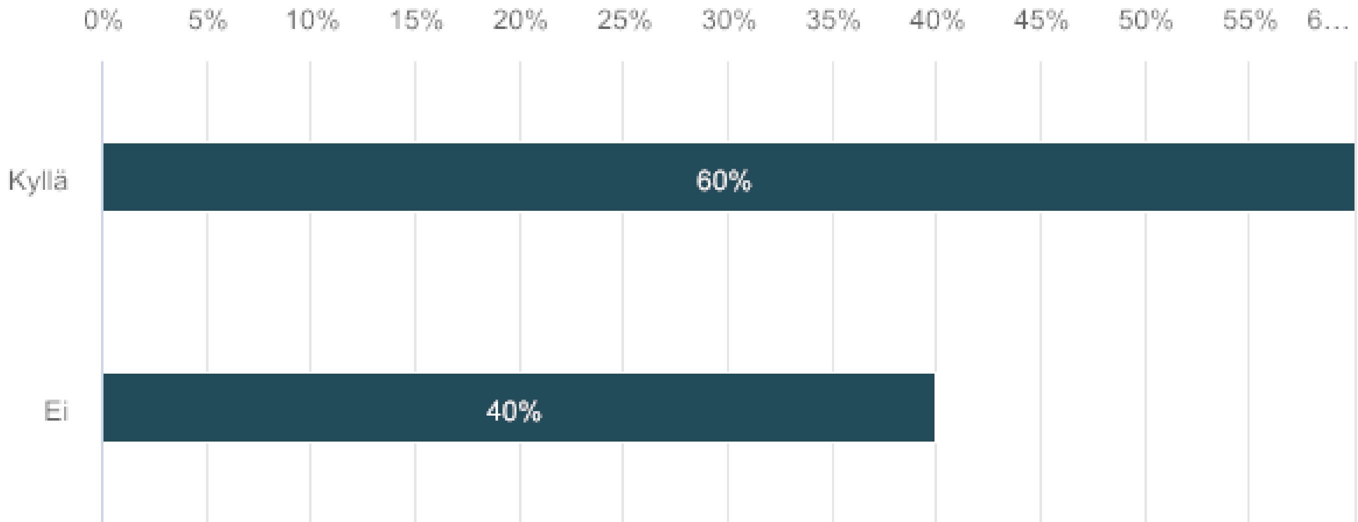
Vastaajien määrä: 5



	n	Prosentti
Kyllä	2	40%
Ei	3	60%

16. Saatko testaustuloksia vedenkestävyydestä PAB-seosten valmistuksen aikana? (MYR [EN 12697-12 menetelmä C] tai sankokoe)

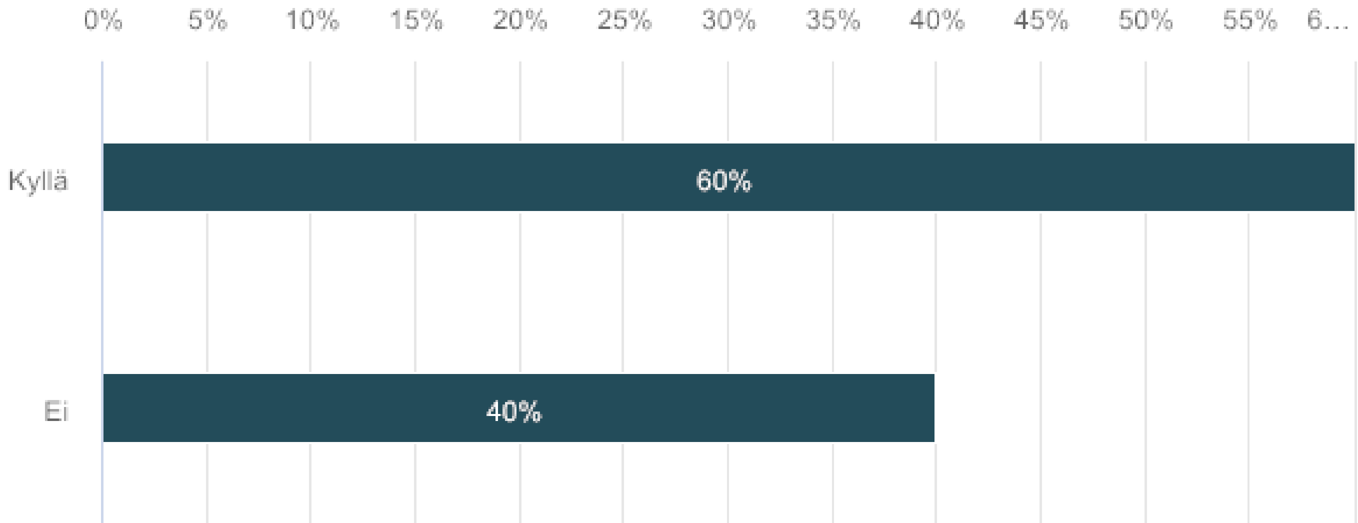
Vastaajien määrä: 5



	n	Prosentti
Kyllä	3	60%
Ei	2	40%

17. Onko ITSR-testi (menetelmä 12697-12 menetelmä A) käytettynä Asfalttinormien 2017 mukaisesti PANK 4301-menetelmän määrittämällä AA-rakeisuudella ja bitumilla 70/100 riittävä todiste PAB-B:n vedenkestävyydestä ?

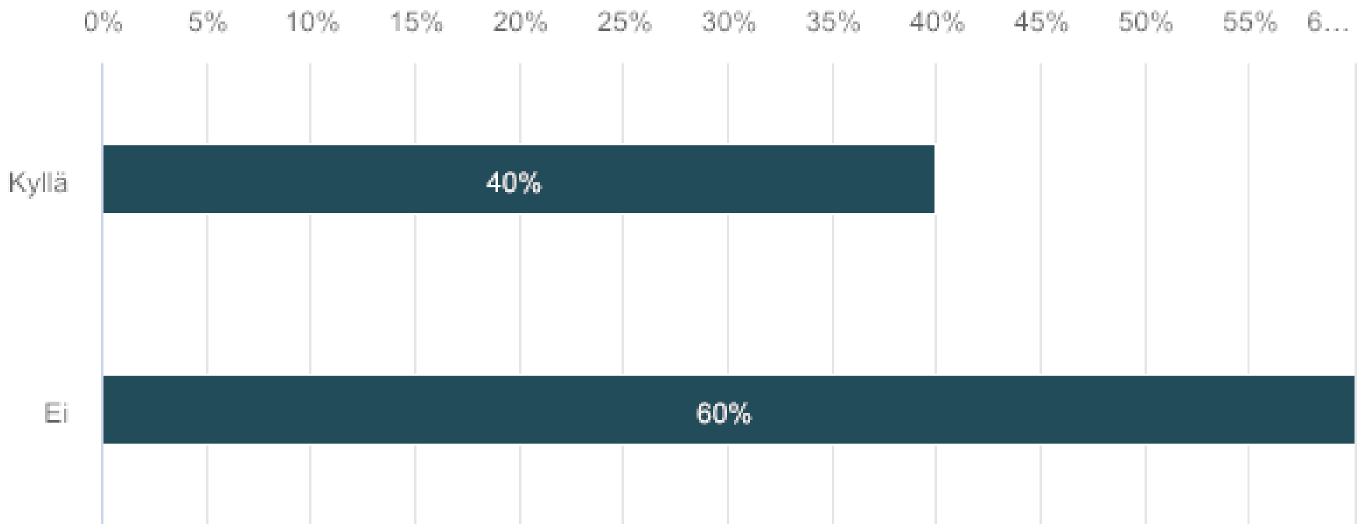
Vastaajien määrä: 5



	n	Prosentti
Kyllä	3	60%
Ei	2	40%

18. Vaaditko PAB-V-kiviaineksen ja V1500-sideaineen yhteensopivuuden testaamisen?

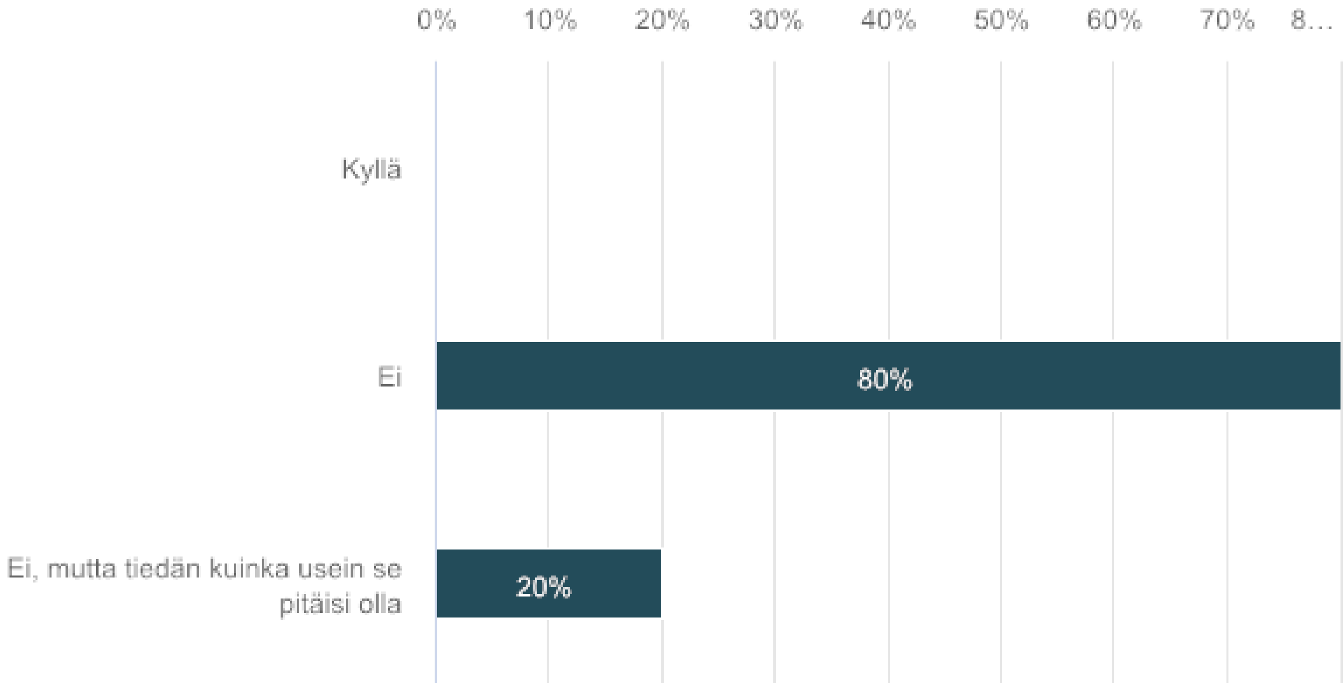
Vastaajien määrä: 5



	n	Prosentti
Kyllä	2	40%
Ei	3	60%

19. Tiedätkö, kuinka usein PAB-massan valmistaja testaa sen vedenkestävyyttä?

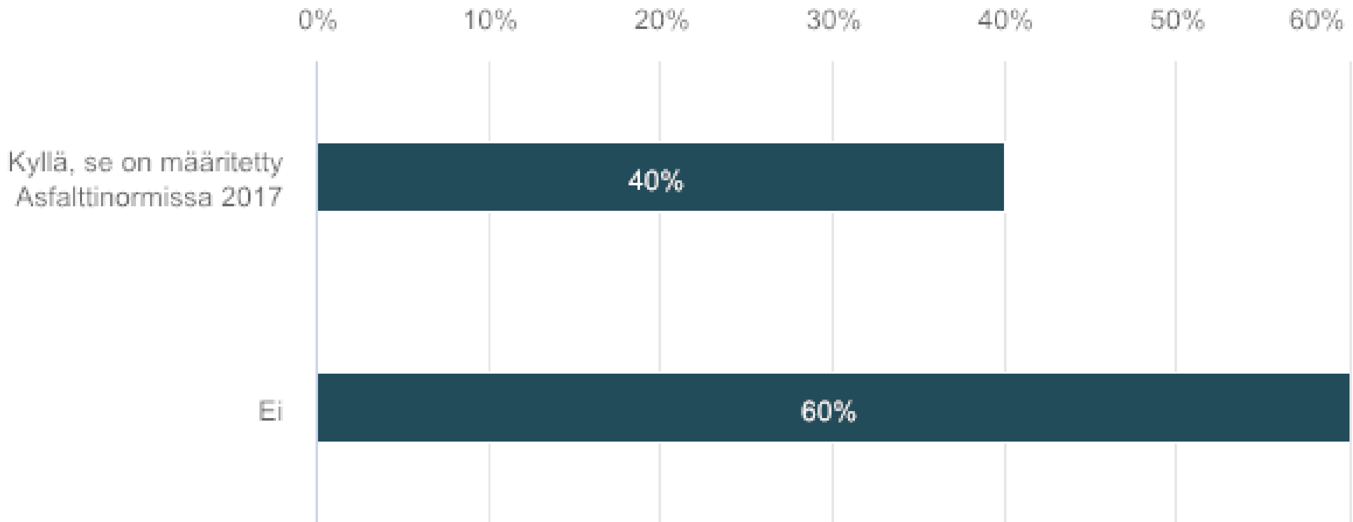
Vastaajien määrä: 5



	n	Prosentti
Kyllä	0	0%
Ei	4	80%
Ei, mutta tiedän kuinka usein se pitäisi olla	1	20%

20. Onko päällysteessä käytettävä teräskuonakiviaineksen täytettävä kriteeri tilavuuden muutoksen sallitulle maksimiarvolle?

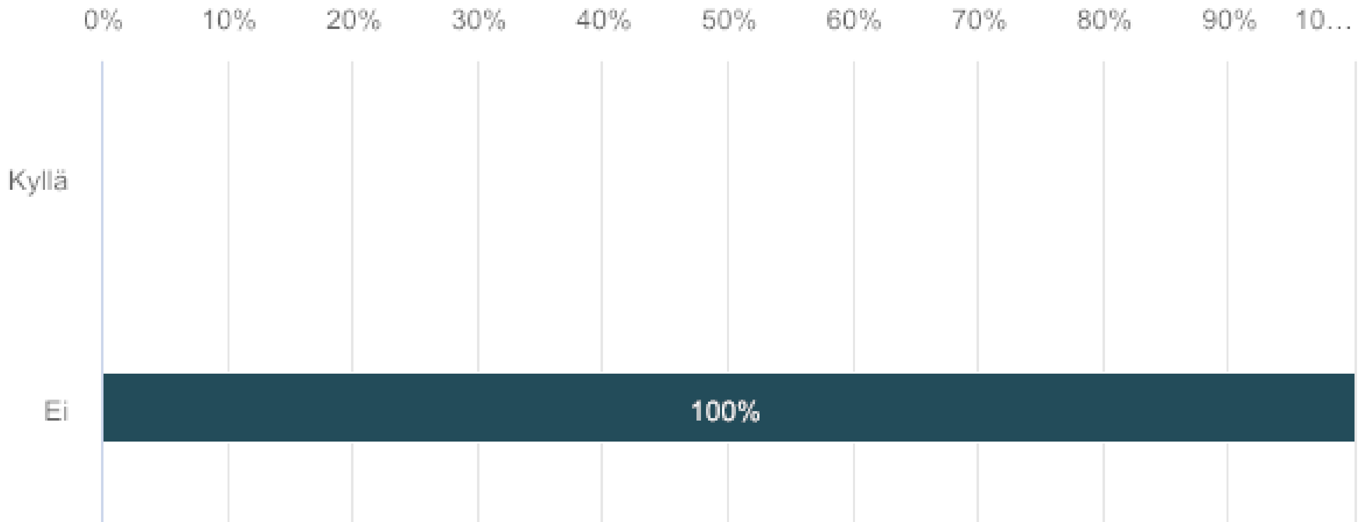
Vastaajien määrä: 5



	n	Prosentti
Kyllä, se on määritetty Asfalttinormissa 2017	2	40%
Ei	3	60%

21. Ilmoittaako tuottaja teräskuonakiviaineksen tilavuuden laajentumisen?

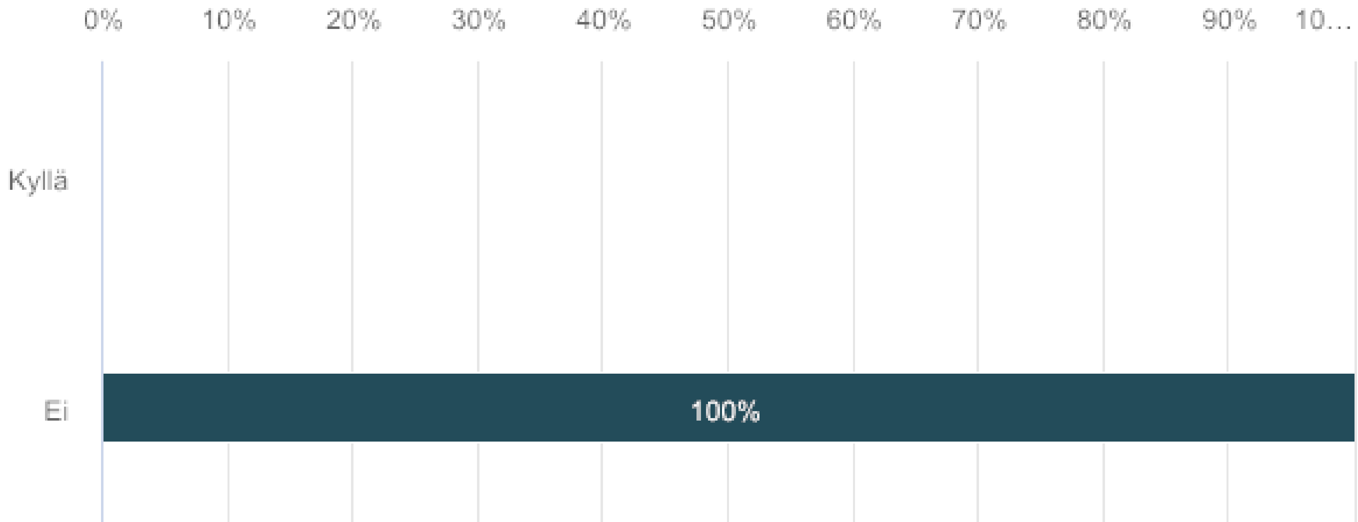
Vastaajien määrä: 5



	n	Prosentti
Kyllä	0	0%
Ei	5	100%

22. Tiedätkö, kuinka usein teräskuonakiviaineksen tuottaja testaa tilavuuden laajentumisen tuotteessaan?

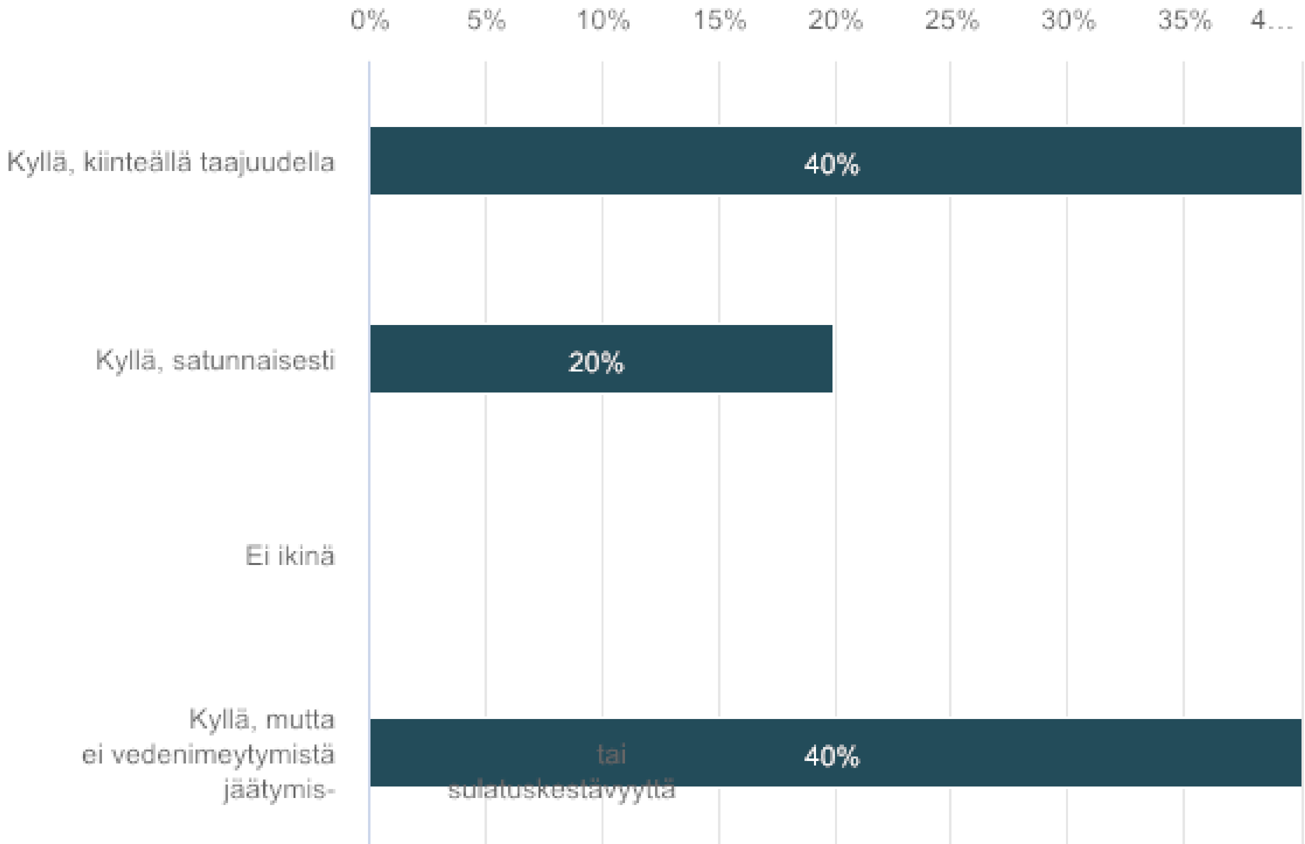
Vastaajien määrä: 5



	n	Prosentti
Kyllä	0	0%
Ei	5	100%

23. Teetkö laadunvarmistuksen suoritusindikaattoreihin (vedenimeytyminen, jäätymis-sulatuskestävyys, tiheys, liiteysluku) liittyviä kiviaineksen testejä, kiviaineksen saavuttua asfaltin tuotantolaitokselle?

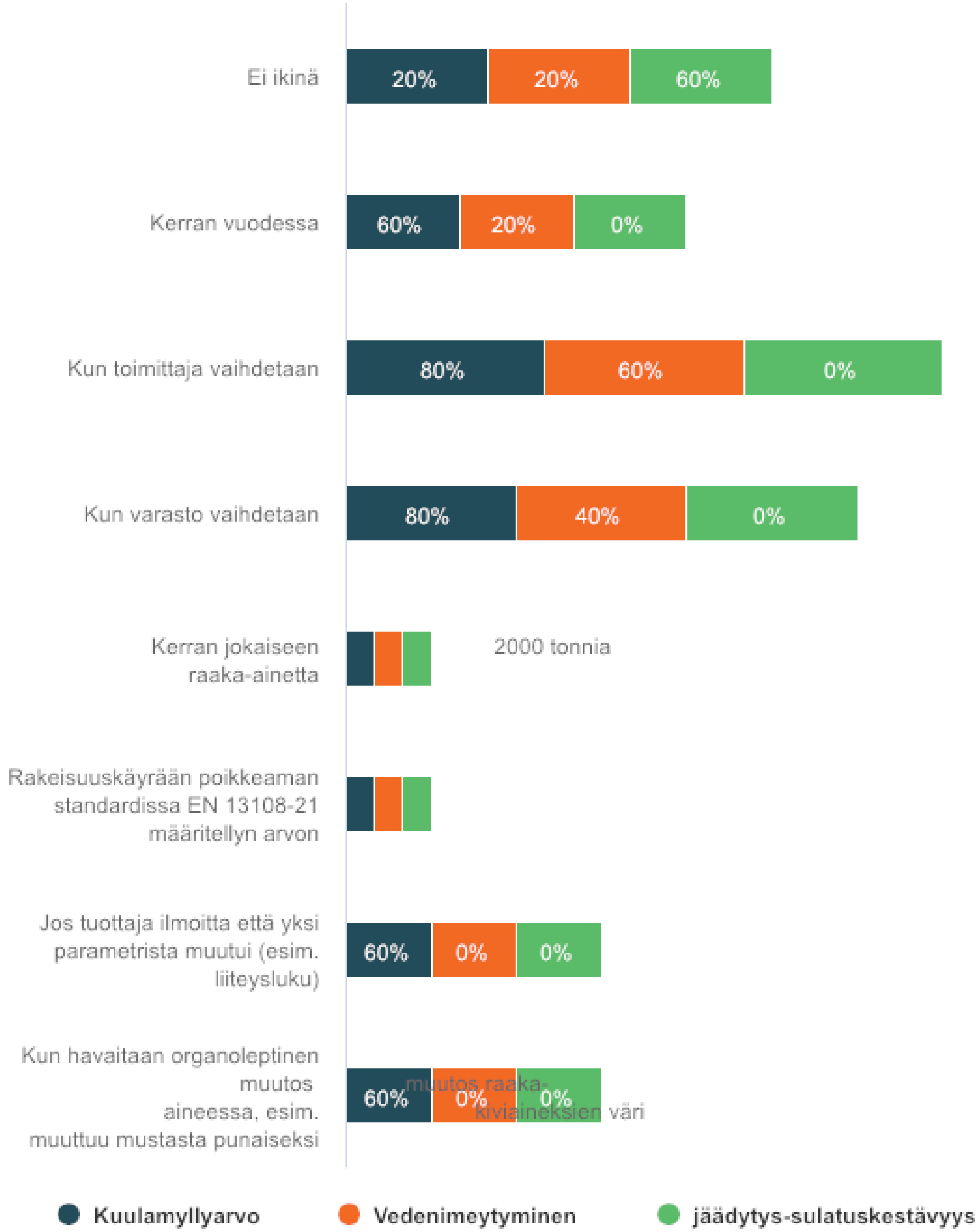
Vastaajien määrä: 5



	n	Prosentti
Kyllä, kiinteällä taajuudella	2	40%
Kyllä, satunnaisesti	1	20%
Ei ikinä	0	0%
Kyllä, mutta ei vedenimeytymistä tai jäätymis-sulatuskestävyyttä	2	40%

24. Kuinka usein suoritat tuotantolaitokselle toimitettujen kiviaineksien suorituskykymerkkien laadunvarmistuksen?

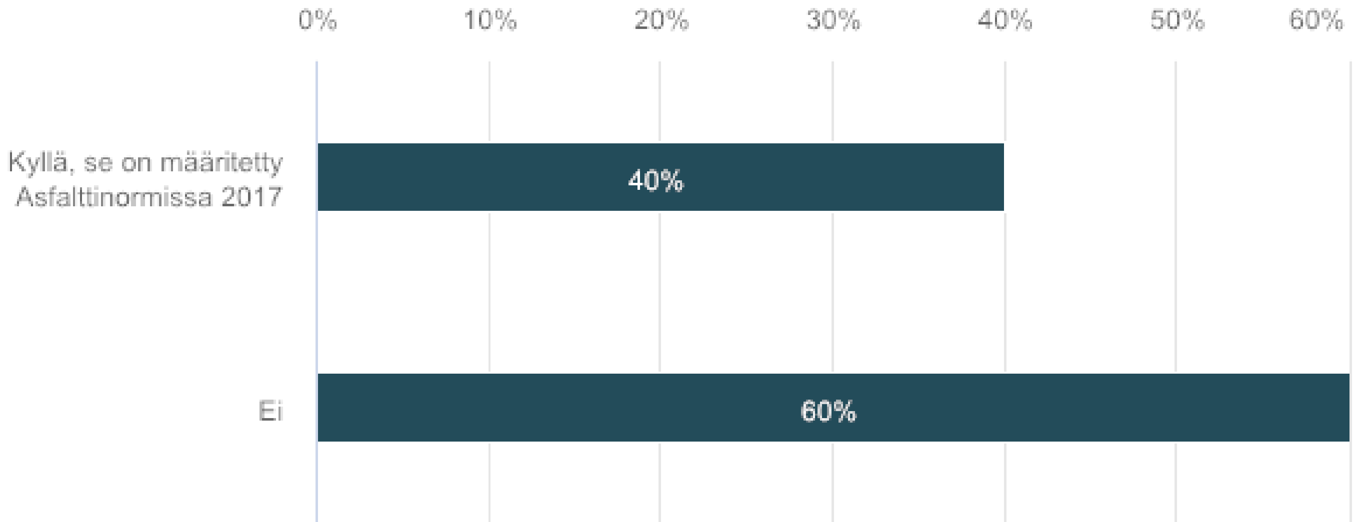
Vastaajien määrä: 5, valittujen vastausten lukumäärä: 30



	Kuulamyllyarvo	Vedenimeytyminen	jäädytys-sulatuskestävyys	Yhteensä	Keskiarvo	Mediaani
Ei ikinä	1	1	3	5	2.4	3
Kerran vuodessa	3	1	0	5	1.25	1
Kun toimittaja vaihdetaan	4	3	0	5	1.43	1
Kun varasto vaihdetaan	4	2	0	5	1.33	1
Kerran jokaiseen 2000 tonnia raaka-ainetta	1	0	0	5	1	1
Rakeisuuskäyrään poikkeaman standardissa EN 13108-21 määritellyn arvon	1	0	0	5	1	1
Jos tuottaja ilmoitta että yksi parametrista muutui (esim. liiteysluku)	3	0	0	5	1	1
Kun havaitaan organoleptinen muutos muutos raaka-aineessa, esim. kiviaineksien väri muuttuu mustasta punaiseksi	3	0	0	5	1	1
Yhteensä	20	7	3	5	1.43	1

25. Onko päällysteessä käytettävä teräskuonakiviaineksen täytettävä kriteeri tilavuuden muutoksen sallitulle maksimiarvolle?

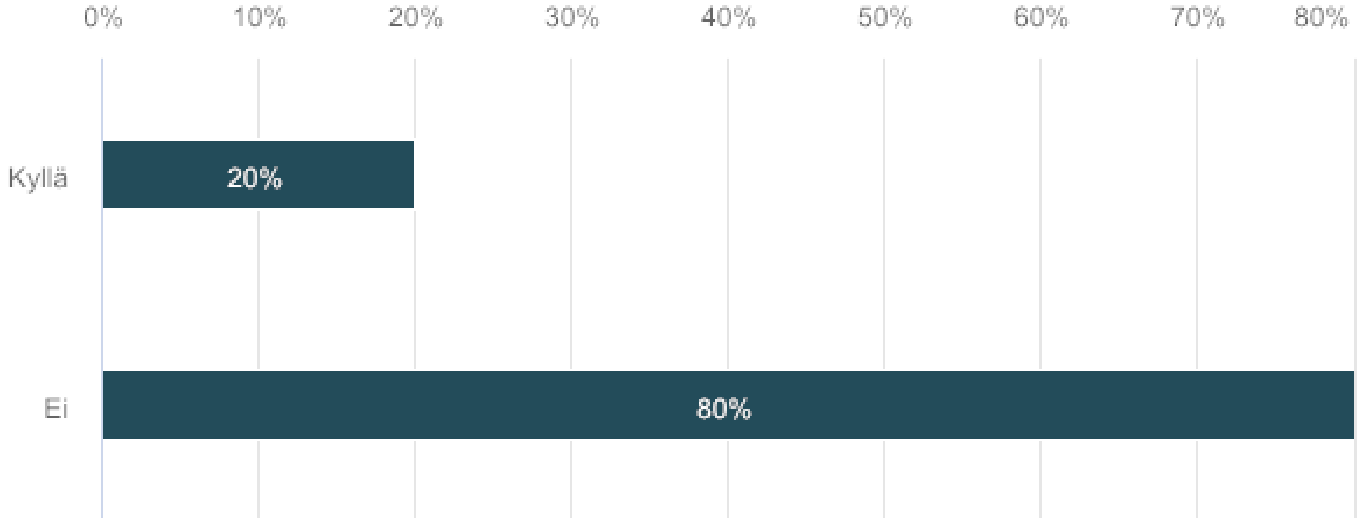
Vastaajien määrä: 5



	n	Prosentti
Kyllä, se on määritetty Asfalttinormissa 2017	2	40%
Ei	3	60%

26. Ilmoittaako tuottaja teräskuonakiviaineksen tilavuuden laajentumisen?

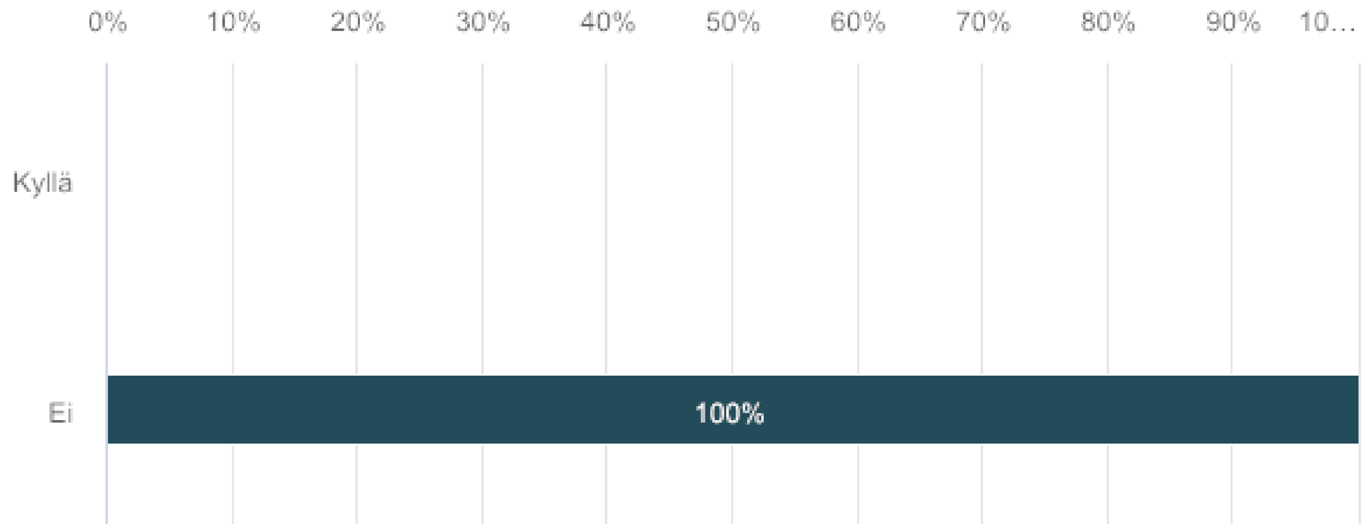
Vastaajien määrä: 5



	n	Prosentti
Kyllä	1	20%
Ei	4	80%

27. Tiedätkö, kuinka usein teräskuonakiviaineksen tuottaja testaa tilavuuden laajentumista tuotteensa?

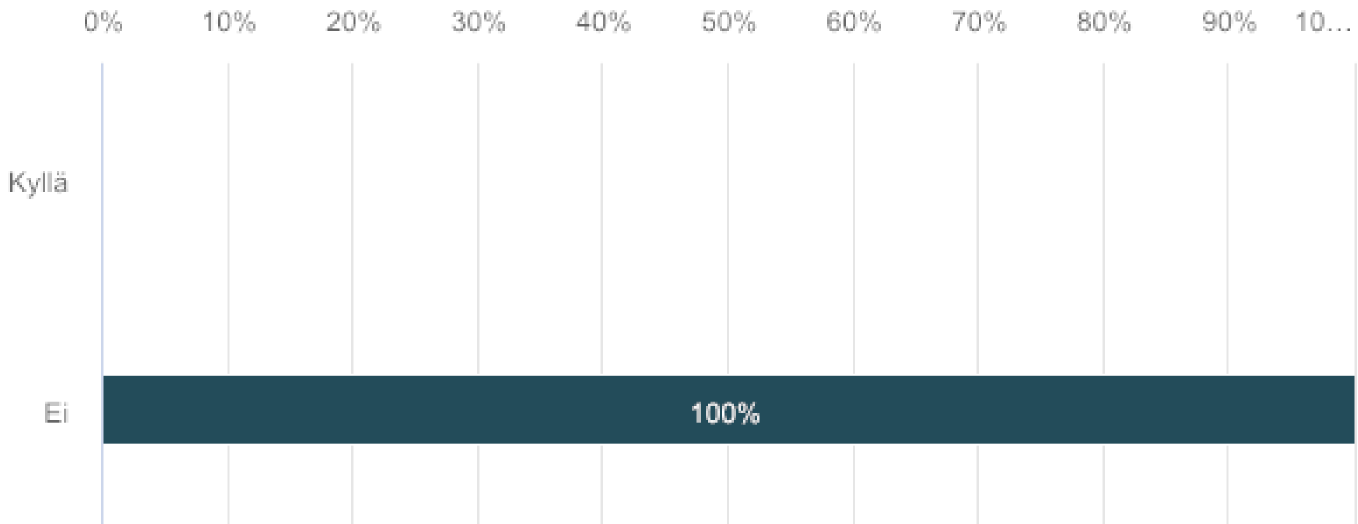
Vastaajien määrä: 5



	n	Prosentti
Kyllä	0	0%
Ei	5	100%

28. Teetkö omia kokeita testataksesi kuonan tilavuuden laajentumisen, vaikka tuottaja sen ilmoittaisi?

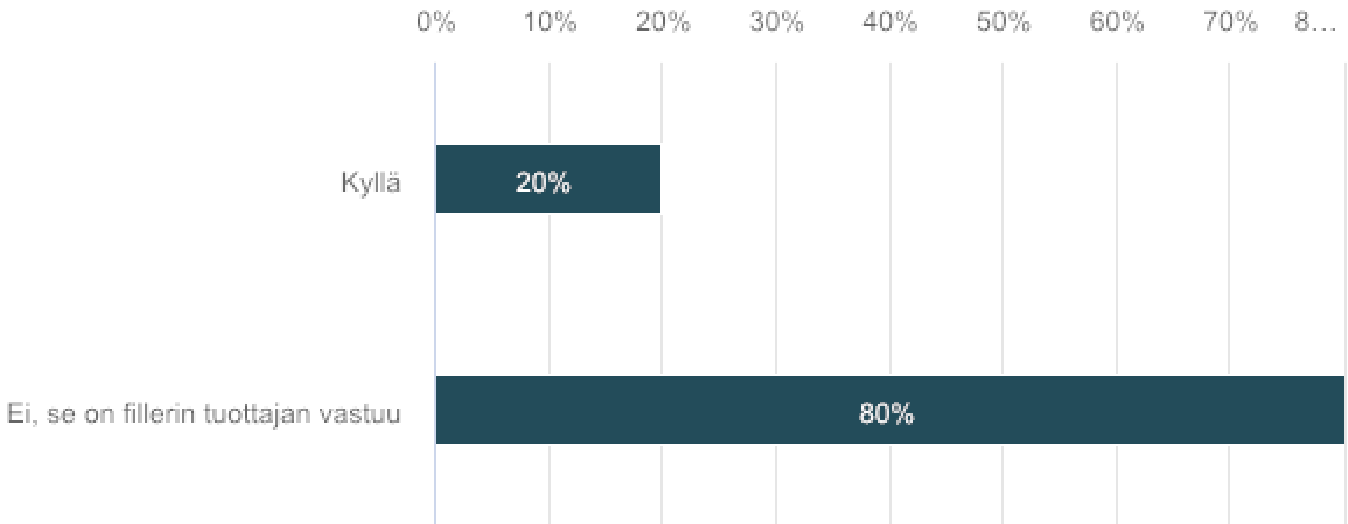
Vastaajien määrä: 5



	n	Prosentti
Kyllä	0	0%
Ei	5	100%

29. Varmistatko fillerin laadun testaamalla sen ominaispinta-alan kerran vuodessa?

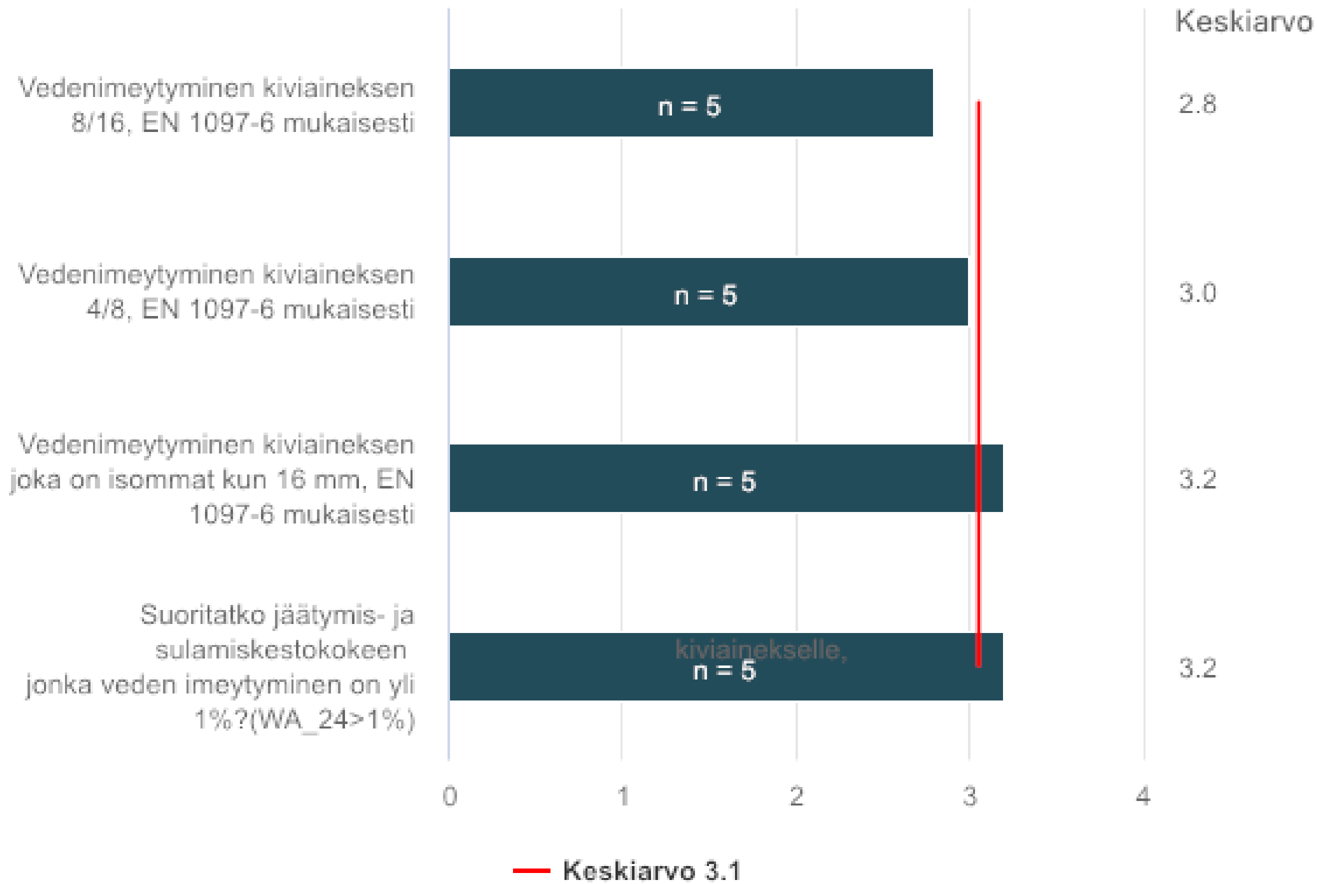
Vastaajien määrä: 5



	n	Prosentti
Kyllä	1	20%
Ei, se on fillerin tuottajan vastuu	4	80%

30. Varmistatko kiviaineksen laadun suorittamalla kyseiset testit:

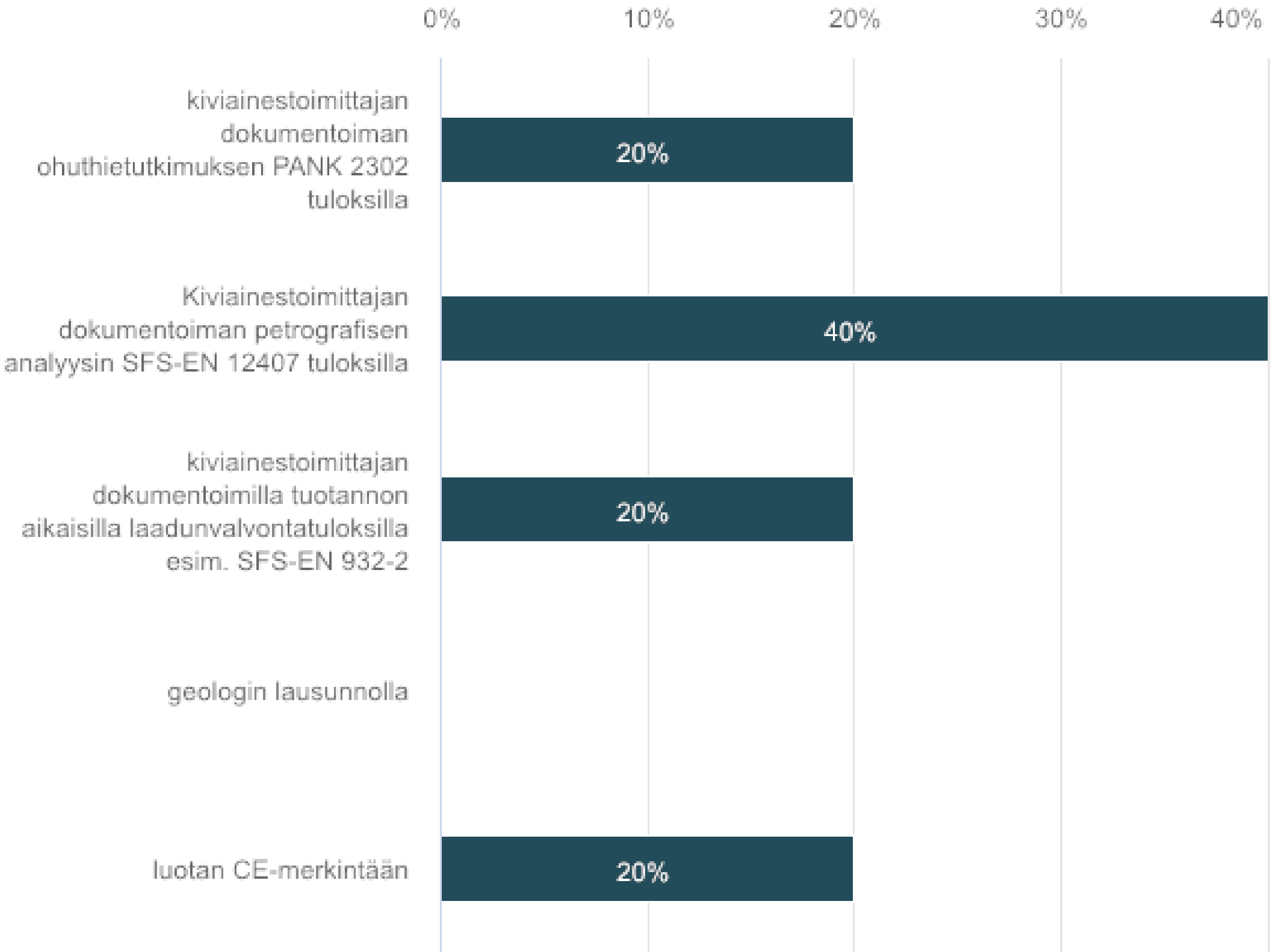
Vastaajien määrä: 5



	Kyllä, tietty taajuus	Kyllä, joskus	Ei ikinä	Kyllä, vain jos ELY vaati sitä	Keskiarvo	Mediaani
Vedenimeytyminen kiviaineksen 8/16, EN 1097-6 mukaisesti	40%	0%	0%	60%	2.8	4
Vedenimeytyminen kiviaineksen 4/8, EN 1097-6 mukaisesti	20%	20%	0%	60%	3	4
Vedenimeytyminen kiviaineksen joka on isommat kun 16 mm, EN 1097-6 mukaisesti	0%	40%	0%	60%	3.2	4
Suoritatko jäätymis- ja sulamiskestokokeen kiviainekselle, jonka veden imeytyminen on yli 1%?(WA_24>1%)	20%	0%	20%	60%	3.2	4

31. Kuinka varmistat, että päällystekiviaines ei sisällä liikaa tarttuvuuden kannalta haitallisia mineraaleja?

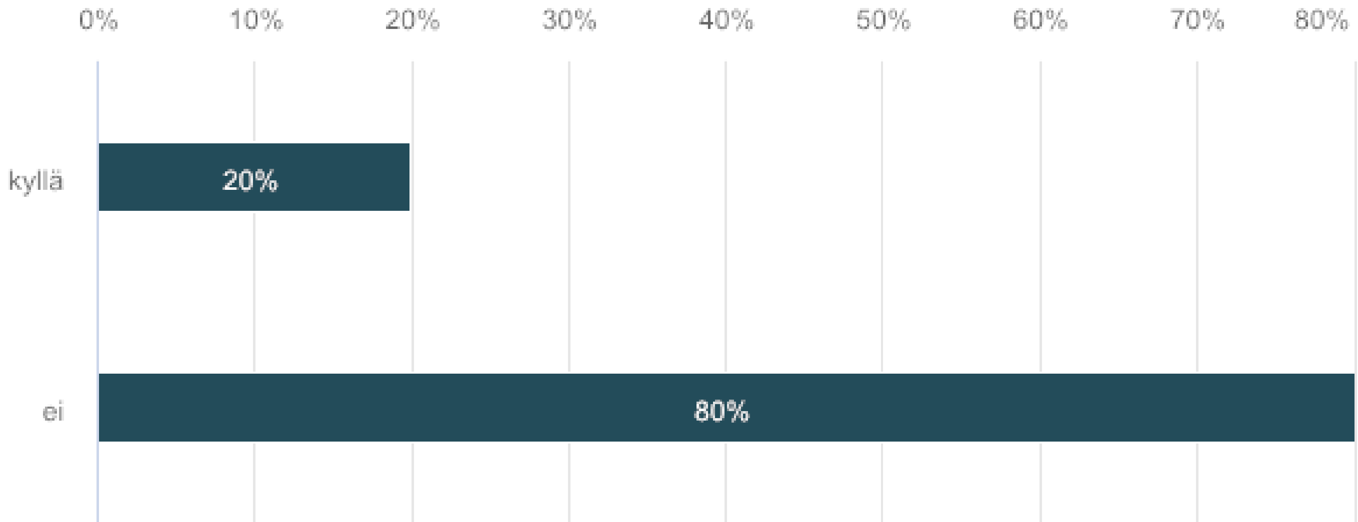
Vastaajien määrä: 5



	n	Prosentti
kiviainestoimittajan dokumentoiman ohuthietutkimuksen PANK 2302 tuloksilla	1	20%
Kiviainestoimittajan dokumentoiman petrografisen analyysin SFS-EN 12407 tuloksilla	2	40%
kiviainestoimittajan dokumentoimilla tuotannon aikaisilla laadunvalvontatuloksilla esim. SFS-EN 932-2	1	20%
geologin lausunnolla	0	0%
luotan CE-merkintään	1	20%

32. Teetkö itse em. kokeita, joilla varmistat että päällystekiviaines ei sisällä liikaa tarttuvuutta heikentäviä mineraaleja:

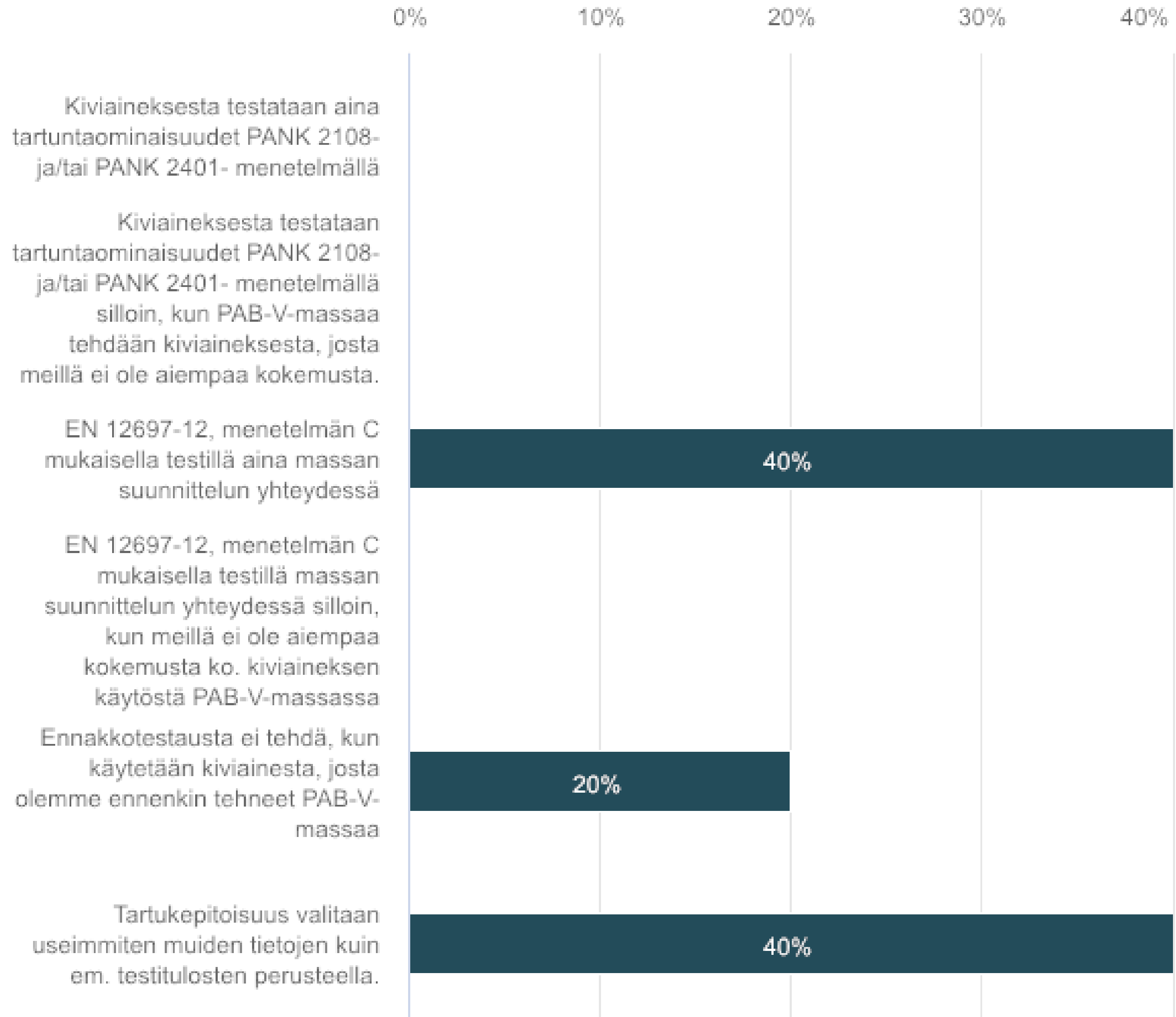
Vastaajien määrä: 5



	n	Prosentti
kyllä	1	20%
ei	4	80%

33. Mitä tietoja käytät PAB-V-massan tartukepitoisuuden valinnassa ?

Vastaajien määrä: 5

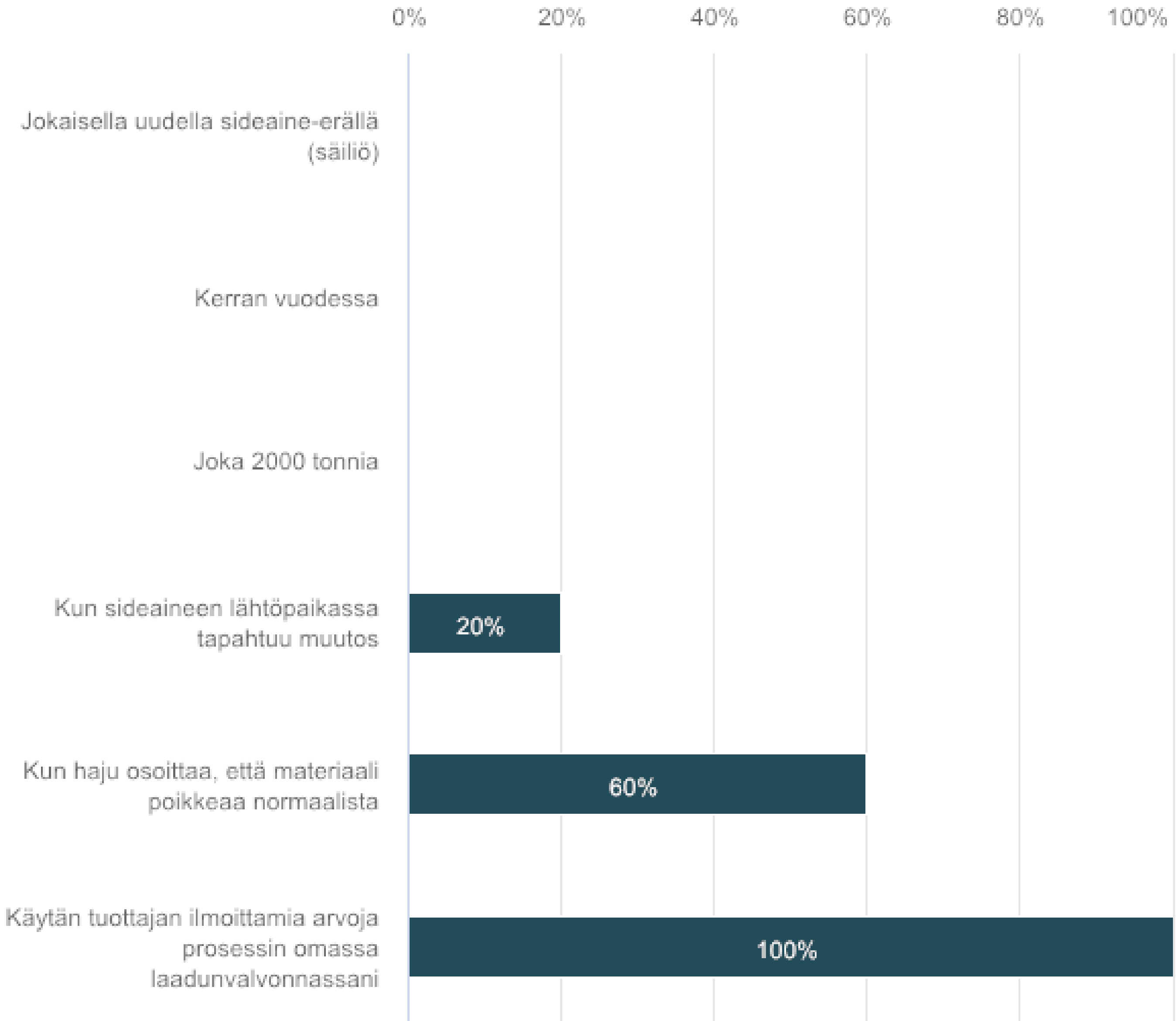


	n	Prosentti
Kiviaineksesta testataan aina tartuntaominaisuudet PANK 2108- ja/tai PANK 2401- menetelmällä	0	0%
Kiviaineksesta testataan tartuntaominaisuudet PANK 2108- ja/tai PANK 2401- menetelmällä silloin, kun PAB-V-massaa tehdään kiviaineksesta, josta meillä ei ole aiempaa kokemusta.	0	0%
EN 12697-12, menetelmän C mukaisella testillä aina massan suunnittelun yhteydessä	2	40%
EN 12697-12, menetelmän C mukaisella testillä massan suunnittelun yhteydessä silloin, kun meillä ei ole aiempaa kokemusta ko. kiviaineksen käytöstä PAB-V-massassa	0	0%
Ennakkotestausta ei tehdä, kun käytetään kiviainesta, josta olemme ennenkin tehneet PAB-V-massaa	1	20%

Tartukepitoisuus valitaan useimmiten muiden tietojen kuin em. testitulosten perusteella.	2	40%

34. Kuinka usein testaat sideaineen reologisia ominaisuuksia (esim. Tunkeuma, pehmenemispiste, viskositeetti)?

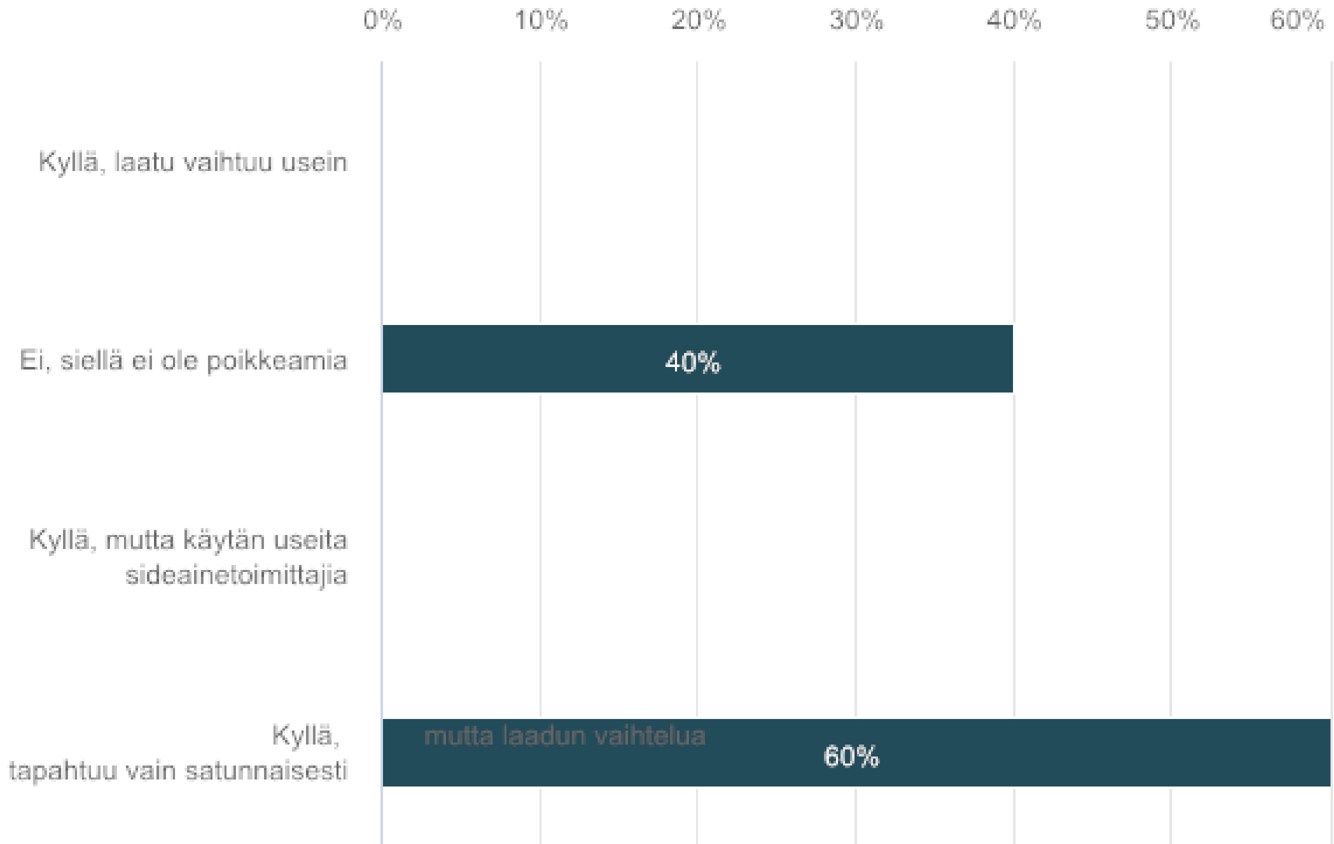
Vastaajien määrä: 5, valittujen vastausten lukumäärä: 9



	n	Prosentti
Jokaisella uudella sideaine-erällä (säiliö)	0	0%
Kerran vuodessa	0	0%
Joka 2000 tonnia	0	0%
Kun sideaineen lähtöpaikassa tapahtuu muutos	1	20%
Kun haju osoittaa, että materiaali poikkeaa normaalista	3	60%
Käytän tuottajan ilmoittamia arvoja prosessin omassa laadunvalvonnassani	5	100%

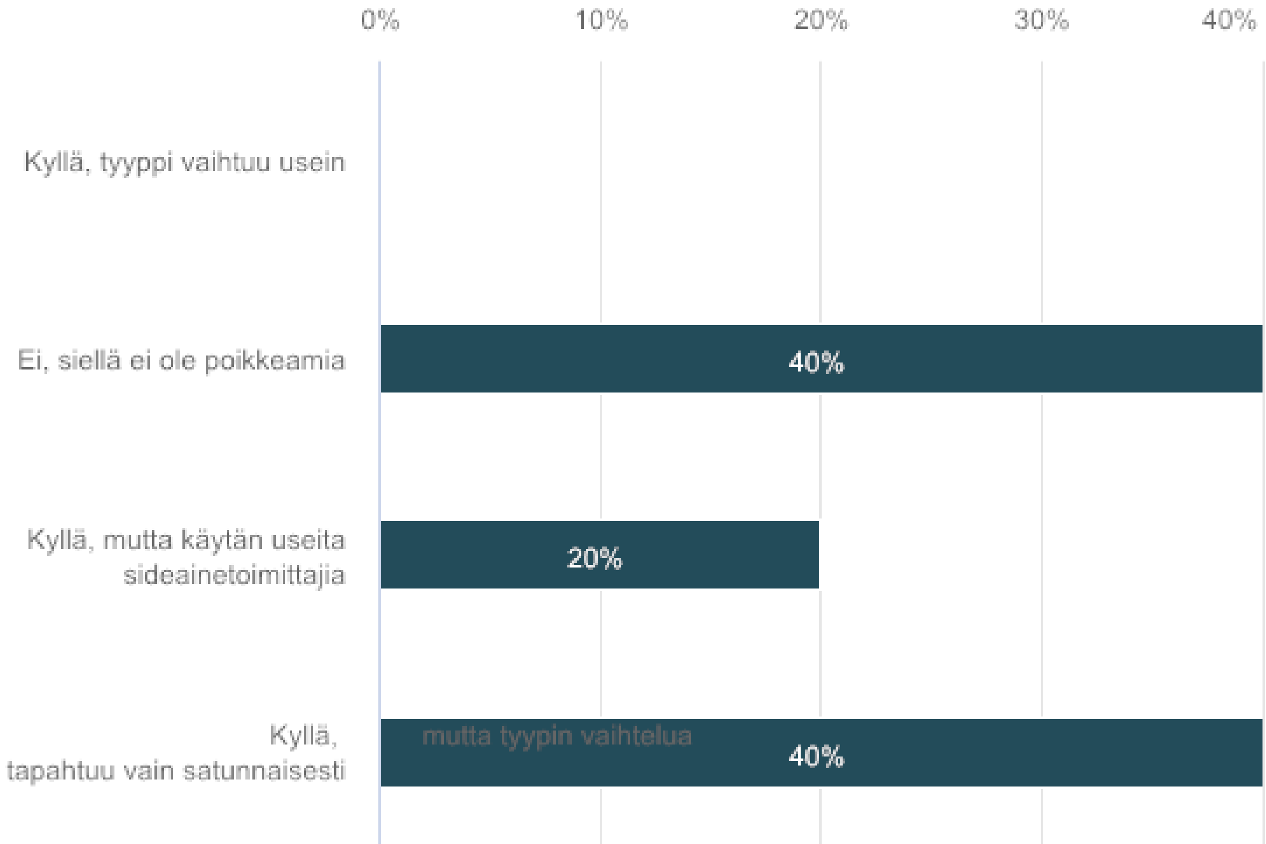
35. Oletko havainnut bitumin laadun muutosta viimeisen 3 vuoden aikana?

Vastaajien määrä: 5



	n	Prosentti
Kyllä, laatu vaihtuu usein	0	0%
Ei, siellä ei ole poikkeamia	2	40%
Kyllä, mutta käytän useita sideainetoimittajia	0	0%
Kyllä, mutta laadun vaihtelua tapahtuu vain satunnaisesti	3	60%

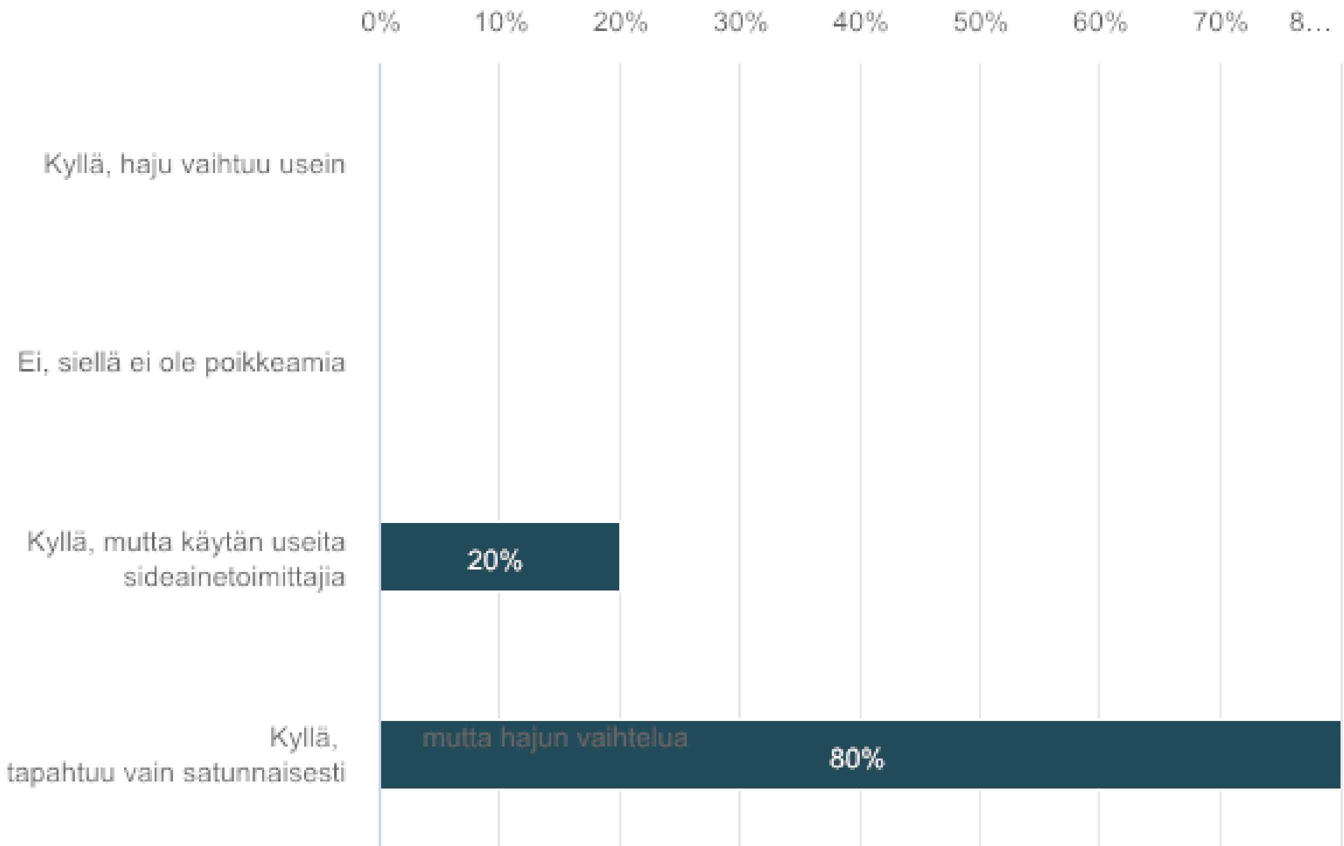
36. Oletko havainnut bitumin tyypin muutosta viimeisen 3 vuoden aikana?
Vastaajien määrä: 5



	n	Prosentti
Kyllä, tyyppi vaihtuu usein	0	0%
Ei, siellä ei ole poikkeamia	2	40%
Kyllä, mutta käytän useita sideainetoimittajia	1	20%
Kyllä, mutta tyypin vaihtelua tapahtuu vain satunnaisesti	2	40%

37. Oletko havainnut bitumin laadun / tyypin / hajun muutosta viimeisen 3 vuoden aikana?

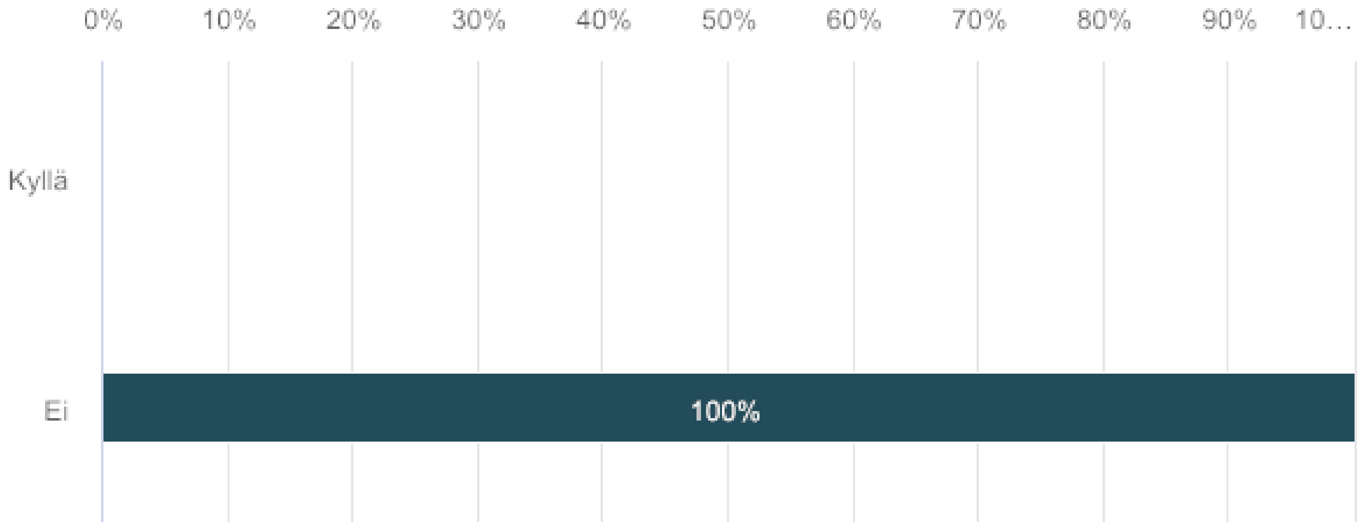
Vastaajien määrä: 5



	n	Prosentti
Kyllä, haju vaihtuu usein	0	0%
Ei, siellä ei ole poikkeamia	0	0%
Kyllä, mutta käytän useita sideainetoimittajia	1	20%
Kyllä, mutta hajun vaihtelua tapahtuu vain satunnaisesti	4	80%

38. Käytätkö spektroskooppisia tekniikoita sideaineiden erottamiseksi toisistaan?

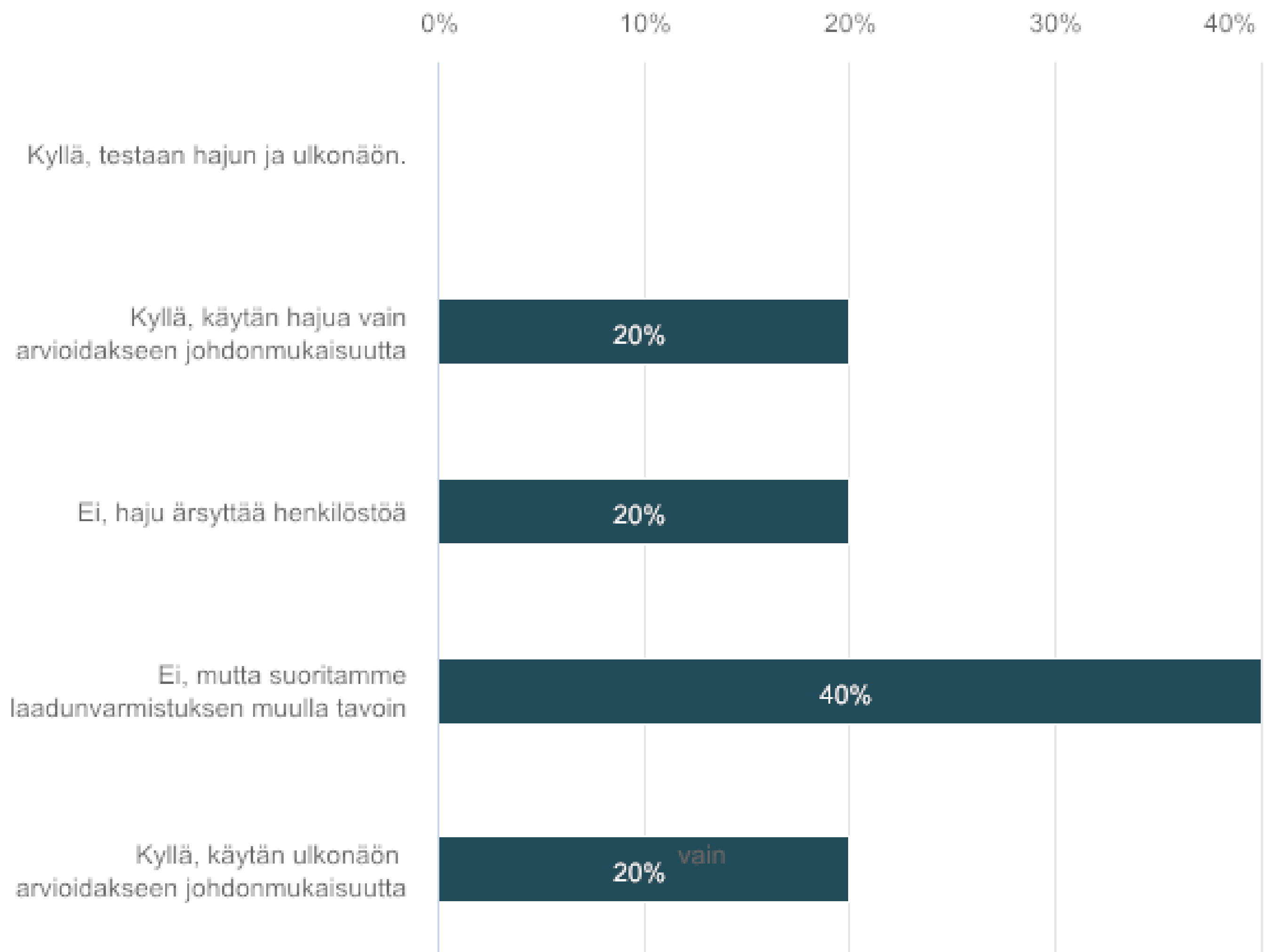
Vastaajien määrä: 5



	n	Prosentti
Kyllä	0	0%
Ei	5	100%

39. Standardi EN 13108-21 ehdottaa lisäaineiden organoleptistä tutkimusta. Suoritatko sitä tarttukkelle?

Vastaajien määrä: 5



	n	Prosentti
Kyllä, testaan hajun ja ulkonäön.	0	0%
Kyllä, käytän hajua vain arvioidakseen johdonmukaisuutta	1	20%
Ei, haju ärsyttää henkilöstöä	1	20%
Ei, mutta suoritamme laadunvarmistuksen muulla tavoin	2	40%
Kyllä, käytän ulkonäön vain arvioidakseen johdonmukaisuutta	1	20%

40. Kuinka usein suoritat vedenkestävyystestauksen menetelmän EN 12697-12 mukaisesti?

Vastaajien määrä: 5

	n	Prosentti
vain tyyppitestauksen massan suunnitteluvaiheessa	5	100%
1x vuodessa	0	0%
1x 2 vuodessa	0	0%
1x 3 vuodessa	0	0%
1x 4 vuodessa	0	0%
1x 5 vuodessa	0	0%

41. Kysymykset liittyen menettelyyn, joka tunnetaan nimellä ITSR-testi (EN 12697-12 menetelmä A)

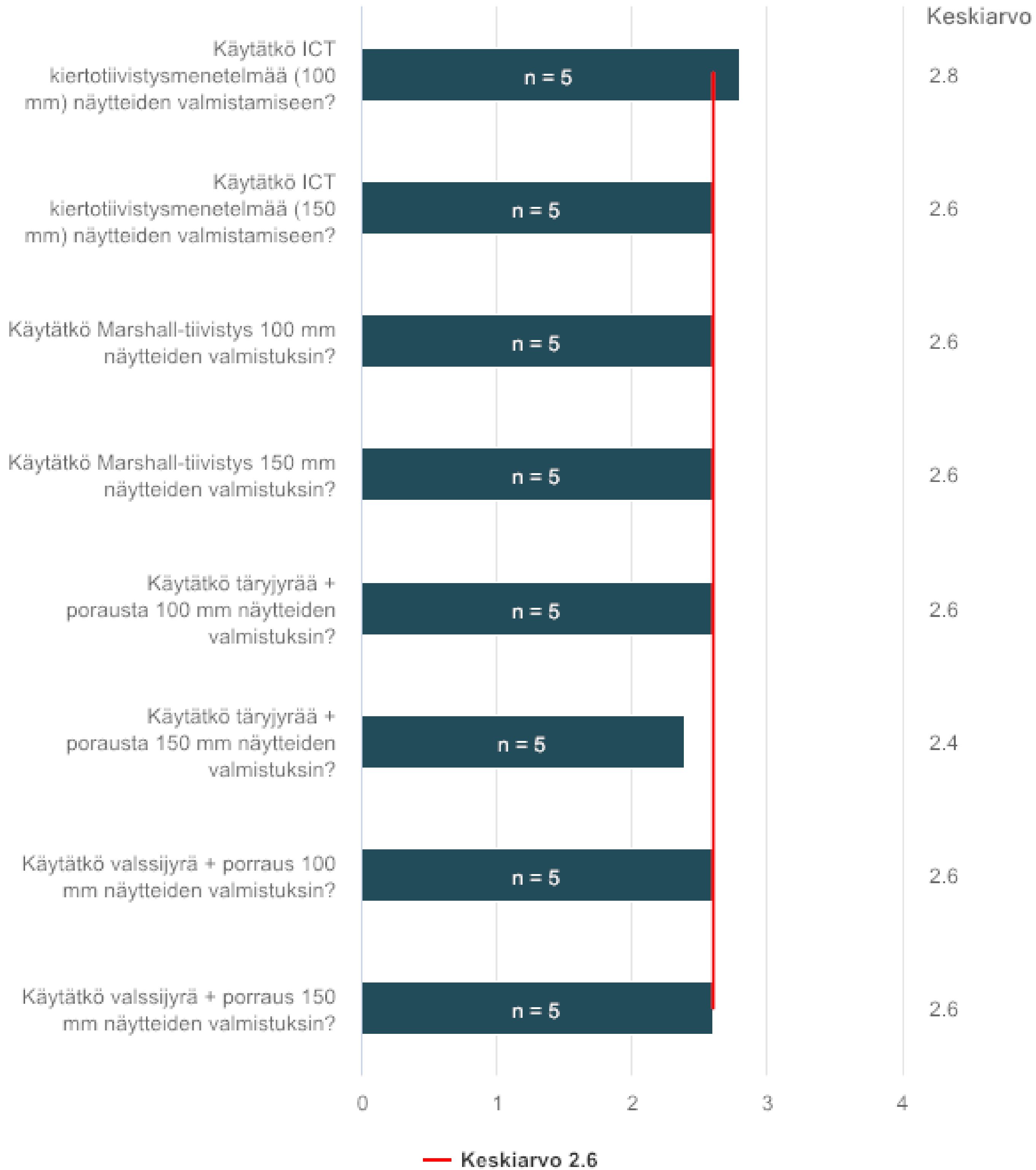
Vastaajien määrä: 5


Keskiarvo
Käytätkö AB-seosten tyyppitestissäsi jäätymis- ja (menetelmä PANK 4306)?
n = 5
2.0
sulamiskestävyyttä
Käytätkö AB-seosten jäätymis- ja (menetelmä PANK 4306)?
n = 5
2.0
suunn
suunnittelussa
Käytätkö SMA-seosten tyyppitestissäsi jäätymis- ja (menetelmä PANK 4306)?
n = 5
2.0
sulamiskestävyystestiä
Käytätkö SMA-seosten su jäätymis- ja sulamiskestävyystestiä (menetelmä PANK 4306)?
n = 5
2.0
Hallitsetko näytteen korkeuseron kuivassa ja märässä ryhmässä +/-5 mm: n sisällä?
n = 5
1.8
Kun verrataan kahta AB-seosta, (erilaiset kiviainekset), käytätkö molemmissa tapauksissa samaa näytteen korkeutta?
n = 5
1.8
Onko näytteen korkeus suhteessa testatun seoksen maksimitiheyteen? (käytätko aina vakiomassa, esim. 1000 g)
n = 5
1.8
Hallitsetko veden kyllästymisastetta märässä näytteessä?
n = 5
1.8
Varmistatko, että sideainepitoisuus on kuiva- ja märkä kokeissa, sallitulla alueella?
n = 5
1.6
Hallitsetko tyhjätilän kaikissa testeissä? (esim. jos testataan 2 eri massa, onko molemilla saman verran tyhjätilaa?) (EN 12697-12, 6.2.3a)
n = 5
2.0
Tiivistätkö kiinteää määrää iskuja /
n = 5
1.6
näytteet käyttämä
0
2
Keskiarvo 1.9

	Kyllä	Ei	Keskiarvo	Mediaani
Käytätkö AB-seosten tyyppitestissäsi jäätymis- ja sulamiskestävyyttä (menetelmä PANK 4306)?	0%	100%	2	2
Käytätkö AB-seosten suunnittelussa jäätymis- ja suunnittelussa (menetelmä PANK 4306)?	0%	100%	2	2
Käytätkö SMA-seosten tyyppitestissäsi jäätymis- ja sulamiskestävyystestiä (menetelmä PANK 4306)?	0%	100%	2	2
Käytätkö SMA-seosten susuunnittelussa jäätymis- ja sulamiskestävyystestiä (menetelmä PANK 4306)?	0%	100%	2	2
Hallitsetko näytteen korkeuseron kuivassa ja märässä ryhmässä +/-5 mm: n sisällä?	20%	80%	1.8	2
Kun verrataan kahta AB-seosta, (esim. erilaiset kiviainekset), käytätkö molemmissa tapauksissa samaa näytteen korkeutta?	20%	80%	1.8	2
Onko näytteen korkeus suhteessa testatun seoksen maksimitiheyteen? (käytätko aina vakiomassa, esim. 1000 g)	20%	80%	1.8	2
Hallitsetko veden kyllästymisastetta märässä näytteessä?	20%	80%	1.8	2
Varmistatko, että sideainepitoisuus on kuiva- ja märkä kokeissa, sallitulla alueella?	40%	60%	1.6	2
Hallitsetko tyhjätilän kaikissa testeissä? (esim. jos testataan 2 eri massa, onko molemilla saman verran tyhjätilaa?) (EN 12697-12, 6.2.3a)	0%	100%	2	2
Tiivistätkö näytteet käyttämällä kiinteää määrää iskuja / kiertoliikkeitä / jne. ? (EN 12697-12, 6.2.3.b)	40%	60%	1.6	2

42. Näytteen valmistusmenetelmä käytettäväksi ITSR-testeissä

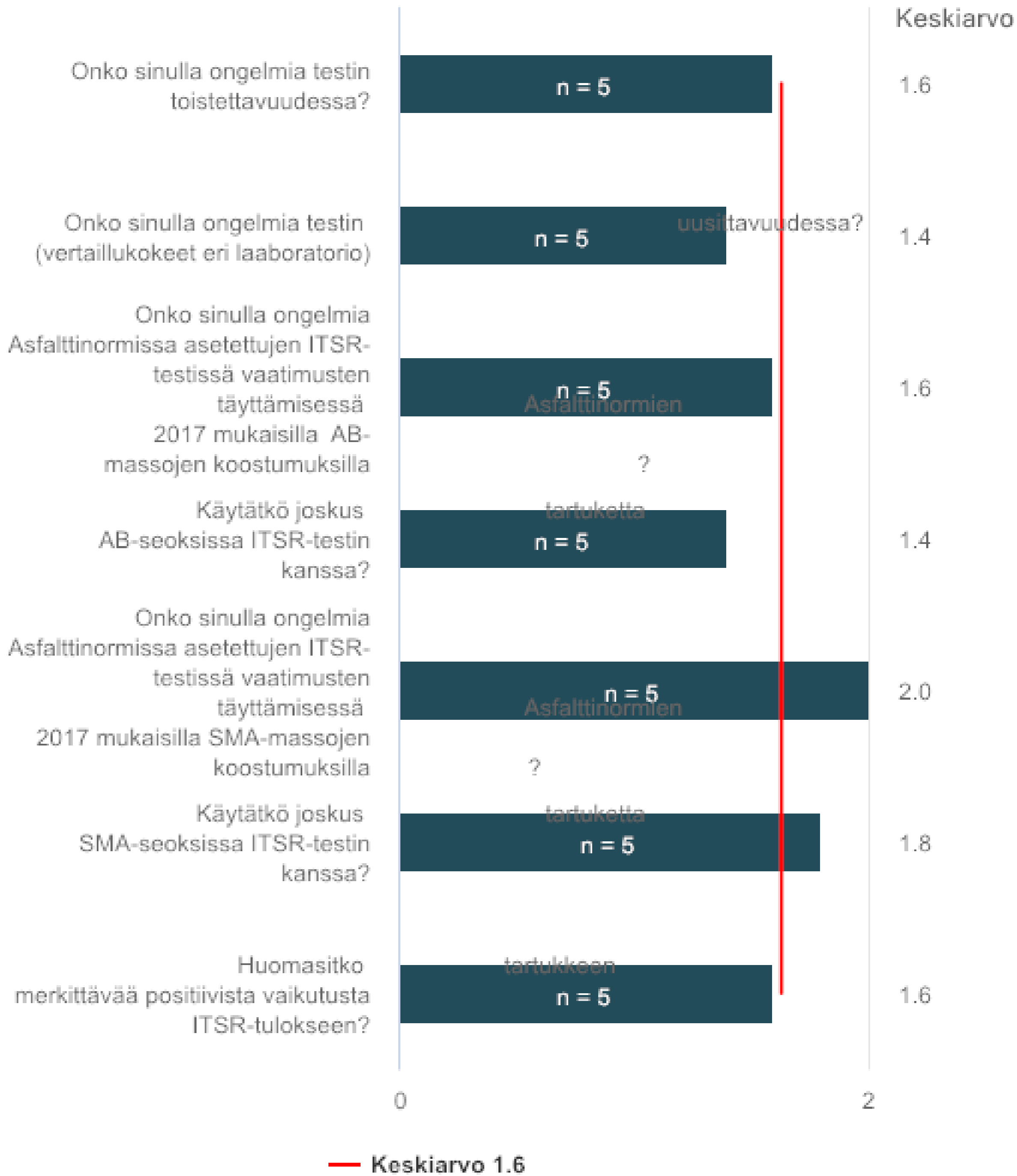
Vastaajien määrä: 5



	Suomessa	Suomen ulkopuolella	Typpitestauksessa	Kaikki vastaukset	Keskiarvo	Mediaani
Käytätkö ICT kiertotiivistysmenetelmää (100 mm) näytteiden valmistamiseen?	20%	0%	60%	20%	2.8	3
Käytätkö ICT kiertotiivistysmenetelmää (150 mm) näytteiden valmistamiseen?	20%	20%	40%	20%	2.6	3
Käytätkö Marshall-tiivistys 100 mm näytteiden valmistuksin?	20%	20%	40%	20%	2.6	3
Käytätkö Marshall-tiivistys 150 mm näytteiden valmistuksin?	20%	20%	40%	20%	2.6	3
Käytätkö täryjyrää + porausta 100 mm näytteiden valmistuksin?	20%	0%	80%	0%	2.6	3
Käytätkö täryjyrää + porausta 150 mm näytteiden valmistuksin?	20%	20%	60%	0%	2.4	3
Käytätkö valssijyrä + porraus 100 mm näytteiden valmistuksin?	20%	20%	40%	20%	2.6	3
Käytätkö valssijyrä + porraus 150 mm näytteiden valmistuksin?	20%	20%	40%	20%	2.6	3

43. Muut kysymykset, jotka liittyvät testiin EN 12697-12 menetelmään A (ITSR)

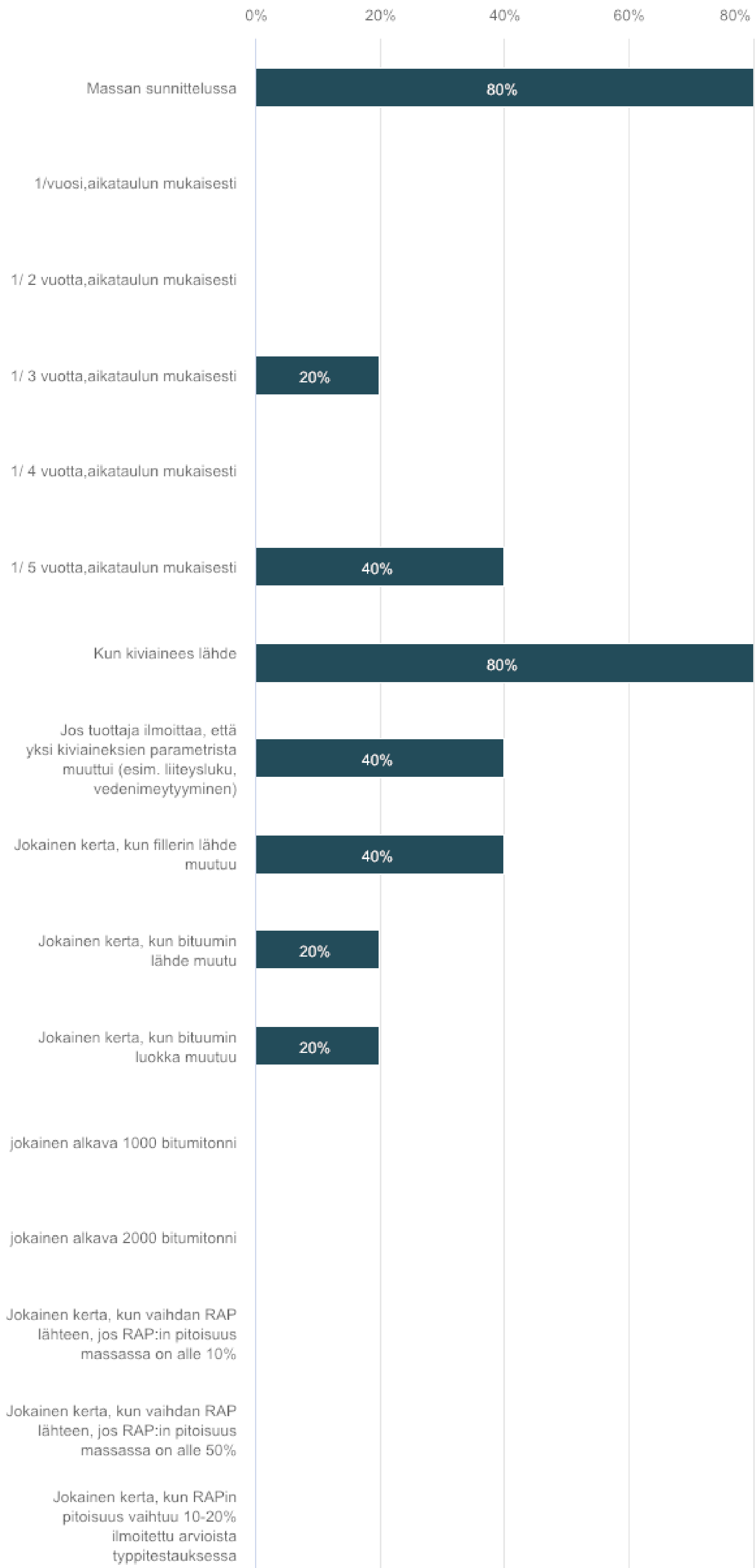
Vastaajien määrä: 5



	Kyllä	Ei	Keskiarvo	Mediaani
Onko sinulla ongelmia testin toistettavuudessa?	40%	60%	1.6	2
Onko sinulla ongelmia testin uusittavuudessa? (vertaillukokeet eri laaboratorio)	60%	40%	1.4	1
Onko sinulla ongelmia Asfalttinormissa asetettujen ITSR-testissä vaatimusten täyttämisessä Asfalttinormien 2017 mukaisilla AB-massojen koostumuksilla?	40%	60%	1.6	2
Käytätkö joskus tartuketta AB-seoksissa ITSR-testin kanssa?	60%	40%	1.4	1
Onko sinulla ongelmia Asfalttinormissa asetettujen ITSR-testissä vaatimusten täyttämisessä Asfalttinormien 2017 mukaisilla SMA-massojen koostumuksilla?	0%	100%	2	2
Käytätkö joskus tartuketta SMA-seoksissa ITSR-testin kanssa?	20%	80%	1.8	2
Huomasitko tartukkeen merkittävää positiivista vaikutusta ITSR-tulokseen?	40%	60%	1.6	2

44. Kuinka usein teet vedenkestävyyskokeita 12697-12-menetelmän A mukaisesti AB-seoksille?

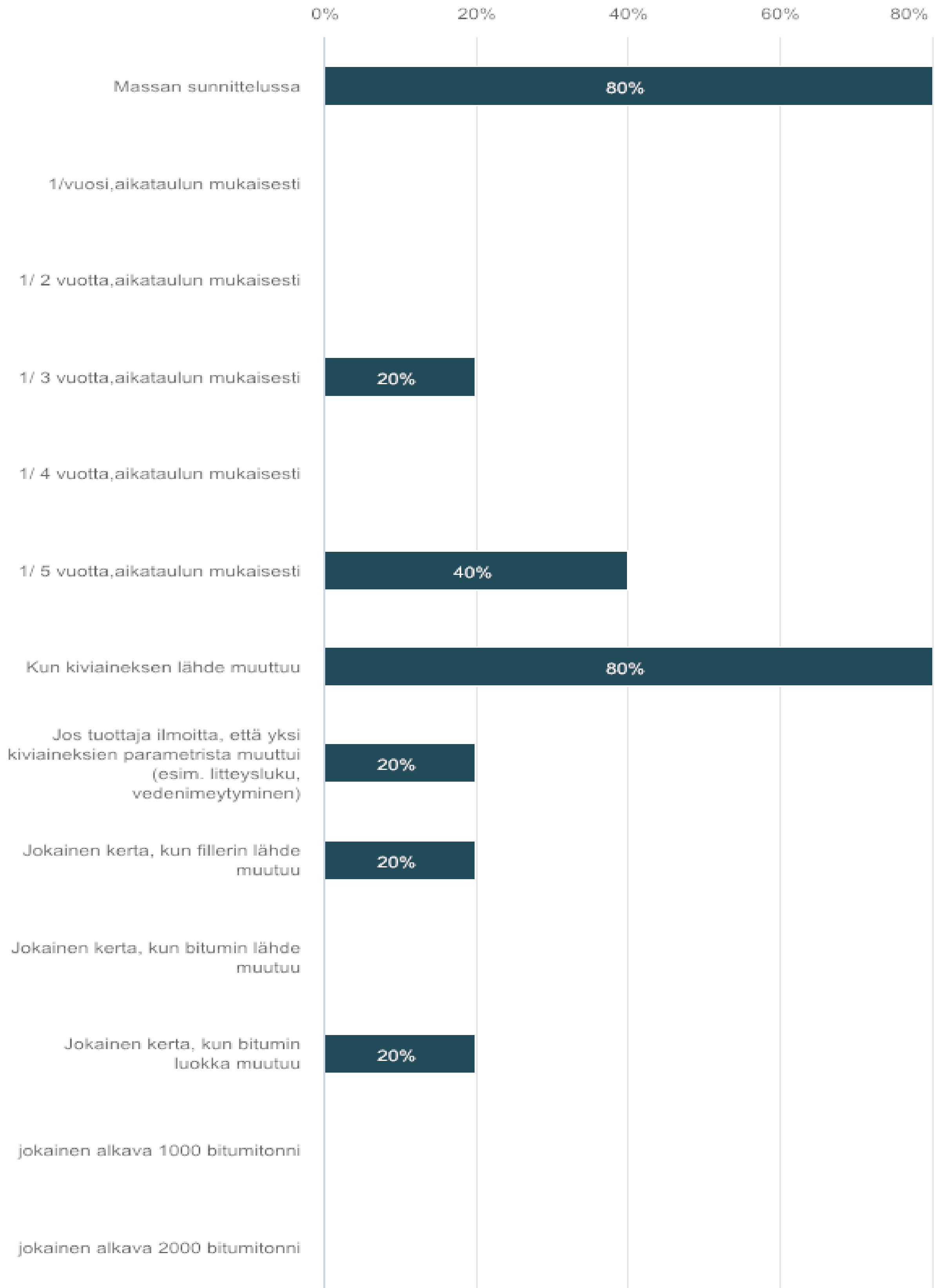
Vastaajien määrä: 5, valittujen vastausten lukumäärä: 17


0%
20%
40%
60%
80%
Massan sunnittelussa
80%
1/vuosi,aikataulun mukaisesti
1/ 2 vuotta,aikataulun mukaisesti
1/ 3 vuotta,aikataulun mukaisesti
20%
1/ 4 vuotta,aikataulun mukaisesti
1/ 5 vuotta,aikataulun mukaisesti
40%
Kun kiviainees lähde
80%
Jos tuottaja ilmoittaa, että yksi kiviaineksien parametrista muuttui (esim. liiteysluku, vedenimeytyyminen)
40%
Jokainen kerta, kun fillerin lähde muutuu
40%
Jokainen kerta, kun bituumin lähde muutu
20%
Jokainen kerta, kun bituumin luokka muutuu
20%
jokainen alkava 1000 bitumitonni
jokainen alkava 2000 bitumitonni
Jokainen kerta, kun vaihdan RAP lähteen, jos RAP:in pitoisuus massassa on alle 10%
Jokainen kerta, kun vaihdan RAP lähteen, jos RAP:in pitoisuus massassa on alle 50%
Jokainen kerta, kun RAPin pitoisuus vaihtuu 10-20% ilmoitettu arvioista typpitestauksessa

	n	Prosentti
Massan sunnittelussa	4	80%
1/vuosi,aikataulun mukaisesti	0	0%
1/ 2 vuotta,aikataulun mukaisesti	0	0%
1/ 3 vuotta,aikataulun mukaisesti	1	20%
1/ 4 vuotta,aikataulun mukaisesti	0	0%
1/ 5 vuotta,aikataulun mukaisesti	2	40%
Kun kiviainees lähde muutuu	4	80%
Jos tuottaja ilmoittaa, että yksi kiviaineksien parametrista muuttui (esim. liiteysluku, vedenimeytyyminen)	2	40%
Jokainen kerta, kun fillerin lähde muutuu	2	40%
Jokainen kerta, kun bituumin lähde muutu	1	20%
Jokainen kerta, kun bituumin luokka muutuu	1	20%
jokainen alkava 1000 bitumitonni	0	0%
jokainen alkava 2000 bitumitonni	0	0%
Jokainen kerta, kun vaihdan RAP lähteen, jos RAP:in pitoisuus massassa on alle 10%	0	0%
Jokainen kerta, kun vaihdan RAP lähteen, jos RAP:in pitoisuus massassa on alle 50%	0	0%
Jokainen kerta, kun RAPin pitoisuus vaihtuu 10-20% ilmoitettu arvioista typpitestauksessa	0	0%

45. Kuinka usein teet vedenkestävyykokeita 12697-12-menetelmän A mukaisesti SMA-seoksille?

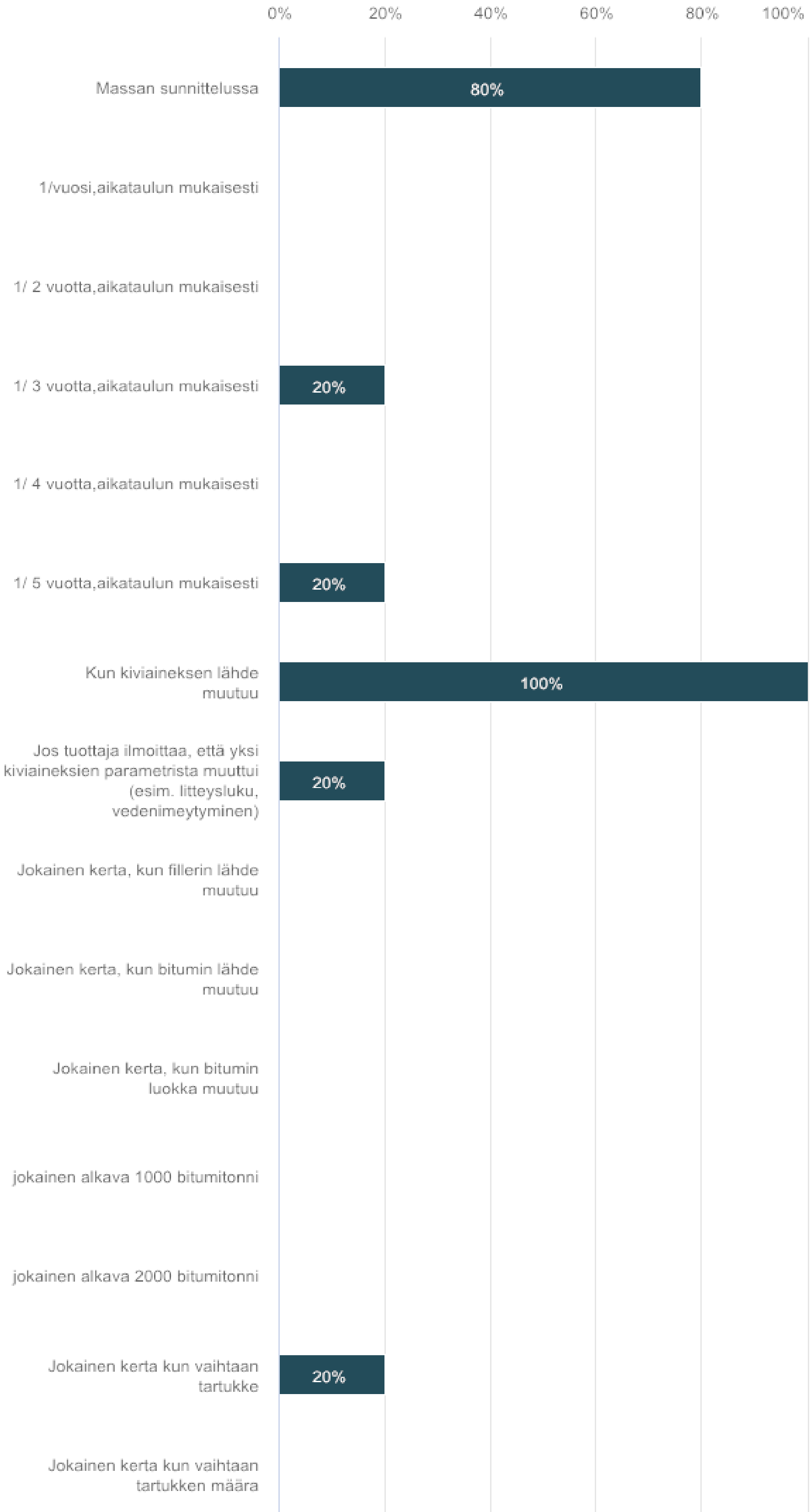
Vastaajien määrä: 5, valittujen vastausten lukumäärä: 14



	n	Prosentti
Massan sunnittelussa	4	80%
1/vuosi,aikataulun mukaisesti	0	0%
1/ 2 vuotta,aikataulun mukaisesti	0	0%
1/ 3 vuotta,aikataulun mukaisesti	1	20%
1/ 4 vuotta,aikataulun mukaisesti	0	0%
1/ 5 vuotta,aikataulun mukaisesti	2	40%
Kun kiviaineksen lähde muuttuu	4	80%
Jos tuottaja ilmoitta, että yksi kiviaineksien parametrista muuttui (esim. litteysluku, vedenimeytyminen)	1	20%
Jokainen kerta, kun fillerin lähde muutuu	1	20%
Jokainen kerta, kun bitumin lähde muutuu	0	0%
Jokainen kerta, kun bitumin luokka muutuu	1	20%
jokainen alkava 1000 bitumitonni	0	0%
jokainen alkava 2000 bitumitonni	0	0%

46. Kuinka usein testaat PAB-massan vedenkestävyyden menetelmällä 12697-12 A käyttäen PANK 4301-menetelmän mukaista AA-rakeisuutta ja bitumia 70/100?

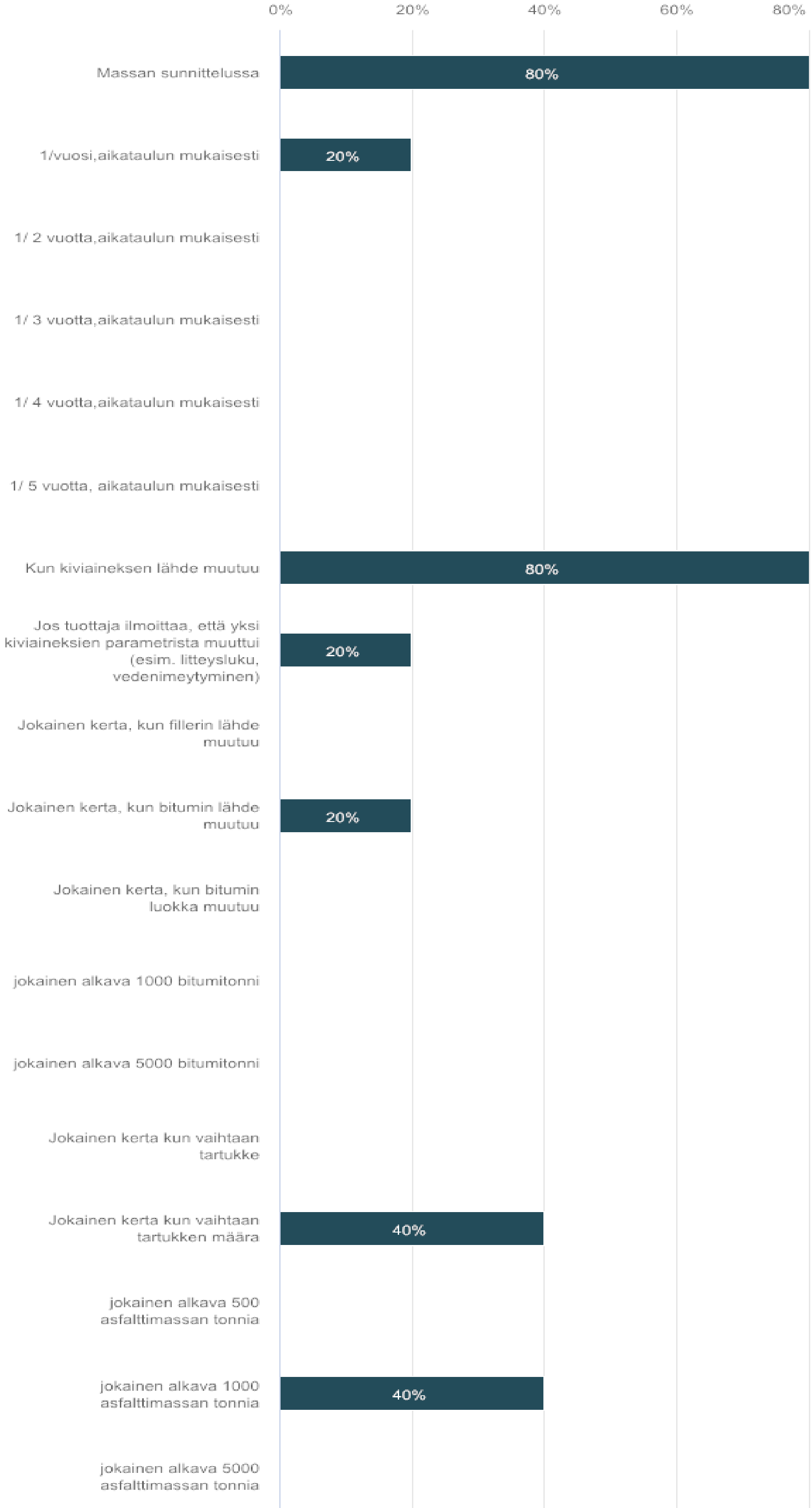
Vastaajien määrä: 5, valittujen vastausten lukumäärä: 13



	n	Prosentti
Massan sunnittelussa	4	80%
1/vuosi,aikataulun mukaisesti	0	0%
1/ 2 vuotta,aikataulun mukaisesti	0	0%
1/ 3 vuotta,aikataulun mukaisesti	1	20%
1/ 4 vuotta,aikataulun mukaisesti	0	0%
1/ 5 vuotta,aikataulun mukaisesti	1	20%
Kun kiviaineksen lähde muutuu	5	100%
Jos tuottaja ilmoittaa, että yksi kiviaineksien parametrista muuttui (esim. litteysluku, vedenimeytyminen)	1	20%
Jokainen kerta, kun fillerin lähde muutuu	0	0%
Jokainen kerta, kun bitumin lähde muutuu	0	0%
Jokainen kerta, kun bitumin luokka muutuu	0	0%
jokainen alkava 1000 bitumitonni	0	0%
jokainen alkava 2000 bitumitonni	0	0%
Jokainen kerta kun vaihtaan tartukke	1	20%
Jokainen kerta kun vaihtaan tartukken määra	0	0%

47. Kuinka usein teet vedenkestavyystestejä EN 12697-12-menetelmän C (MYR-koe) mukaisesti PAB-seoksille?

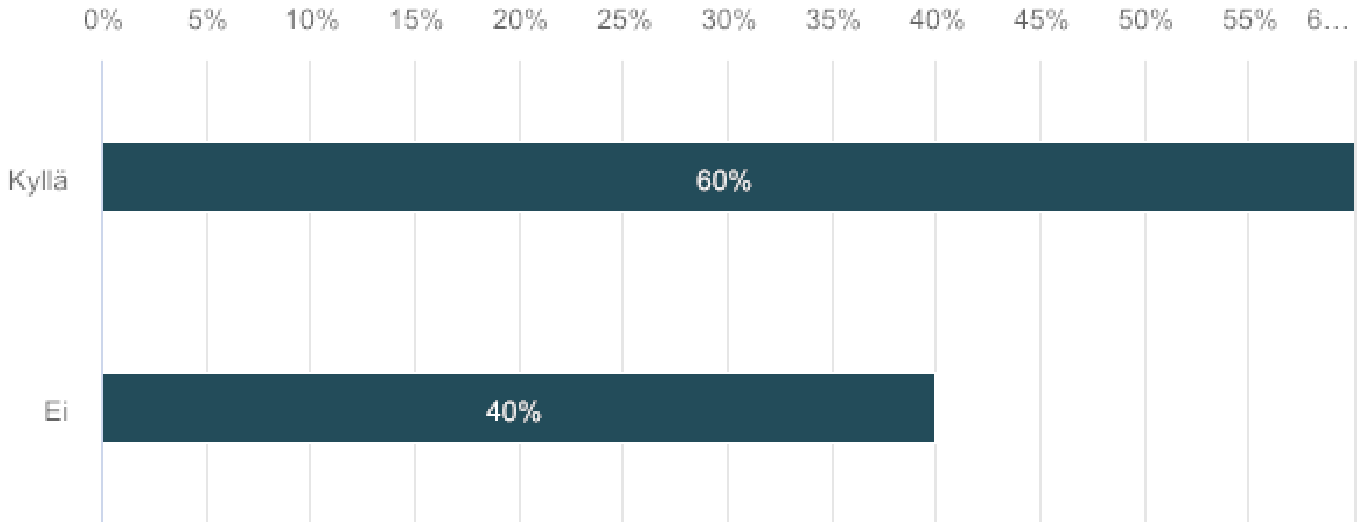
Vastaajien määrä: 5, valittujen vastausten lukumäärä: 15



	n	Prosentti
Massan sunnittelussa	4	80%
1/vuosi,aikataulun mukaisesti	1	20%
1/ 2 vuotta,aikataulun mukaisesti	0	0%
1/ 3 vuotta,aikataulun mukaisesti	0	0%
1/ 4 vuotta,aikataulun mukaisesti	0	0%
1/ 5 vuotta, aikataulun mukaisesti	0	0%
Kun kiviaineksen lähde muutuu	4	80%
Jos tuottaja ilmoittaa, että yksi kiviaineksien parametrista muuttui (esim. litteysluku, vedenimeytyminen)	1	20%
Jokainen kerta, kun fillerin lähde muutuu	0	0%
Jokainen kerta, kun bitumin lähde muutuu	1	20%
Jokainen kerta, kun bitumin luokka muutuu	0	0%
jokainen alkava 1000 bitumitonni	0	0%
jokainen alkava 5000 bitumitonni	0	0%
Jokainen kerta kun vaihtaan tartukke	0	0%
Jokainen kerta kun vaihtaan tartukken määra	2	40%
jokainen alkava 500 asfalttimassan tonnia	0	0%
jokainen alkava 1000 asfalttimassan tonnia	2	40%
jokainen alkava 5000 asfalttimassan tonnia	0	0%

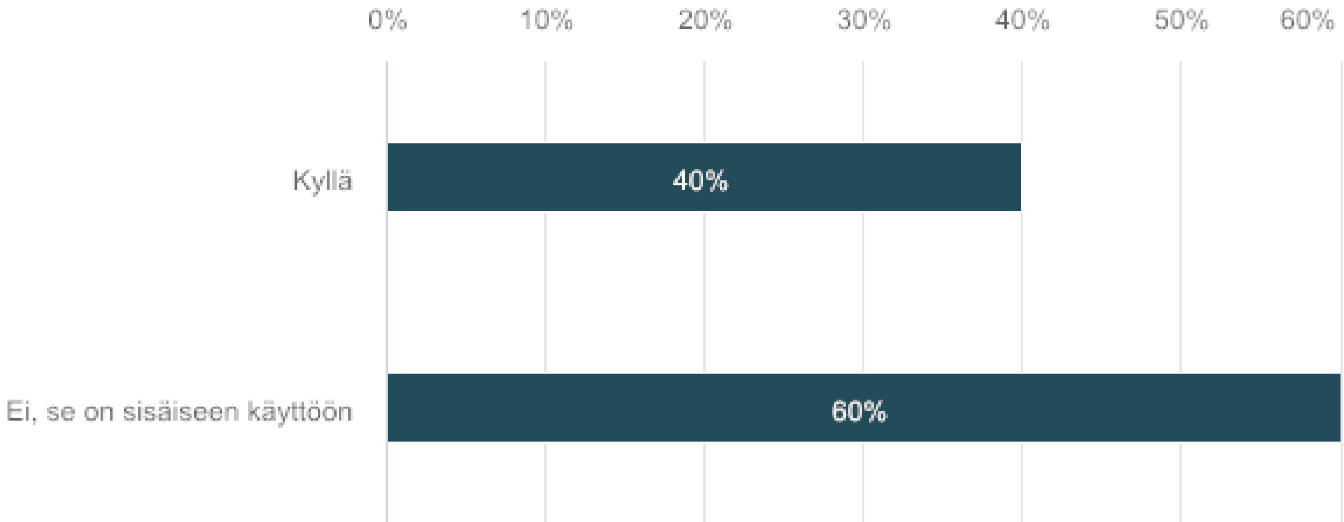
48. Teetkö sankokokeen tuotannon aikana?

Vastaajien määrä: 5



	n	Prosentti
Kyllä	3	60%
Ei	2	40%

49. Raportoitko sankokokeen tulokset johonkin?
Vastaajien määrä: 5



	n	Prosentti
Kyllä	2	40%
Ei, se on sisäiseen käyttöön	3	60%

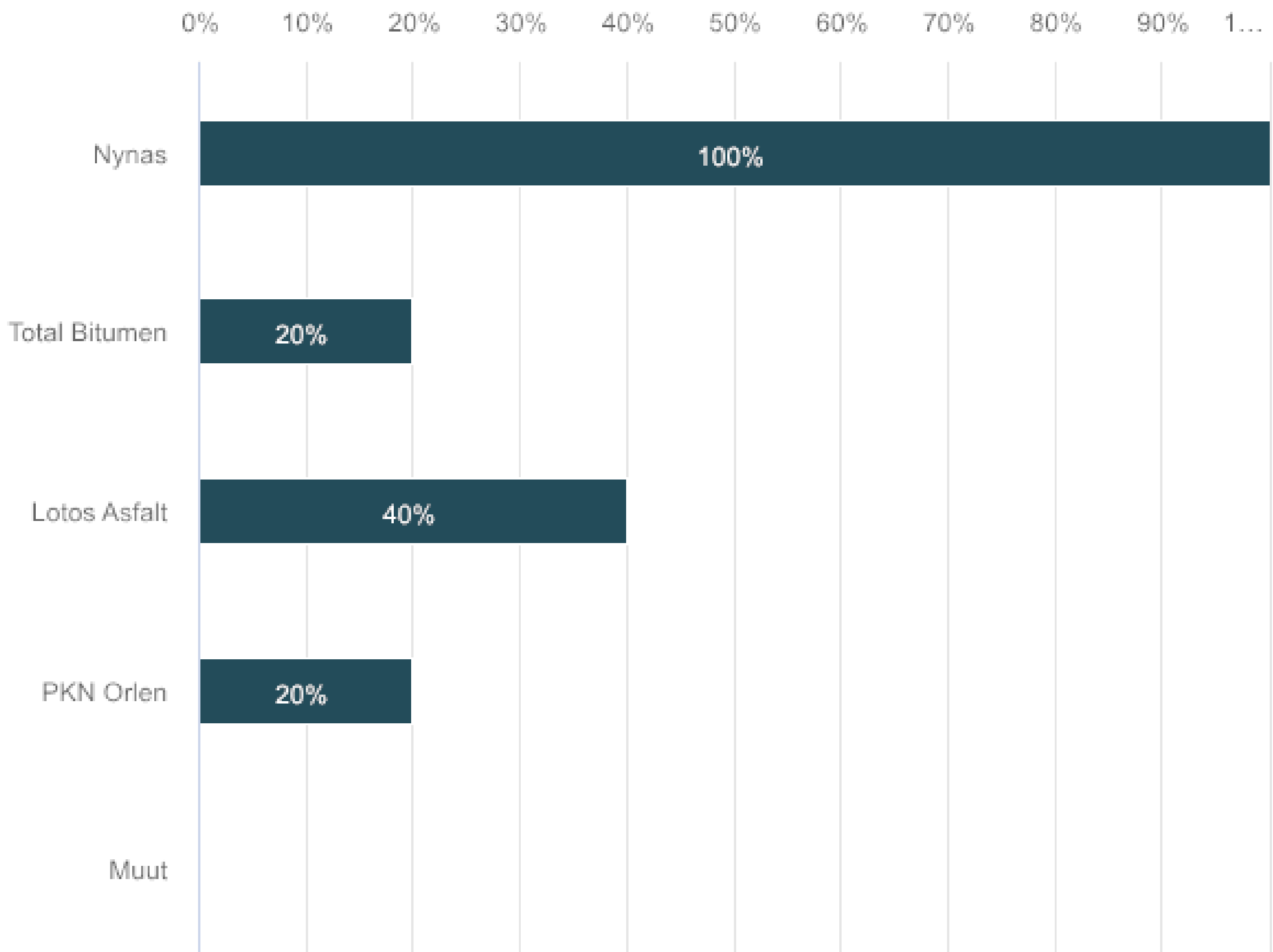
50. Mitkä tartukkeet ja muut asfalttimodifioijat ovat sinulle tuttuja (käytetty aikaisemmin tai olleet yhteydessä toimittajien/tuottajien kanssa)? Jos et tiedä tarttuvuuden edistäjän kemiallista koostumusta, mutta tiedät vain tuotteen merkin tai nimen, lisää se "muuhun".Tämän kysymyksen tavoitteena on antaa tietoa tutkimussuunnitteluun tulevaisuudessa. Voit ohittaa kysymyksen, jos pidät sitä arkaluontoisena tietona.Tämä kysymys on vapaaehtoinen.

Vastaajien määrä: 1

aminipohjainen, nimet/tuottajat:	polyfosforihappo, nimi/tuottajat:	sammutettu kalkki, nimi/tuottajat:	orgaanosilaanit, nimi/tuottajat:	vahat, nimi/tuottajat:	zeoliitti, nimi/tuottajat:	polymeeri, nimi/tuottajat:	elvyttimet, nimi/tuottajat:	kumirouhe, nimi/tuottajat:	polymeerikuitu, nimi/tuottajat:	muut, nimi/tuottajat:
										Kalkki kivifilleri, Nordkalk

51. Mitkä alla olevista bitumin toimittajista ovat toimittaneet bitumin sinulle? Tämän kysymyksen tavoitteena on antaa tietoa tutkimussuunnitteluun tulevaisuudessa. Voit ohittaa kysymyksen, jos pidät sitä arkaluontoisena tietona.

Vastaajien määrä: 5, valittujen vastausten lukumäärä: 9



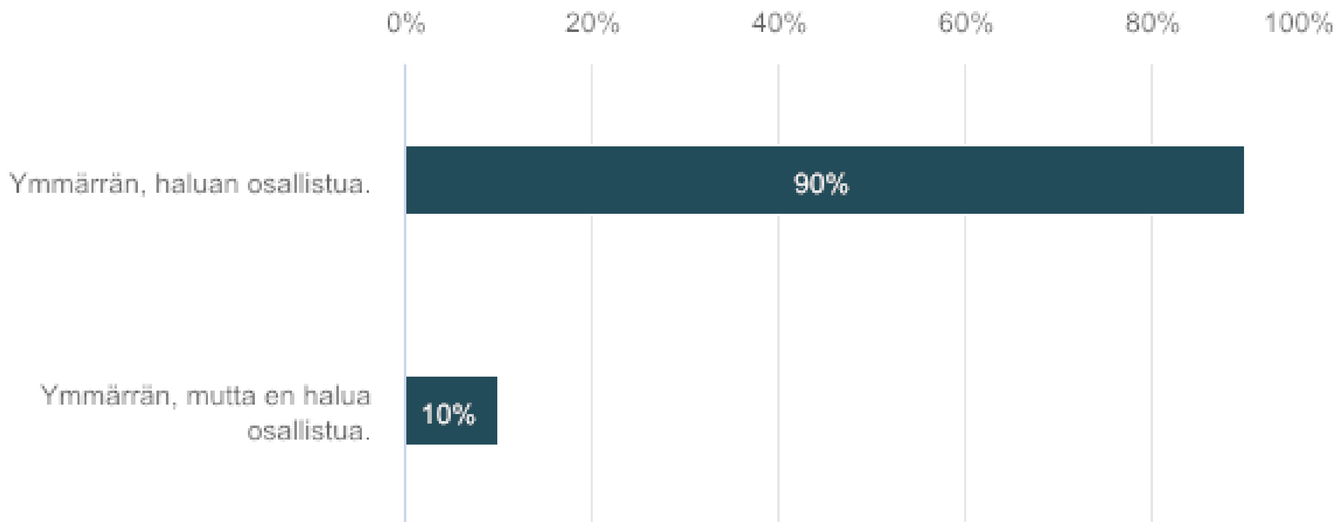
	n	Prosentti
Nynas	5	100%
Total Bitumen	1	20%
Lotos Asfalt	2	40%
PKN Orlen	1	20%
Muut	0	0%

Avoimeen tekstikenttään annetut vastaukset

Vastausvaihtoehdot	Teksti

52. Tutkimukseen Project BITU 2020 osallistuva täyttääOlen ymmärtänyt, että tutkimukseen osallistuminen on vapaaehtoista ja voin milloin tahansa ilmoittaa, etten enää halua osallistua tutkimukseen, mutta siihen asti kerättyjä tutkimusaineistoja voidaan hyödyntää tutkimuksessa tutkimuksen tietosuojailmoituksen mukaisesti. Ymmärrän, että yrityksen nimi voidaan julkaista julkaisujen ja tutkimusaineistojen yhteydessä listalla, jolla kaikki kutsutut yritykset on mainittu.Olen saanut riittävät tiedot tämän tutkimuksen tietosuojailmoituksesta, minulla on ollut mahdollisuus saada vastauksia kysymyksiini, olen ymmärtänyt saamani tiedot ja haluan osallistua tutkimukseen.Yhteystiedot:Michalina Makowska, +358 50 431 8103, michalina.makowska@aalto.fiAalto-yliopisto

Vastaajien määrä: 10



	n	Prosentti
Ymmärrän, haluan osallistua.	9	90%
Ymmärrän, mutta en halua osallistua.	1	10%

The effect of adhesion promotor on results of the Rolling Bottle Test during ASTO

The pictures are reprinted from [20] and present the degree of coverage by bitumen in reference (upper row on each page) and the comparison to the samples with modification by various adhesion promotors. The products were either proposed by producers or already used in trials in Finland.

The information provided in the report in respect of the used additives are provided below:

1. RAISAMIN DT

n-tall propylene diamine, the product contains long chain fatty amines (C16-C18) paste with flame point of 190°C, melting point of 40-45°C, density 0,82 kg/m3 non-soluble in water

2. RAISAMIN 200

Alkyl- and alkylenepolyamine mixture, containing long chain alkenylamines (C16-C18), Brown liquid, flame point of 130 °C, melting point around 5°C, density 0.945 kg/m3, viscosity 180-220 cP
Partially soluble in water.

3. Amine D

Large amine mixture obtained from modification of resins. Example of the amine inside was dehydrobiethylamine. The product is stable under 100°C, the viscosity at 25°C is 87 Pas and its flame point is 192°C. The amine content in the product is 92%, of which 3% are secondary amines.

4. Valke A – Mäntyhartsi (Tall resin)

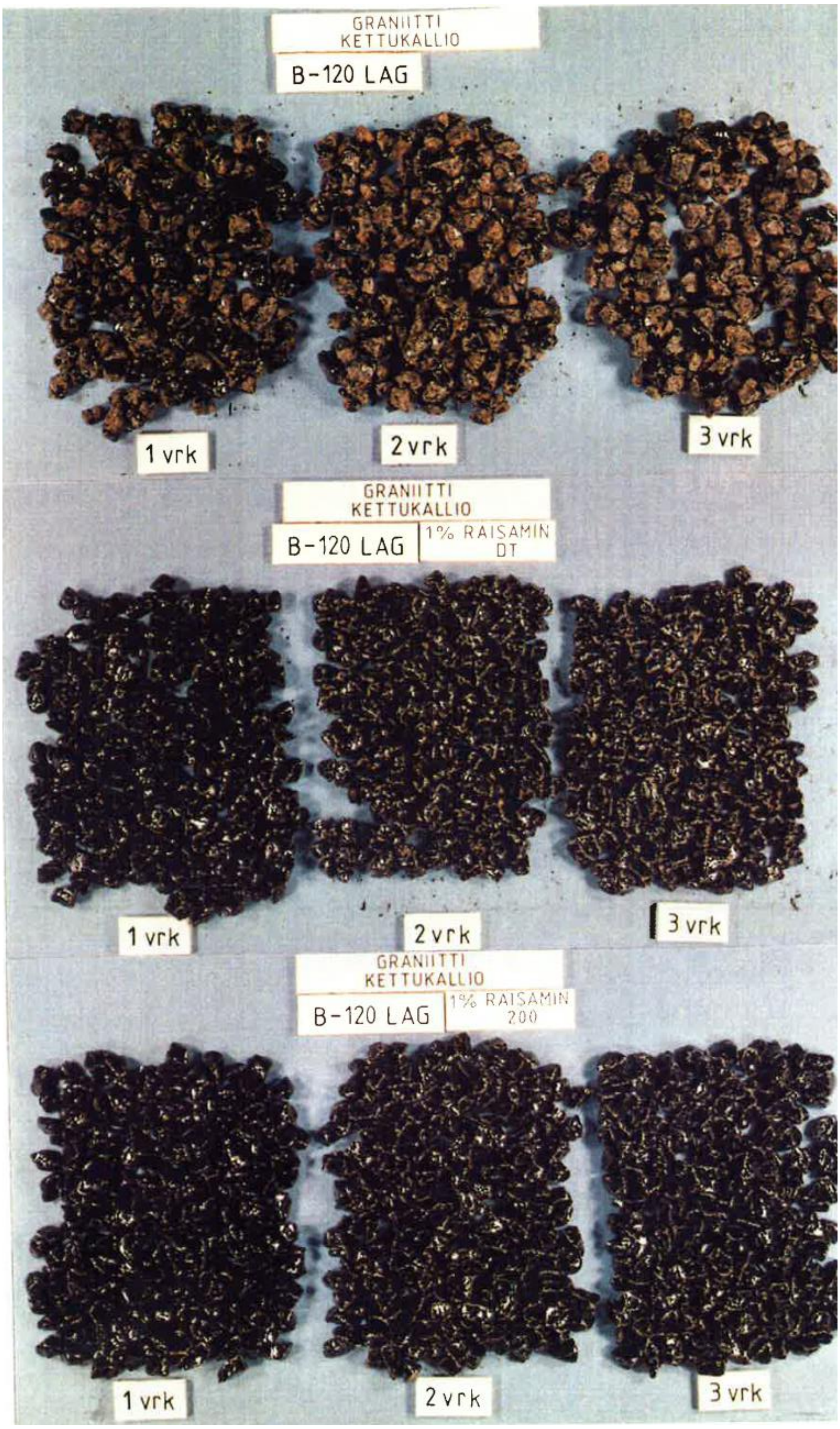
83% resin acids and 4% fatty acids. The acidic number of the product is 162 mg KOH/g. Softening point is 63°C

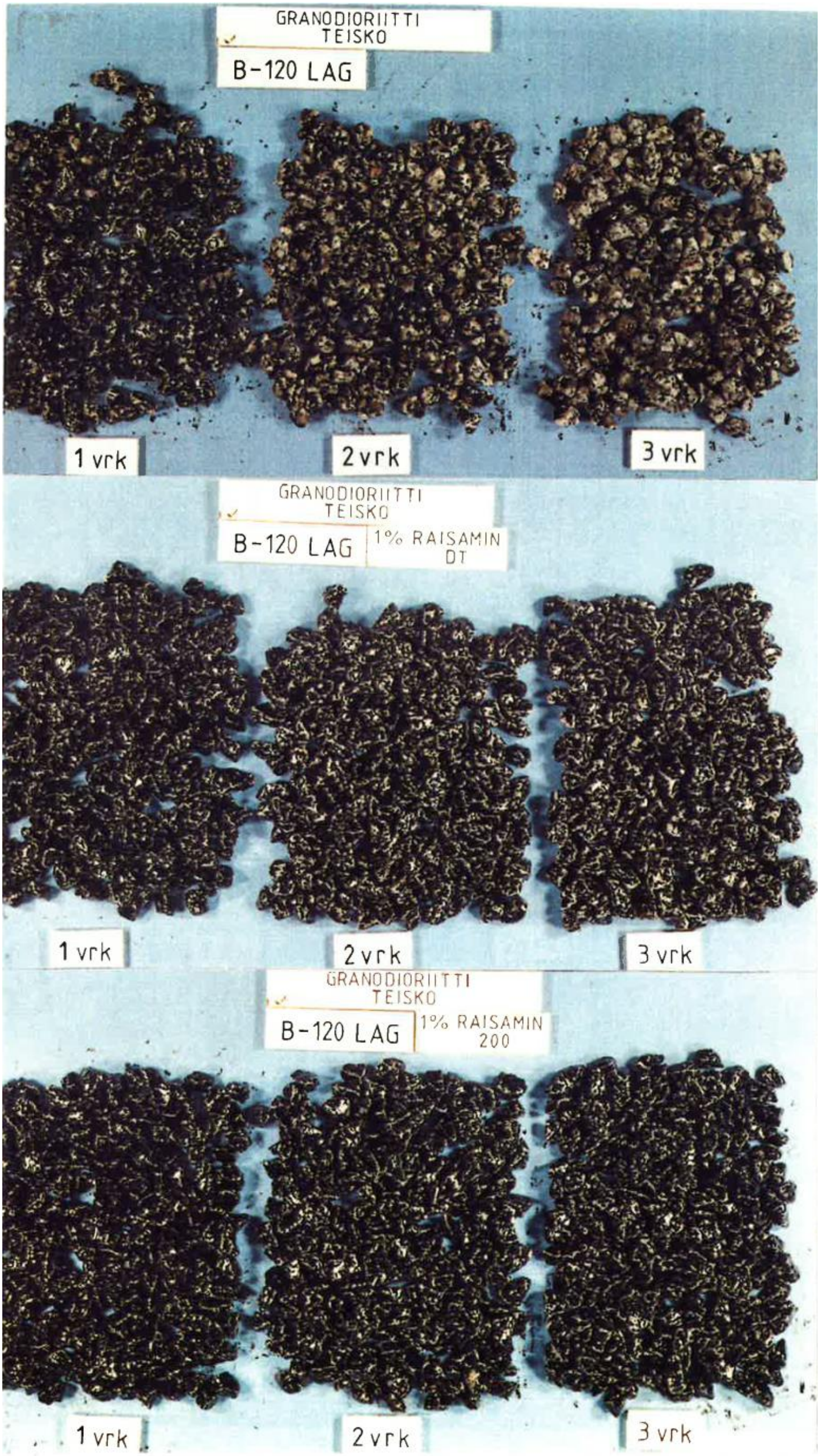
5. Vampula - limestone filler (CaCO3)

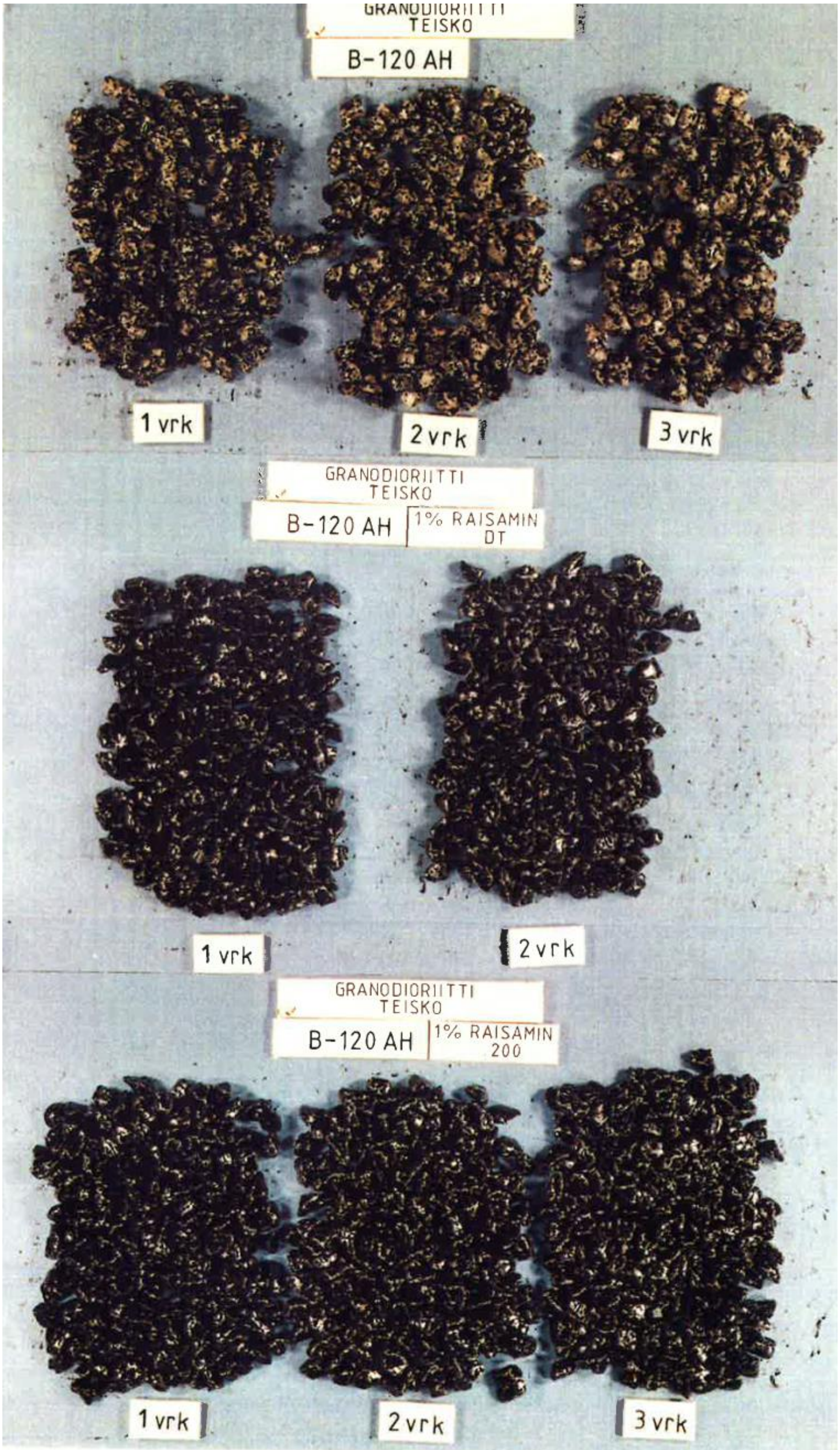
Acidity 3,6 µmol/g and 0,6 µmol/m2
Solubility in hydrochloric acid >80%

6. Tytyri - industrial fine lime Ca(OH)2

Acidity 3,96 µmol/g and 2,19 µmol/m2


GRANIITTI
KETTUKALLIO
B-120 LAG
1 vrk
2 vrk
3 vrk
GRANIITTI
KETTUKALLIO
B-120 LAG
1% RAISAMIN DT
1 vrk
2 vrk
3 vrk
GRANIITTI
KETTUKALLIO
B-120 LAG
1% RAISAMIN 200
1 vrk
2 vrk
3 vrk

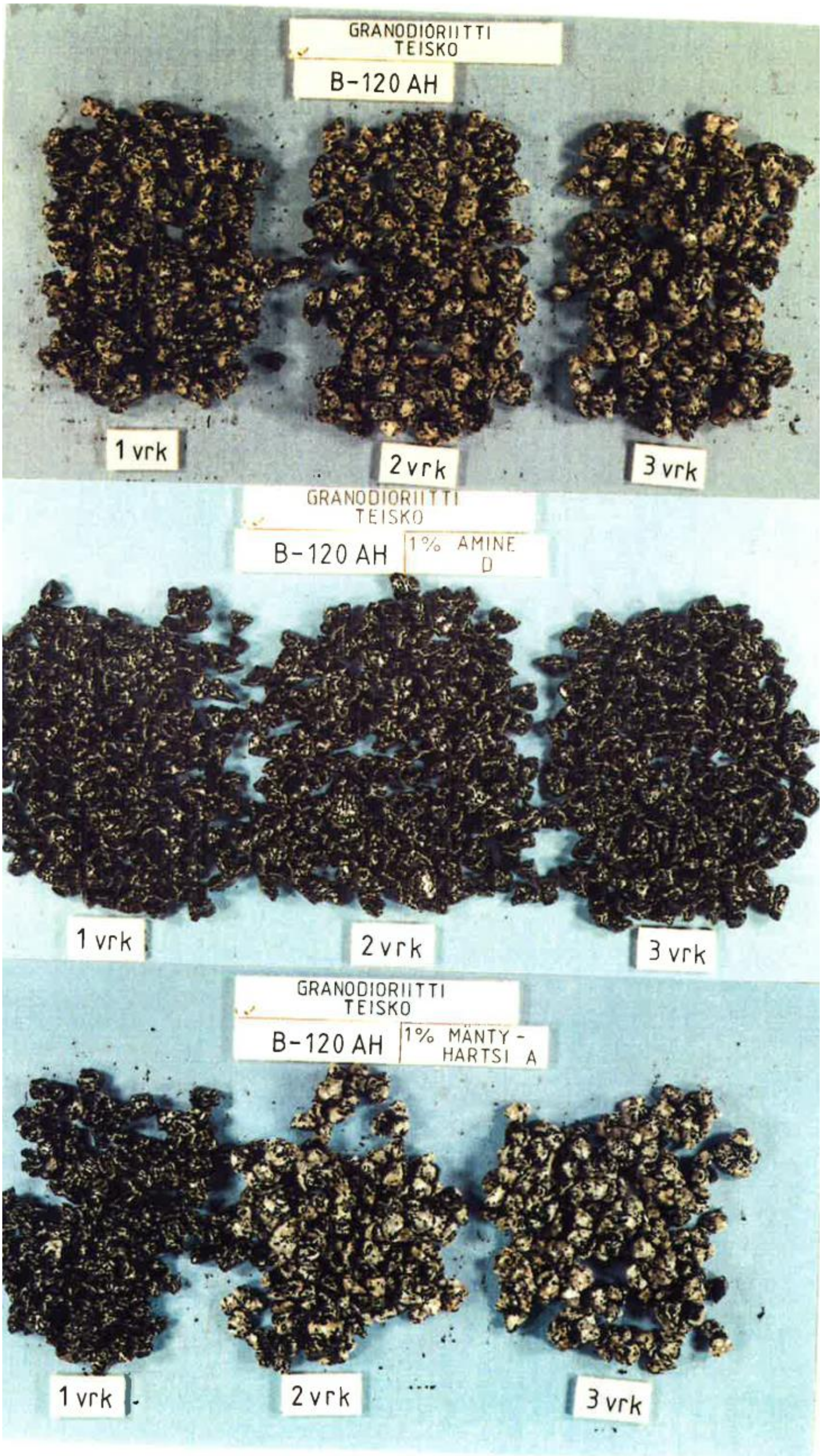

GRANODIORIITTI
TEISKO
B-120 LAG
1 vrk
2 vrk
3 vrk
GRANODIORIITTI
TEISKO
B-120 LAG
1% RAISAMIN DT
1 vrk
2 vrk
3 vrk
GRANODIORIITTI
TEISKO
B-120 LAG
1% RAISAMIN 200
1 vrk
2 vrk
3 vrk

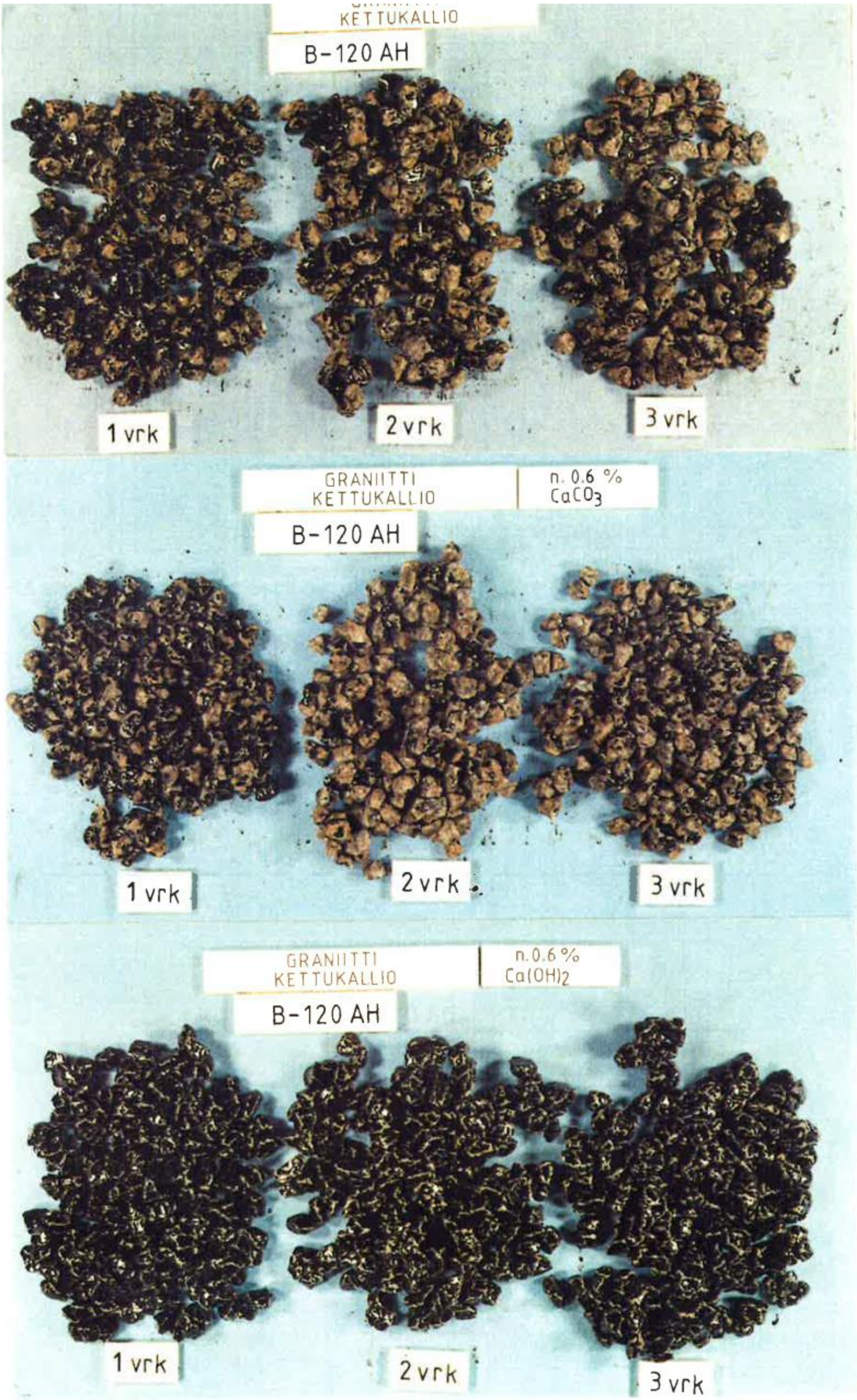

GRANODIORIITTI
TEISKO
B-120 AH
1 vrk
2 vrk
3 vrk
GRANODIORIITTI
TEISKO
B-120 AH
1% RAISAMIN
DT
1 vrk
2 vrk
GRANODIORIITTI
TEISKO
B-120 AH
1% RAISAMIN
200
1 vrk
2 vrk
3 vrk

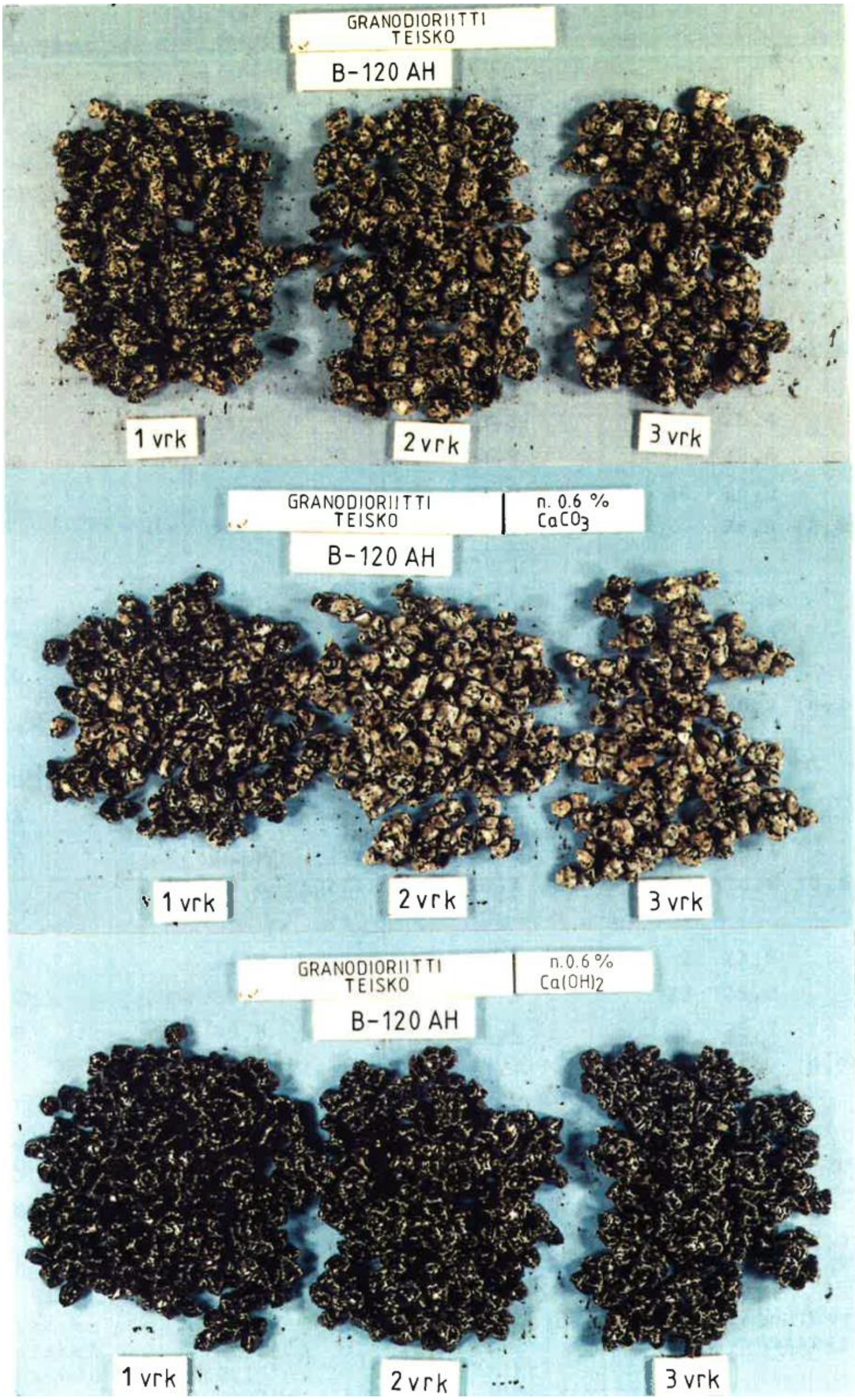

GRANIITTI
KETTUKALLIO
B-120 AH
1 vrk
2 vrk
3 vrk


GRANIITTI
KETTUKALLIO
B-120 AH
1% AMINE D
1 vrk
2 vrk
3 vrk


GRANIITTI
KETTUKALLIO
B-120 AH
1% MÄNTY-HARTSI A
1 vrk
2 vrk
3 vrk


GRANODIORIITTI
TEISKO
B-120 AH
1 vrk
2 vrk
3 vrk
GRANODIORIITTI
TEISKO
B-120 AH
1% AMINE D
1 vrk
2 vrk
3 vrk
GRANODIORIITTI
TEISKO
B-120 AH
1% MÄNTY-HARTSI A
1 vrk
2 vrk
3 vrk


KETTUKALLIO
B-120 AH
1 vrk
2 vrk
3 vrk
GRANIITTI
KETTUKALLIO
n. 0,6 %
$CaCO_3$
B-120 AH
1 vrk
2 vrk
3 vrk
GRANIITTI
KETTUKALLIO
n. 0,6 %
$Ca(OH)_2$
B-120 AH
1 vrk
2 vrk
3 vrk


GRANODIORIITTI
TEISKO
B-120 AH
1 vrk
2 vrk
3 vrk
GRANODIORIITTI
TEISKO
n. 0.6 %
$CaCO_3$
B-120 AH
1 vrk
2 vrk
3 vrk
GRANODIORIITTI
TEISKO
n.0.6 %
$Ca(OH)_2$
B-120 AH
1 vrk
2 vrk
3 vrk

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