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Electrical and Dielectric Properties of Uncoated and Coated Wood-Free Paper for Electrophotography



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PREFACE

The research summarized in this thesis has been conducted as a part of the research programs of Stora Enso Oyj to develop paper and board substrates for the electrophotographic printing processes in partnerships with several universities. The main part of the summarized research work has been done in Stora Enso's R&D facilities and in cooperation with Åbo Akademi University and Vilnius University. I would like to thank the personnel involved in these institutions for their advice, expertise, and knowledge.

In particular, I would like to thank professors Jarl B. Rosenholm and Jouko Peltonen from Åbo Akademi University, professor Kaj Backfolk from Lappeenranta University, and professors Jonas Sidaravicius and Robertas Maldzius from Vilnius University. The level of knowledge and experience accumulated into this group is indeed convincing and I am proud to have had the opportunity to work in such teams during the scientific part of my career. I would also like to thank all the authors and co-authors in the publications included in this thesis as well as those in the supporting publications. I am grateful for all the interesting work we have done, for all the interesting discussions, and for all that we have learned.

Stora Enso Oyj is acknowledged for its vision of exploring new areas. The Finnish Funding Agency for Innovation TEKES is acknowledged for the financial support for this research.

Finally, I would like to thank my wife Leena for her crucial support, which has made everything possible. I wish to dedicate this work to her.

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Publications I - VI

ABSTRACT

The print substrate influences the print result in dry toner electrophotography, which is a widely used digital printing method. The influence of the substrate can be seen more easily in color printing, as that is a more complex process compared to monochrome printing. However, the print quality is also affected by the print substrate in grayscale printing. It is thus in the interests of both substrate producers and printing equipment manufacturers to understand the substrate properties that influence the quality of printed images in more detail. In dry toner electrophotography, the image is printed by transferring charged toner particles to the print substrate in the toner transfer nip, utilizing an electric field, in addition to the forces linked to the contact between toner particles and substrate in the nip. The toner transfer and the resulting image quality are thus influenced by the surface texture and the electrical and dielectric properties of the print substrate.

In the investigation of the electrical and dielectric properties of the papers and the effects of substrate roughness, in addition to commercial papers, controlled sample sets were made on pilot paper machines and coating machines to exclude uncontrolled variables from the experiments. The electrical and dielectric properties of the papers investigated were electrical resistivity and conductivity, charge acceptance, charge decay, and the dielectric permittivity and losses at different frequencies, including the effect of temperature. The objective was to gain an understanding of how the electrical and dielectric properties are affected by normal variables in papermaking, including basis weight, material density, filler content, ion and moisture contents, and coating. In addition, the dependency of substrate resistivity on the electric field applied was investigated.

Local discharging did not inhibit transfer with the paper roughness levels that are normal in electrophotographic color printing. The potential decay of paper revealed that the charge decay cannot be accurately described with a single exponential function, since in charge decay there are overlapping mechanisms of conduction and depolarization of paper. The resistivity of the paper depends on the NaCl content and exponentially on moisture content although it is also strongly dependent on the electric field applied. This dependency is influenced by the thickness, density, and filler contents of the paper. Furthermore, the Poole-Frenkel model can be applied to the resistivity of uncoated paper.

The real part of the dielectric constant ϵ' increases with NaCl content and relative humidity, but when these materials cannot polarize freely, the increase cannot be explained by summing the effects of their dielectric constants. Dependencies between the dielectric constant and dielectric loss factor and NaCl content, temperature, and frequency show that in the presence of a sufficient amount of moisture and NaCl, new structures with a relaxation time of the order of 10^{-3} s are formed in paper. The ϵ' of coated papers is influenced by the addition of pigments and other coating additives with polarizable groups and due to the increase in density.

The charging potential decreases and the electrical conductivity, potential decay rate, and dielectric constant of paper increase with increasing temperature. The dependencies are exponential and the temperature dependencies and their activation energies are altered by the ion content.

The results have been utilized in manufacturing substrates for electrophotographic color printing.

SVENSK SAMMANFATTNING

Tryckunderlaget (pappret) påverkar utskriftsresultatet i elektrofotografi vilket är en allmänt använd digital tryckmetod. Effekten av substratets egenskaper kan lättare ses i färgtryck, eftersom det är en mer komplex process jämfört med svartvita utskrifter. Dock påverkas utskriftskvaliteten av trycksubstratet även i enfärgstryckning. Det är således av intresse för både substrattillverkare och tryckutrustningstillverkare att mer i detalj förstå de substrategenskaper som påverkar kvaliteten för tryckta bilder. I elektrofotografi trycks bilden genom att laddade färgpartiklar, s.k. toner överförs till trycksubstratet i färgöverföringsnypet med hjälp av ett elektriskt fält, utöver de krafter som är kopplade med kontakterna mellan tonerpartiklarna och substratet. Färgöverföringen och den resulterande bildkvaliteten påverkas således av ytstrukturen och trycksubstratets elektriska och dielektriska egenskaper.

I denna avhandling studerades pappers elektriska och dielektriska egenskaper samt effekterna av substratens ojämnheter på tryckkvaliteten. Förutom användningen av kommersiella papper gjordes kontrollerade provserier på pilotpappersmaskin och pilotbetrykare för att utesluta okontrollerade variabler från experimenten. Elektriska och dielektriska egenskaper som studerades hos de undersökta pappren var elektriskt resistivitet, ledningsförmåga, laddningsacceptans, laddningsavtagandet, samt dielektricitetskonstanten och dielektriska förluster vid olika frekvenser, omfattande även effekten av temperatur. Målet var att öka förståelsen för hur normala variabler i papperstillverkning, inklusive ytvikt, materialtäthet, fyllmedelshalt, jon- och fukthalter samt bestrykning påverkar de elektriska och dielektriska egenskaperna. Substratets resistivitets beroende av det pålagda elektriska fältet undersöktes också.

I studien framkom bland annat att en lokal urladdning inte hämmar överföringen av färg på papper med normal ytråhet. Minskningen av papprets potential visade att laddningsavtagandet inte kan beskrivas exakt med en enda exponentiell funktion på grund av överlappande mekanismer för elektrisk ledning och papprets depolarisering. Resistiviteten hos papperet beror av NaCl halten och exponentiellt av fukthalten, men dessutom starkt av det pålagda elektriska fältet och detta beroende är influerat av papprets tjocklek, täthet och av papprets fyllmedelshalt. Vidare framkom det ur studien att Poole-Frenkel-modellen kan tillämpas på resistiviteten (motståndsförmågan) hos obestruket papper. Den reella delen av dielektricitetskonstanten ϵ' ökar med NaCl innehållet och relativ fuktighet men eftersom dessa material inte kan polariseras fritt kan inte ökningen förklaras genom att summera effekterna av deras dielektricitetskonstanter. Beroenden mellan dielektricitetskonstanten, den dielektriska förlustfaktorn, NaCl-halten, temperaturen och frekvensen visar att i närvaro av tillräcklig mängd fukt och NaCl bildas nya strukturer i pappret med en relaxationstid av storleksordningen 10^{-3} s. Bestrukna pappers ϵ' påverkas av pigmenttillsatser och andra bestrykningstillsatser med polariserbara grupper och på grund av täthetsökningen. Papprets laddningspotential minskar och elektriska ledningsförmågan, laddningsavtagandet och dielektricitetskonstanten ökar med ökande temperatur. Beroendena är oftast exponentiella. Dessa termala energi- och temperaturberoende processer samt deras aktiveringsenergies ändras av joninnehållet.

Resultaten har utnyttjats vid tillverkning av papper avsedda för elektrofotografisk färgtryckning.

LIST OF PUBLICATIONS

- I. Sirviö, P., Backfolk, K.: **Effect of roughness of low-grammage coated papers on print quality in color electrophotography.**
(2008) Journal of Imaging Science and Technology, 52 (1), pp. 010505-010509.
- II. Sirviö, P., Backfolk, K., Maldzius, R., Sidaravicius, J., Montrimas, E.: **Dependence of paper surface and volume resistivity on electric field strength.**
(2008) Journal of Imaging Science and Technology, 52 (3), pp. 0305011-0305018.
- III. Sirviö, P., Sidaravicius, J., Lozovski, T., Kuskevicius, S., Backfolk, K.: **Dosed charging: Application to the investigation of papers.**
(2009) Journal of Electrostatics, 67 (5), pp. 730-736.
- IV. Sirviö, P., Sidaravicius, J., Maldzius, R., Backfolk, K., Montrimas, E.: **Effect of NaCl and moisture content on electrical and dielectric properties of paper.**
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- V. Maldzius, R., Sirviö, P., Sidaravicius, J., Lozovski, T., Backfolk, K., Rosenholm, J.B.: **Temperature-dependence of electrical and dielectric properties of papers for electrophotography.**
(2010) Journal of Applied Physics, 107 (11), art. no. 114904.
- VI. Backfolk, K., Sidaravicius, J., Sirviö, P., Maldzius, R., Lozovski, T., Rosenholm, J.B.: **Effect of base paper grammage and electrolyte content on electrical and dielectric properties of coated papers.**
(2010) Nordic Pulp and Paper Research Journal, 25 (3), pp. 319-327.

The author had main responsibility for the design of experiments, analysis of the results and of preparation of the manuscripts in publications I-IV. The author was responsible for the design of experiments and participated integrally in the analysis of the results and in the preparation of manuscripts in publications V and VI.

SUPPORTING PUBLICATIONS

- SI. Sirviö, P.: **The Effects of the Process Parameter Adjustments on Print Quality in a Web Fed Electrophotographic Color Printing Process.**
(2000) Proceedings of the IS&T's International Conference on Digital Printing Technologies, pp. 293-296.
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- SIII. Sirviö, P.: **About Paper Properties for Modern Dry Toner Presses.**
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(2007) Polymer Testing, 26 (8), pp. 1031-1040.
- SVII. Backfolk, K., Sirviö, P., Ihalainen, P., Peltonen, J.: **Thermal and topographical characterization of polyester- and styrene/acrylate-based composite powders by scanning probe microscopy.**
(2008) Thermochemica Acta, 470 (1-2), pp. 27-35.
- SVIII. Ihalainen, P., Backfolk, K., Sirviö, P., Peltonen, J.: **Thermal analysis and topographical characterization of films of styrene-butadiene blends.**
(2008) Journal of Applied Polymer Science, 109 (1), pp. 322-332.
- SIX. Kalade, J., Maldzius, R., Sidaravičius, J., Sirviö, P., Backfolk, K.: **Dependence of electrostatic field on properties of paper in a two-dimensional toner image transfer Nip.**
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- SX. Ihalainen, P., Backfolk, K., Sirviö, P., Peltonen, J.: **Topographical, chemical, thermal and electrostatic properties of latex films.**
(2010) Colloids and Surfaces A: Physicochemical and Engineering Aspects, 354 (1-3), pp. 320-330.
- SXI. Kalade, J., Maldzius, R., Sidaravicius, J., Sirvio, P., Backfolk, K.: **Dependence of electrostatic field strength on image transfer roller dimensions in a toner transfer region.**
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(2013) Journal of Electrostatics, 71 (1), pp. 35-40.
- SXIV. Myllys, M., Häkkänen H., Korppi-Tommola J., Backfolk K., Sirviö P., Timonen J.: **X-ray microtomography and laser ablation in the analysis of ink distribution in coated paper.**
(2015) Journal of Applied Physics 117, 144902.

1. INTRODUCTION

Most digital color printing is done today using dry toner electrophotographic processes. In dry toner electrophotographic printing, *Figure 1*, the image is developed with charged dry toner particles (the colorants) on a photoconductive drum or belt having a latent electrostatic image. The developed image is transferred from the photoconductor to the print substrate using an electric field. This transfer can be made directly from the photoconductor to the substrate, or by first transferring the toner particle image to an intermediate belt or drum and from there to the print substrate. In order to collect four colors on the print substrate, this process is repeated in the printing device, or all the colors are first collected on the intermediate belt or drum and transferred in one pass to the substrate.

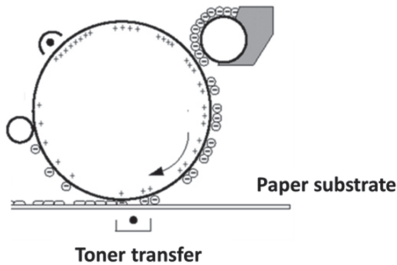


Figure 1. Electrophotographic toner transfer.

The electrical and dielectric properties of substrates have a significant effect on the print quality and runnability in dry toner electrophotographic printing, because the charged toner particles are transferred onto the substrate utilizing an electric field that is affected by these substrate properties. Static electricity affects the transportation of sheets in the printing presses. The electric field is also influenced by air gaps in the nip, in addition to the impact of substrate roughness, which affects the toner-substrate contact. The amount of toner transferred and the evenness of this transfer influences print quality. Especially the evenness of even-tone color areas is known to be challenging with substrates that are not optimal for the end use, *Figure 2*.

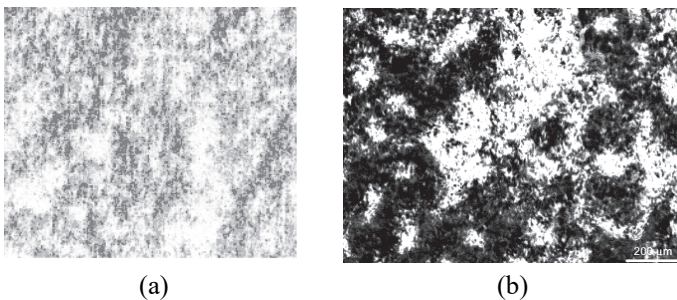


Figure 2. Typical unevenness defects in dry toner print density: a – 8 cm x 8 cm full coverage area with deletions due to a significantly too low electric field in toner transfer, b – small scale graininess of print due to paper roughness [Publication I].

2. OBJECTIVES

It is well known that adding ions or moisture to paper increases its dielectric constant and increases the dielectric loss as well as conductivity. Furthermore, the real part of the dielectric constant decreases with increasing frequency. However, it is not evident what the exact nature of these dependencies is and how they are influenced by basic substrate properties. Nor are the deviations from the expected dependencies well understood. Thus, for a more detailed analysis, controlled trial sets are required in addition to commercial samples, which have multiple unknown variables.

The objective of this work was to study in more detail how the dielectric permittivity and dielectric losses at different frequencies, electrical resistivity (conductivity), charge acceptance, and charge decay of paper substrates are dependent on the variables normally used in papermaking, including basis weight, material density, ion content, moisture content and surface treatment or coating. Another aim was to gain a better understanding of how temperature influences the dielectric and electrical properties and what are the mathematical forms of these dependencies. Also, the dependency of conductivity on the applied electric field was investigated, including fields relevant to the electrophotographic printing processes. In these processes, electric fields are essentially higher than the fields generally used for the measurement of the electrical properties of paper, and it is of interest to know the behavior under the conditions relevant to those existing in printing devices.

This information has been utilized in the development of substrates for electrophotographic color printing processes. The results and data may be useful in designing printer constructions, i.e., so that the electrical and dielectric properties of papers and, increasingly today, paper board for packaging are better understood and taken into account. It is also possible that printed electronics applications may in future be mass-produced on fiber-based substrates, in which development work their electrical and dielectric properties are of interest, similarly to the very interesting work published on cable papers several decades ago.

3. LITERATURE REVIEW

In electrophotography, the transfer of charged toner particles to the print substrate is made with an electric field. The strength of the electric field in the toner layer in the toner transfer nip, and thus the force applied to the toner particles is influenced by the electrical and dielectric properties of the print substrate. The electric field (and toner transfer) is also influenced by, for example, air gaps in the nip, in addition to the impact of substrate roughness that inhibits toner-substrate contact. The amount of toner transferred and the evenness of the transfer influences print quality.

3.1 Effect of substrate topography on toner transfer

Paper surface topography and the substrate texture influence both the electric field and the substrate-toner contact in the transfer nip and thus the print quality. In the literature, the inhibiting effects of air gaps in the electrophotographic toner transfer nip are identified as originating from the field collapse due to Paschen discharge, the effects of air gaps on the dielectric thickness of the system in the transfer

nip, or from the insufficient net force to transfer all toner particles to the printing substrate. This net force is also affected by the lack of adhesion forces between substrate and toner. [1-12]

Rimai and Quesnel investigated toner transfer to fabrics and to commercial papers and observed a failure in toner transfer in depressions of the substrate, ascribed to a lack of toner-paper adhesion [12]. Qualitative observations analogous to this were reported by Wright et al. [13]. Based on experimental results, Provatas et al. [14] concluded that optical print density mottle is dependent on filler (PCC) distribution in paper and on thickness (or material density) variations, but it was not reported whether there were any differences in roughness between the trial papers. Both quantitative and qualitative effects of paper roughness on edge raggedness [15], signal-to-noise ratio [16] and print density [17] in monochrome printing have been reported based on sets of uncoated commercial papers but since these results refer mostly to the raggedness of lines and text they cannot be directly related to variations in toner transfer in larger areas.

Al-Rubaiey et al. [18], investigated the effect of the coating layer on toner transfer, linking the amount of toner transferred to the resistivity of paper and to the transfer current. They also compared calendered and uncalendered paper samples and found that calendering enhanced toner transfer; this was explained by the increase in density of the paper but the possible effect of the smoother paper surface was not discussed.

The electric field in the materials in a toner transfer nip, including the air gap caused by substrate topography, has been modeled using capacitor models [6, 19-23], where the paper is often considered to be a uniform material. Considering in addition the actual locally varying paper properties, Cassidy et al. and Provatas et al. [14, 19, 20] concluded that toner print density variations are due to variations in thickness and in filler distribution, as such in line with the experimental results of Al-Rubaiey et al. [18]. However, the effect of paper surface smoothness was not considered. Kallunki et al. [21] presented a continuum model and investigated the influence of substrate roughness on the electric field in the toner transfer nip, and found that the calculated field force variations due to paper roughness correlated well with the transferred toner layer thickness in a cross-sectional analysis of a printed sample [21, 24]. Tong et al. [23] extended the analytical one-dimensional model by analogy with the models of Yang and Hartmann [25] and Kallunki et al. [21] to a 3D model.

3.2 Electrical resistivity and dielectric properties of paper and the effect of electric field on electrical resistivity

Suitable electrical resistivity and electric field are needed for an efficient transfer of charged toner particles [18]. The resistivity and polarization characteristics of paper influence the toner transfer efficiency and the resulting print quality, since these paper properties influence the electric field in the toner transfer nip. The ion and moisture contents of a paper substrate influence the electrical and dielectric properties of the paper and thus the toner transfer efficiency. In addition they affect the printing process reliability, especially in high-volume production, through the level of static electricity. Salts, mainly NaCl, and optimization of moisture content are often used to optimize the resistivity of paper for a certain end use. The optimal electrical and dielectric properties are also partly dependent on the speed of the printing process according to the time available for the electric field development in the transfer nip. It is also evident that, in addition to the printing speed, the optimal resistivity range depends on the printer construction, e.g. if the printing process involves several or only a single toner transfer to the paper.

The quantitative requirements for the electrical properties of paper in electrophotography are not strictly defined and the recommendations focus on resistivity values. For example, Lim [26] pointed out that the surface resistivity range accepted and usually adopted in the paper industry is 10^{10} - 10^{12} Ω . In agreement with Lyne [27], Kulmala et al. [16] considered the same surface resistivity range.

The DC resistivity measurement arrangements typically utilized to measure paper resistivity are in fact time-dependent [28], and if correlating the measurement arrangement to an actual printing process, the time scales differ significantly. This complicates any absolute comparison of the DC resistivity values that are traditionally measured in the paper industry and the situation in the dynamic toner transfer nip and thus the amount of toner transferred. Acknowledging these limitations, DC resistivity can be used to predict the behavior of substrates, e.g., in electrophotographic processes [17, 26, 27, 29].

Electrical conduction in paper is assumed to be ionic, charge carriers passing through the paper along fibers [28, 30, 31]. According to Murphy [30], the mechanism of conduction is that the charge carriers are cations, which move via the hydroxyl groups of polysaccharide chains, but these can move only if the hydroxyl group is occupied by a water molecule [30], so that the amount of water in the paper and the concentration of mobile charge carriers determine the conductivity of paper. However, Simula and Niskanen concluded that the model proposed by Murphy assumed an unrealistically high number of carboxyl groups able to supply paths for cations for conduction in an electric field [31]. In addition, any model assuming zero conductivity at zero moisture content does not agree with the experimental results [31, 32]. Simula and Niskanen proposed a model in which resistivity is linearly proportional to the amount of hydroxyl groups without a water molecule since, as in Murphy's model, these sites would stop the conduction path. The exponential dependence between DC resistivity and moisture content has been clearly demonstrated with various substrates, although different mathematical equations for this dependence have been proposed [30, 31].

Smith [33] found an exponential dependence between conductivity and charge carrier (ion) concentration in kraft pulp handsheets and suggested a mechanism where, below a certain water adsorption limit, conduction would be limited due to the absence of water molecules when the ion concentration is increased. O'Sullivan [34] concluded that the conductivity of cellulose (cellophane, which is hydrated cellulose) containing 1 % or more salt is determined primarily by the moisture content, but when the salt content is clearly below this, the salt (ion) content also becomes an important factor for the level of conductivity.

Soetanto et al. demonstrated that the salt content changes the conductivity because of hygroscopic effects also [35]. Due to the significance of the moisture content for the resistivity of paper, the magnitude of this mechanism needs to be considered when analyzing the relationship between salt addition and the resistivity of paper.

The impact of moisture and ion content on the dielectric properties of pulp and paper has been extensively investigated, with many of the data sets published in relation to the dielectric properties of capacitor papers dating from the 1940s and 50s [36-38]. Some of the more recent works refer to the polarization of paper from the aspect of electrophotography, using commercial coated and copy papers and viscose-kraft laboratory handsheets [31, 39]. With paper, the most important mechanisms of dielectric polarization are electronic, atomic, and molecular polarization [28], but space charges in the paper structure [40] and thus their polarization cannot be ruled out. The effect of changes in paper moisture content on the dielectric properties is strongly frequency-dependent, because the frequency determines which polarization processes are able to contribute to the polarization. At low frequencies, all these mechanisms have enough time, and can take place. In fine paper, made of wood-free pulp, the main components influencing polarization are carboxyl and hydroxyl groups on the fibers as well as water molecules and free ions. It is, however, not possible to simply add the dielectric constants;

as Simula et al. stated, for instance, water molecules contribute to the dielectric constant of paper less than their dielectric constant would suggest, thus indicating that not all water molecules are able to polarize freely [39].

In most cases reported in the literature, the experiments on the electrical and dielectric properties of various types of fiber-based materials, including paper, have been made at only one temperature, but there are also reports describing the temperature-dependence of the dielectric properties of papers [29, 31, 41] and cotton [32, 42, 43]. The experimental results for cotton cellulose can be used to provide understanding of paper properties and for comparing whether similar phenomena occur in paper substrates. Conductivity [31], and consequently paper charging characteristics, depend on the temperature. For the electrophotographic imaging process, it is worthwhile making dielectric measurements at relatively low frequencies, but testing the dielectric constant and loss at higher frequencies can provide additional information on the mechanisms and materials contributing to the polarization, pinpointing areas of interest, which is useful for higher frequency applications.

Paper is subjected to high electric fields during electrophotographic toner image transfer. The electric field strength in the transfer nip is in the range of $10^5 - 10^6$ V/cm, but paper conductivity is usually measured at lower electric fields in the paper industry and in reported results. Simula [29, 44], for example, measured paper DC resistivity at an electric field of approximately 10^4 V/cm (paper thickness ~ 100 μm and 100 V applied). The results on field resistivity dependence are partly contradictory; Lim [26] did not observe changes in resistivity when varying the applied voltage from 90 to 500 V. However, it has been reported and proven that paper resistivity depends on the electric field strength [28, 45]. This field dependence is attributed to the mechanism of ionic drift in the fiber network [28, 30, 33, 46, 47]. In the case of a hopping drift mechanism, the conductivity should indeed be field-dependent, as shown, for example, by Lin [47], who considered the decrease in energy or potential barrier between two dissociated sites due to the electric field. Hanneson et al. [40] investigated the electrical conductivity of capacitor tissue papers. At high voltages, the logarithm of the steady state current in their data was proportional to the square root of the applied voltage; the Schottky theory and the Poole-Frenkel effect were discussed as possible mechanisms.

3.3 Charging and charge decay of paper

The electrical and dielectric properties of paper impact the amount of charges on the paper surface, their drift towards the photoreceptor surface, the rate of this leakage over time, and thus the strength of the electric field in an electrophotographic toner transfer nip and its time dependence.

The charging and the charge decay of paper are usually considered to be dependent on the surface and/or volume resistance, although no unambiguous correlations with charging ability or charge decay rate have been established [16, 25, 28, 39, 48, 49]. It is of interest to understand the charging dynamics of paper more deeply; especially since it has been noted that electrophotographic transfer efficiency correlates better with charge decay rate than with resistivity [39, 50]. The charge decay time is strongly linked to the moisture content of the paper [51]. Jonassen et al. [49] investigated the DC bulk and surface resistances of paper along with charge decay and found a correlation between surface resistance and charge decay half-time, but this dependence was influenced by the number of paper sheets stacked in the measurement – this indicated that the bulk properties of the material also influence the surface resistance. They also concluded that ohmic electrical conductance is not the only mechanism responsible for charge dissipation since the charge decay was not strictly exponential, which is an important and interesting observation. Correlations between paper charging properties and electrical and dielectric properties have also been investigated and considered, e.g. by

Kulmala et al. [16], Gullichsen et al. [28], and Simula [44]; some degree of correlation exists but these correlations are not unambiguously strong. Chen et al. [50] concluded that toner transfer efficiency may be better correlated with the time constant of the charge relaxation of paper (the product of dielectric constant and the resistivity) than with resistivity alone. This result indicates that both conductivity of paper and its polarization, and their dependence on the moisture and ion contents of the paper, are important for the toner transfer.

Tse and Klein [52] discussed the charge dissipation of paper from a methodological perspective and pointed out that the data normally presented on paper charging and discharging properties are insufficient for the purposes of deeper analysis, as typically only the maximum voltage and charge decay half time are reported. The determination of these two data points are useful but can also lead to inaccurate conclusions in specific end uses, e.g. in the field of electrostatic transfer, since the charging and discharging of paper materials is a complex combination of charge generation, transport, and trapping. The proposal of the authors [52] that the dynamics of the entire discharging process should be monitored is an important one, because during paper charging and discharging, polarization effects and the redistribution of the electric field can take place in the paper bulk, and these phenomena are dependent on material, time, temperature, and humidity. Monitoring the charging process also provides additional information on the dynamics of the charge acceptance of paper substrates.

3.4 Effect of coating

Coated papers and boards are required in graphical printing market segments and in printing of packaging. Therefore, it is worth extending the analysis of electrical and dielectric properties to these substrates as well. The coating layer and base paper also provide interesting possibilities for separately adjusting the surface structural, electrical, and dielectric properties of the substrate and its layers, as well as for tailoring them for printing processes and for specialty end uses.

As far as coated papers are concerned, only few studies on electrical and dielectric properties have been reported. In any case, in relation to the base paper of a coated paper, the same reports describing the properties of uncoated papers are relevant, including the electrical conductivity of cellulose [30], wood [46, 47], different paper grades [28, 40, 53], and the relation between electrical conductivity and interfiber bonding [33].

Simula et al. [29, 39, 44] investigated the electrical and dielectric properties of substrates including coated papers. In their analysis of surface vs. volume resistivity, a linear correlation was observed, but coated papers deviated slightly from this linear relationship [44]. This deviation indicated that the electrical conductivity of coated papers could not be regarded as being limited to the surface layer alone. The experimental resistivity values reported showed significant variations between commercial paper grades, evidently originating from differences in grammage, density, filler content, and other parameters of the commercial samples.

Simula [29] also showed that the dielectric constant of coated papers is higher than that of uncoated papers, both at low (100 Hz) and high (1 MHz) frequencies. At 100 Hz, the ϵ' increased linearly with increasing relative humidity (RH) up to 40 %, after which a more rapid increase occurred. The curve was steeper for coated paper, but the critical RH value seemed to be the same for both coated and uncoated papers. The sudden change in dielectric constant at 40 % RH is attributed to a polarization mechanism which begins to occur at this humidity; at high frequency, the increase in the dielectric constant was linear over the range from 20 to 50 % RH for both coated and uncoated papers. The

dielectric constant of coated paper was, however, twice the value of uncoated paper, and was attributed to the high dielectric constant of CaCO_3 , which is normally the main coating pigment in these types of papers.

Morsy [54] investigated the dielectric properties of papers coated with clay-based mixtures by adding different thickeners and showed that coating of a base paper with a clay-based mixture increased both the dielectric constant ϵ' and the dielectric loss ϵ'' , whereas thickeners generally decreased both ϵ' and ϵ'' . For example, the dielectric constant ϵ' at 100 Hz frequency of the paper with casein added to the coating was the same as that of the base paper. The dielectric loss ϵ'' at this frequency became even lower than the dielectric loss of the base paper with the investigated thickeners. The increase in dielectric constant of the clay-coated paper was considered to be due to the higher density of the coated paper. The contribution of the dielectric constant of the clay coating as such also probably had an impact.

Al-Rubaiey et al. [18] investigated toner transfer efficiency with coated papers and found that the formulation of the coating controls the electrical behavior of the coated paper. The volume and surface resistivities of coated papers were directly related and the resistivities of both uncalendered and calendered papers were almost the same in that sample set, although the transfer efficiency changed noticeably. In an investigation of the influence of RH on the charging of coated papers, at 20 % RH the surface potential was in the range of 600 V compared to only 100 V at 50 % RH [18]. When the surface potential value was compared with the surface resistivity of coated papers, there was no clear correlation, although a tendency could be seen.

4. EXPERIMENTAL

4.1 Materials

In order to establish dependencies between paper properties and the electrical and dielectric properties of the substrates, sample sets were made on pilot and lab scale in addition to commercial samples. The aim of the pilot substrates was to allow the exclusion of uncontrolled variables in the interpretation of the results.

4.1.1 Paper samples and coating formulations for determining the effect of roughness on toner transfer

Internally sized woodfree 80 and 90 g/m^2 commercial base papers (manufacturer Stora Enso Oyj) were pilot-coated, using blade and filmpress coating techniques, at a speed of 800 m/min. Different coating formulations and rheologies were utilized to give differences in the coating layer structure. Three coating formulations were used: dispersions 1 and 3 consisted of 75 parts of engineered ground calcium carbonate with a narrow particle size distribution and 25 parts of fine Brazilian kaolin with 11 parts of styrene/butadiene and 11 parts of styrene/acrylate latex, respectively. Dispersions 1 and 3 differed in latex type. Coating dispersion 2 consisted of 100 parts of engineered ground calcium carbonate with a narrow particle size distribution, and 11 parts of styrene/butadiene latex. In all the coating recipes, the following additives were used: 0.5 parts of sodium carboxymethyl cellulose, 0.5

parts of fully hydrolyzed polyvinyl alcohol, 0.5 parts of calcium stearate, 0.3 parts of synthetic alkali-swelling emulsion (ASE), and 1.3 parts of a sulfonated stilbene derivative. Three different coat weights were targeted: 6, 9, and 12 g/m² per side, and different roughness levels and different surface structures were obtained by varying the nip pressure in the soft calender, which was operated at 150 °C, 0 kN/m, 25 kN/m and 150 kN/m nip pressure. The Print-Surf roughness (measured in accordance with ISO 8791-4 with 1.0 MPa clamping pressure) of these two-sided coated papers ranged from 1.4 to 6.2 µm, grammage from 91 to 117 g/m², and thickness from 93 to 133 µm. Conditioning the papers in 24 - 71 % relative humidity for the printing trials resulted in an absolute moisture range of 2.6 - 6.2 %. Further details of these samples can be found in Publication I.

4.1.2 Paper samples for resistivity, charging, charge decay, and dielectric measurements

The uncoated papers investigated in evaluating the dependence of paper resistivity on the electric field were made on a pilot paper machine targeting filler contents of 0 %, 15 %, and 30 % (precipitated calcium carbonate) and grammages of 90, 160, and 230 g/m². The paper samples were internally sized with alkyl ketene dimer (AKD), and cationic starch was used with AKD (1.5 kg/tonne) and as a wet end additive (8 kg/tonne). A two-component retention system was used with bentonite and polyacryl amide (PAM) additives, of 1.7 kg/t and 0.2 kg/t, respectively. The pulp mixture contained 80 % hardwood and 20 % softwood. The paper was not surface sized and no colorants or fluorescent whitening agents (FWA) were used. The sets of samples were then calendered on a laboratory soft-nip calender (soft and hard rolls, speed 25.5 cm/s) at a temperature of 100 °C and a pressure of 25 kN/m. The sheets were run through the nip twice, so that both sides of the sheet were calendered in the same way. Samples were also produced by calendering both sides twice at a nip pressure of 50 kN/m and a calender temperature of 100 °C. Additional details of the samples can be found in Publication II.

In order to determine the charging and charge decay properties of the paper, commercial uncoated (manufactured by Stora Enso Oyj, grammage 90 - 300 g/m², density 0.9 - 1.0 g/cm³) and coated paper (manufactured by Stora Enso Oyj, 130 - 200 g/m², 1.2 - 1.3 g/cm³), and paper made on the pilot-scale paper machine described above with 0.7 g/cm³ density and 90, 160, and 230 g/m² basis weights were used. Further details of these commercial and pilot paper samples can be found in Publication III.

For the analysis of the effects of NaCl and moisture content on the electrical and dielectric properties of the paper, NaCl salt was dosed on the pilot-machine base paper described above by applying the salt at the size press as an aqueous solution on both sides of the web. The target dosage levels were 0, 0.75, 1.5, 3, and 6 kg/t with the 160 g/m² paper, and the same amounts per unit area on the 90 and 230 g/m² base paper; the actual NaCl content of the trial paper was then determined by ion chromatography of the water-soluble chloride, where the sample pre-treatment procedure and determination of water-soluble chloride complied with ISO 9197. The NaCl content [kg/t] of the 160 g/m² paper was in good agreement with the target dosage. The NaCl content of the 90 and 230 g/m² paper was 0 - 10.6 kg/t and 0 - 4.3 kg/t, respectively. The 90 g/m² and 160 g/m² base paper took up similar amounts of NaCl per square meter, but the uptake by the 230 g/m² was greater, linked to the higher pore volume with the higher grammage. Samples were laboratory-calendered on both sides twice at a nip pressure of 50 kN/m and a temperature of 100 °C, resulting in Print-Surf roughness values of between 3.2 µm to 5.0 µm and density values of between 0.73 g/cm³ and 0.84 g/cm³. Paper produced that was similar to the above yet differing in thickness or density was used in order to investigate the temperature dependency of the electrical and dielectric properties. Further details of these uncoated pilot paper machine samples are available in Publications IV and V.

Base paper made similarly was used in the investigation of the electrical and dielectric properties of coated paper; so that the NaCl surface-treated base paper reels were soft calendered (75 kN/m, 100 °C) for a more controlled coating result, before being blade-coated on a pilot coater. The pigment coating dispersion was applied with a blade coating unit to achieve three different coat weights (8, 12 and 20 g/m² per side), the highest of which was double-coated: 8 g/m² (first pass) and 12 g/m² (second pass). The coating speed was 400 m/min. The following coating recipe was used for all samples: 75 pph calcium carbonate, 25 pph kaolin, 11 pph styrene/butadiene latex, and 0.8 pph sodium carboxymethylcellulose. A reference coating sample was prepared using the same coating dispersion and applied manually onto aluminum foil, in order to determine the electrical and dielectric properties of the coating. Further details of these pilot coater samples with pilot-made base paper can be found in Publication VI.

4.2 Methods

4.2.1 Measurement of paper topography, roughness, print quality, and toner transfer

To evaluate the print result and toner transfer, samples were printed on a commercial color printer (Xerox DocuColor 2060) using conventional two-component polyester-based toners with a printing speed of 60 pages per minute, at 23 °C and a 50 % RH laboratory environment, the papers having first been acclimatized to said environment. The device transfers the toner image onto an intermediate belt and transfers the image to the paper in a single step in an electrophotographic transfer nip. A sub-set of paper with a moisture content of between 24 and 71 % relative humidity (2.6 - 6.2 % abs.) was utilized in order to determine the effect of surface roughness on toner transfer with different paper resistivities. This same sub-set of samples was further printed in an electrophotographic lab printer (Torrey Pines Research) with different transfer fields (corona voltages between 3700 and 4200 V) to see whether there were air breakdown discharges at the locations of surface cavities. These prints were made in a 40 % RH, 23 °C environment at a printing speed of 10 cm/s using a conventionally prepared two-component cyan toner.

The print mottle was evaluated numerically with a scanner-based mottle measurement system, (TAPIO PapEye, manufactured by TAPIO Technologies). The sample is scanned at 300 dpi resolution in the grayscale mode and wavelet analysis is applied to calculate values for different bandwidths (0.17 mm, 0.34 mm, 0.67mm, 1.34 mm, 2.54 mm, 5.1 mm, 10.2 mm) of 9 cm² full-tone images, with four parallel measurements. From these values, a single mottling value is calculated, which is a weighted average of several bandwidths; however, the data of separate bandwidths was also used in the analysis. The print mottle was measured from full-tone single toner layer areas (100 % of cyan, magenta, yellow, or black) and from full-tone areas of two toner layers (200 %: red, green, or blue). Print mottle defects were studied also with a light microscope.

Paper surface roughness was characterized using a Print-Surf instrument (Lorentzen & Wettre) with 1.0 MPa clamping pressure in compliance with ISO 8791-4. Surface profiles of the selected samples were obtained using a Rodenstock RM-600 white-light laser profilometer operated at 5 mm lines using 2.5 µm steps and with a depth resolution of less than 1 µm. In this analysis, volume and surface resistivities were measured with an HP 4339B high resistance meter with an HP 16008B resistivity cell, in accordance with ASTM D257-78, using a test voltage of 100 V and a charge time of 30 s, with ten replicate measurements. Further details of the measuring methods and experiments can be

found in Publication I. In all the other resistivity (conductivity) analyses reported, apart from this specific trial, the resistivity (conductivity) was measured as described in 4.2.2.

4.2.2 Measurement of paper resistivity

The DC volume resistivity of paper was evaluated in accordance with ASTM D257 [55]. The DC surface resistivity was evaluated using raker-type electrodes [Publication II]. The small distance between the electrodes allows the measurement of surface resistivity at higher field strengths before electric breakdown occurs. The volume resistivity was calculated from the resistance values using paper thickness values obtained under a pressure equal to that used in the dielectric measurements. The thickness values were obtained under a pressure of 1.5 kPa in all the evaluations apart from the evaluation of the effects of the electric field strength on paper resistivity, where the thickness values were obtained at an electric field strength of 500 V/cm (pressure of 25 kPa). The mechanical pressure was kept constant in all measurements.

All the measurements were performed at 23 ± 1 °C and 50 ± 2 % relative humidity (RH), except for the investigation of the effects of temperature and moisture content, in which the electrical and dielectric properties were measured at five different temperatures and RHs: at 50% in 16 °C, 23 °C, 33 °C, 43 °C, and 53 °C, and at 23 °C in 25 %, 30 %, 40 %, 50 %, and 60 % RH. The impact of hysteresis was minimized by conditioning the samples in a similar way. All these measurements were made with the chosen temperature maintained with a precision of ± 1 °C. The papers were conditioned for at least 8 h prior to the tests.

The moisture isotherms of the paper samples containing different amounts of NaCl were obtained to evaluate the relative importance of hygroscopic effects and other mechanisms on the electrical properties of paper when the salt content is varied. For this measurement, the paper samples were first conditioned to 50 % RH and the moisture content at 23 °C was then determined at different relative humidity levels, starting from 20 % RH and ending at 80 % RH, in steps of 10 % RH. The conditioning time at each RH level was 24 hours, after which the A4-size sheets were weighed on a balance with a resolution of 0.001g (Mettler PC440). The dry weight of each sheet was determined according to SCAN-P4. The reported moisture content values are average values of three samples.

4.2.3 Measurement of charge acceptance and charge decay of paper

Paper charging-discharging processes were investigated by repeatedly depositing relatively small charges onto the paper surface using corona charging [Publication III], *Figure 3*. The electrical current flowing through the paper sample, the displacement current which determines the charge deposited on the paper, and the paper surface potential during charging were all monitored in every charging cycle. After charging stopped, the potential decay was recorded. A calibration measurement was made for the electrometer calibration in each cycle. A charge dose, ΔQ , was deposited on the paper during each rotation, and the potential of the paper was measured with an electrometer. The charging frequency was changed in the range of 5 to 10 Hz and the charge dose ΔQ was selected between 10^{-10} C and 10^{-4} C, depending on the sample and corona voltage. The measurement accuracy was ± 0.55 V in the range from 5 V to 100 V and ± 7 V in the range from 100 V to 3500 V. The equilibrium potential is the maximum paper potential under given experimental conditions and is

achieved when the charge leakage and charge deposition rates become equal. This value is the charging potential or charge acceptance of the paper.

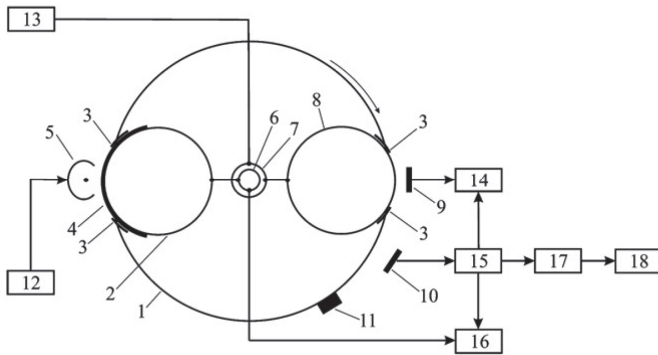


Figure 3. Device for dosed charging of papers. 1 – rotating cylinder, 2 – cylindrical paper sample holder, 3 – dielectric insulating film, 4 – paper sample, 5 – corona charger, 6 – charging current collector, 7 – calibration voltage collector, 8 - calibration cylinder, 9 – non-vibrating electrometer probe, 10 - synchronizer sensor, 11 - synchronizer transducer, 12 - high DC voltage source, 13 - calibration voltage source, 14 – electrometer, 15 - synchronizing device, 16 – charge integrator, 17 – A-D converter, 18 – computer. [Publication III]

4.2.4 Measurement of dielectric properties of paper

Dielectric properties were evaluated by measuring the electrical capacitance C and the dielectric loss factor $\text{tg}\delta$. The capacitance C was first measured with the paper inserted into the holder, where the electrodes were pressed against the paper using a spring (pressure 1.5 kPa). The distance between the electrodes was fixed. The paper was then withdrawn and the capacitance C_0 measured for the same distance between the electrodes. The relative dielectric constant is given by $\epsilon = C/C_0$. This procedure also makes it possible to determine the paper thickness $h = \epsilon_0 S/C_0$, where ϵ_0 is the permittivity of air (~vacuum) and S is the area of the electrodes. This thickness was used in the calculation of the electric field strength. The measurement of C and $\text{tg}\delta$ was performed at frequencies from 100 Hz to 1 MHz. Further details of the measurement method are available in Publication IV.

As with the determination of resistivity, the dielectric properties were measured at five different relative humidities (RH): 25 %, 30 %, 40 %, 50 %, and 60 % and at five different temperatures: 16 °C, 23 °C, 33 °C, 43 °C, and 53 °C. The treatment of the paper samples was similar to that for the resistivity measurements described in Chapter 4.2.2.

5. RESULTS AND DISCUSSION

In this chapter, the main results from Publications I-VI are summarized in four main areas. The first area is the effects of surface topography and moisture content on electrophotographic toner transfer, including the effects of electric field or toner transfer voltages [Publication I]. This links the results of the other publications to the electrophotographic print quality, since the electric field (as can also be seen in supporting publication SI), and the moisture content, which directly influences the resistivity and the dielectric properties of paper and consequently also its charging and charge decay properties, had a clear impact on the print result. The second area concentrates more deeply on the resistivity of paper. The dependency of the resistivity (conductivity) of paper on the electric field, on the NaCl content and moisture content, on temperature, coat weight, and the base paper conductivity of coated papers is described in Publications II, IV, V, and VI. The third area is the dielectric properties of paper and specifically the effects of moisture and ion (NaCl) contents and temperature on the dielectric constant and loss [Publications IV and V]. The fourth area focuses on the charge acceptance and charge decay of the paper [Publications III and V].

5.1 Effects of substrate topography and moisture content on electrophotographic toner transfer

The effects of coating formulation, structure, and finishing conditions on the print mottle of the prints produced with a commercial printer under standard conditions (50 % RH, 23 °C) are shown in *Figure 4*. The surface roughness had a greater effect on the print mottle, particularly in the 100 % areas, than did the coating formulation. The small changes in mineral composition did not lead to any substantial differences in print graininess when the paper was smooth. An increase in coat weight reduced the print mottle, since it clearly changed the topography (roughness) of the substrate. There were also obvious differences at low coat weights, which can be ascribed to the effects of coating layer uniformity. A small linear increase in the print mottle was found up to a Print-Surf roughness level of 3-3.5 μm , after which the increase was more pronounced with substantially more scatter. Print mottle at low roughness levels, below a Print-Surf roughness of approximately 3 μm , was thus at the same level regardless of coating method, coat weight, or coating formulation. At higher roughness levels, the characteristics of the coated paper began to have a greater influence on the print quality, where the coating method, formulation, and particularly coating uniformity played a role.

Paschen discharge was not the cause of non-transfer: in analysis with a profilometer the sample with a PPS value of 5.0 had differences between the lowest (cavity bottoms) and the highest (peak tops) regions of less than 3 μm and such a gap was further decreased in the toner transfer nip due to electrostatic pressure. Typical fields used in the toner transfer nip mean that the gap needs to be less than 8 - 10 μm to avoid air breakdown [56]; this was also confirmed by microscopy analysis of samples printed with different transfer fields on a test-bench, which gave no indication of Paschen discharging on solid areas.

In the 200 % areas, it was evident, considering the increase in scatter, that roughness alone cannot fully explain print mottle. In the case of a cavity in rough paper, the 100 % toner layer did not transfer at all and in the case of 200 %, typically the toner layer nearest to the paper had transferred, as could be seen in microscopy analysis [Publication I]. When the toner layer thickness was increased to 200 %, the print mottle was more dependent on coating layer uniformity, and thus also on the uniformities of the dielectric thickness and conductivity in addition to the surface topography.

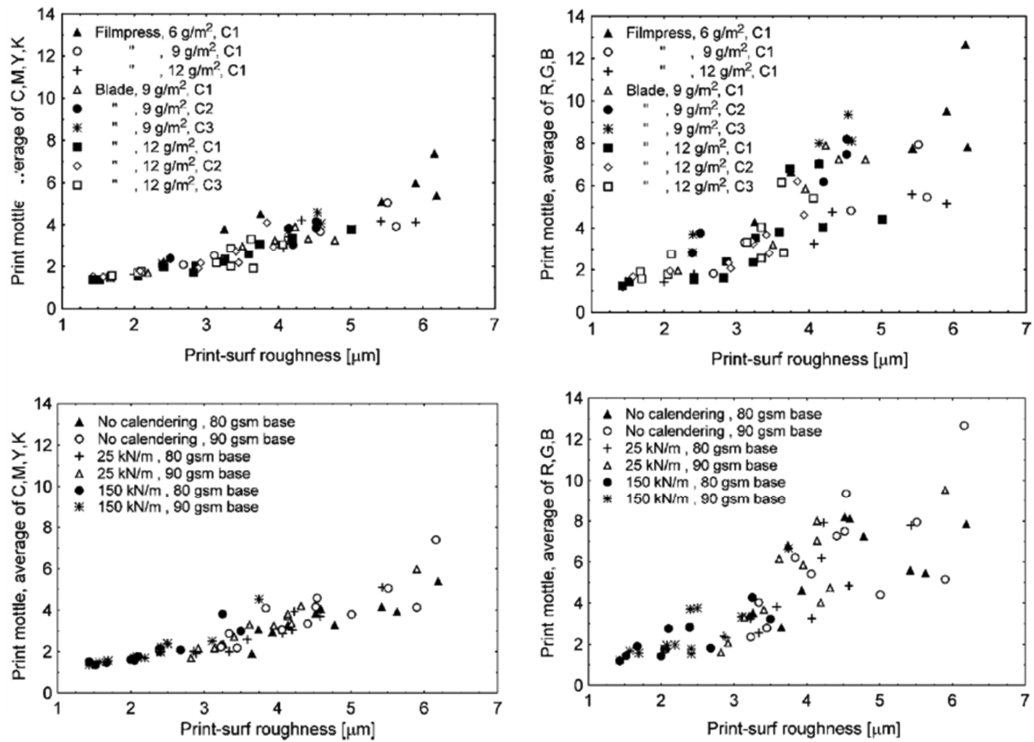


Figure 4. Average print mottle of 100 % toner layer areas (left) and 200 % toner layer areas (right) versus paper roughness. Filmpress- and blade-coated pilot papers with different coat weights [g/m²] and different coating dispersions C1, C2, and C3 (top), and effect of base paper grammage and calendering (bottom). Printed at 50% RH, 23 °C. [Publication I]

The relationships between toner layer thickness, paper roughness, and moisture content of paper influencing its resistivity and dielectric thickness were examined with paper samples conditioned to different absolute moisture levels, *Figure 5*. With 100 % coverage, the roughness of the paper dominated the print result over the moisture content compared to the situation with a 200 % toner layer. With an increasing toner layer thickness and low moisture content (3.5 % or lower), the impact of roughness was further amplified. This was contributed by the increase of local differences in the electrical properties of the paper, since the differences in the conductivity and in the dielectric thickness of the coating vs. the base paper [Publication VI] increase with the decreasing moisture content of the paper. This effect could be seen especially with small bandwidths corresponding to the size of defects observed in microscopy evaluations [Publication I]. Motivating a further analysis of paper resistivity (conductivity) and its dielectric properties, it is also evident from *Figure 5* that the electrical and dielectric properties connected to the moisture content of the paper have a significant effect on the print result in electrophotography.

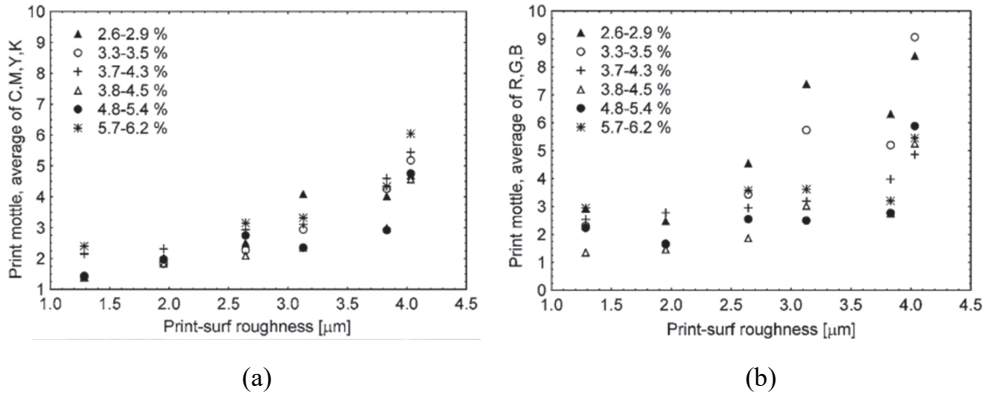


Figure 5. Effect of paper roughness and moisture content on average print mottle for 100 % toner layer areas (a) and 200 % toner layer areas (b). [Publication I]

Since it was concluded that discharge was not the mechanism behind the unevenness of printed areas with this sample set (PPS10 roughness 1.4 - 6.2 μm), possible causes of print mottle are that the electrical field in the toner layer is insufficient for complete toner transfer due to air gaps present in the nip and/or lack of toner-paper contact.

The profilometer data revealed several locations at which the distances between peaks correspond to typical defects seen in the microscopy analysis and in print evenness measurement data, where most of the print mottle defects caused by an increase in roughness were detected in bandwidths of 0.17 - 0.67 mm [Publication I]. The correlation between print mottle and roughness was stronger for 100 % toner coverage areas than for 200 % areas. Furthermore, since, according to the microphotographs, in the 100 % areas the toner had not transferred at all to the deepest cavities in the paper surface, whereas in the 200 % areas only the toner particles nearest to the paper surface had transferred to the regions of depression in the paper surface, it can be concluded that the paper-toner contact and adhesion are critical for transfer, as reported by Rimai and Quesnel [12].

The air gaps induced by the roughness of the substrate also reduce the electrical field [21]. The effects of air gaps in toner transfer may also be considered by using a continuum model and Maxwell equations [6, 19-23], even though this approach has limitations in the case of paper in a dynamic transfer nip. Paper has a time-dependent charge development and contains charge carriers with a certain mobility. Paper is a complex non-uniform structure, the complexity of which cannot be taken fully into account in a relatively simple continuum model. Conduction in paper is assumed to be ionic and the mobility of these ions is dependent on the moisture content of the paper [28, 30, 33, 46, 47], so there is normally a density of free charges in paper. Also, since the dielectric properties of paper depend on the frequency and moisture [39], the time available may be insufficient for a complete dielectric orientation during toner transfer. It can thus be assumed that a static one-dimensional model approximation could best be used to describe dry paper or paper with a minimum concentration of ions (salts) in a slow transfer process. Since continuum models are often utilized to describe the fields in the toner transfer nip, it is of interest to test this approach for evaluating the roughness effect, as the samples include dry paper samples with reduced ion mobility. For simplicity, one-side coated paper is considered.

The following one-dimensional continuum model is applied: four stacked uniform layers: base paper (thickness d_b , dielectric constant ϵ_b), a coating layer (d_c , ϵ_c), air (d_a , ϵ_a), and a toner layer (d_t , ϵ_t).

The charge density is zero in all layers other than the toner layer with a charge density ρ_t . The Poisson equation for the toner layer can be written as

$$\frac{\partial^2 \phi_t(z)}{\partial z^2} = -\frac{\rho_t}{\varepsilon_t} \quad (1)$$

The boundary conditions for potential ϕ are V_0 at $z = 0$ (the non-printed paper surface) and $\phi = 0$ at the top of the toner layer ($z = d_b + d_c + d_a + d_t$). Then, by analogy with Kallunki et al. [21] but including the coating layer, the electric field component in the toner layer perpendicular to the paper surface (i.e., in the z direction) is

$$E_t = \frac{1}{\varepsilon_t \left(\frac{d_b}{\varepsilon_b} + \frac{d_c}{\varepsilon_c} + \frac{d_a}{\varepsilon_a} + \frac{d_t}{\varepsilon_t} \right)} \left(V_0 - \frac{\rho_t d_t^2}{2\varepsilon_t} \right) + \frac{\rho_t}{\varepsilon_t} (z - d_b - d_c - d_a) \quad (2)$$

The field can be approximated by inserting values [57-59] of transfer voltage and negatively charged toner in Equation 2, $V_0 = 3000$ V, $\rho_t = -10$ C/m³, $\varepsilon_t = 2$, and using a toner layer thickness $d_t = 10$ μ m. The relative permittivity of the base paper (ε_b) was, in this case, approximately 3 at a frequency of 100 Hz. The data of Simula et al. [39] indicates that the relative permittivity of the pigment coating layer is significantly higher than that of the base paper, as also confirmed by later results [Publication VI]. In this numerical evaluation, an estimate of $\varepsilon_c = 5$ was used.

At a location on a 90 g/m² base paper (thickness 108 μ m), which has a 9 μ m deep cavity covered with a 3 μ m coating (6 μ m air in the transfer nip due to the paper), the calculated field in the middle of a 10 μ m toner layer is 29 V/ μ m. Respectively, for a 80 g/m² base paper, which has a thickness of 96 μ m, a 12 μ m coating layer and no air gap, the corresponding value is 36 V/ μ m. Such differences can be used in part to describe the influence of different paper on the electric field in a toner transfer nip even with the limitations described and also noting the importance of adhesion forces in toner transfer.

A summary of the main results of this section:

- Toner transfer is affected by air gaps but is not governed by the Paschen discharging effects with the paper normally used in electrophotographic printing.
- The moisture content of the paper and the electric field strength in the transfer influence the print quality in electrophotography.

5.2 Electrical resistivity/conductivity of paper

5.2.1 Dependency on electric field

Surface and volume electrical resistivities are strongly dependent on the electric field, *Figure 6*. Both decrease with an increasing field even if the nature and magnitude of the decrease are different. The character of the resistivity–field dependency is affected by the paper properties such as density and filler content. The surface resistivity of the trial points with 28 % filler (PCC) was higher than with the corresponding trial points for papers without filler, which is in line with the results of Soetanto et al. [35]. This effect can be influenced by the equilibrium moisture content of paper, which decreases with increasing filler content. However, the volume resistivity results did not show any clear trend in dependency between filler content and resistivity.

Simula et al. [29] measured the electrical properties of commercial papers at fields of approximately 10^4 V/cm and the results were comparable with those in *Figure 6*. Lim [26] and Sapielha et al. [53] presented data for commercial papers concerning the relationship between surface and volume resistivity (or conductivity). The contradictions between the results reported in the literature are influenced by material differences in the commercial samples or the different electric fields applied, this being a significant parameter (*Figure 6*) for comparability of any resistivity measurements.

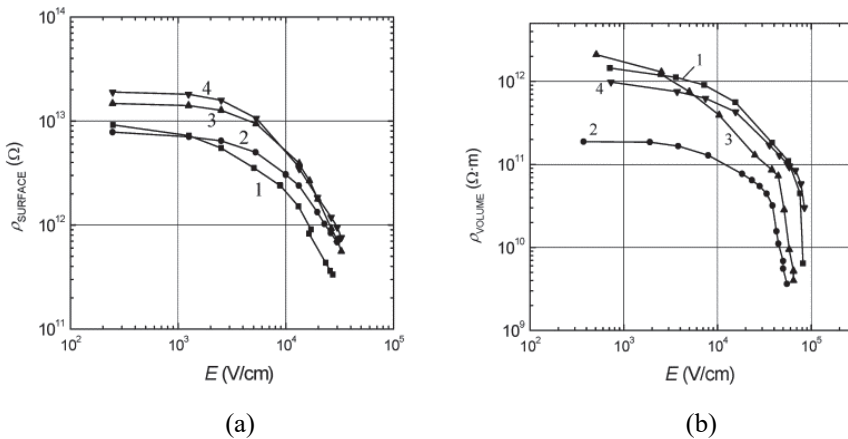


Figure 6. Surface (a) and volume (b) resistivity versus electric field strength for papers 1: without filler; 2 and 3: with 15 % PCC filler; 4 - with 28 % PCC filler. 1, 2 and 4: $91 - 95 \text{ g/m}^2$, density $0,65 - 0,67 \text{ g/cm}^3$, 3: 157 g/m^2 , $0,77 \text{ g/cm}^3$. [Publication II]

Surface resistivity decreased less than the volume resistivity with an increasing electric field, and as expected, surface resistivity was almost independent of paper thickness, *Figure 7*. However, volume resistivity decreased with high thickness even when the electric field decreased with thickness. The causes of such a decrease in volume resistivity may be influenced by the formation of space charges in thick paper. It can be assumed that, since paper is not a homogenous material and may consist of several layers (e.g. top-side coating and/or top-side surface sizing, paper bulk, reverse-side coating and/or reverse-side surface sizing), charge carriers can be trapped in between the different material layers or interfaces inside the paper if these layers cause energy barriers for conduction. These barriers and the resulting accumulation of charges and local variations in the electric field inside the paper due to its layers can then impact the electrical and dielectric properties of the paper.

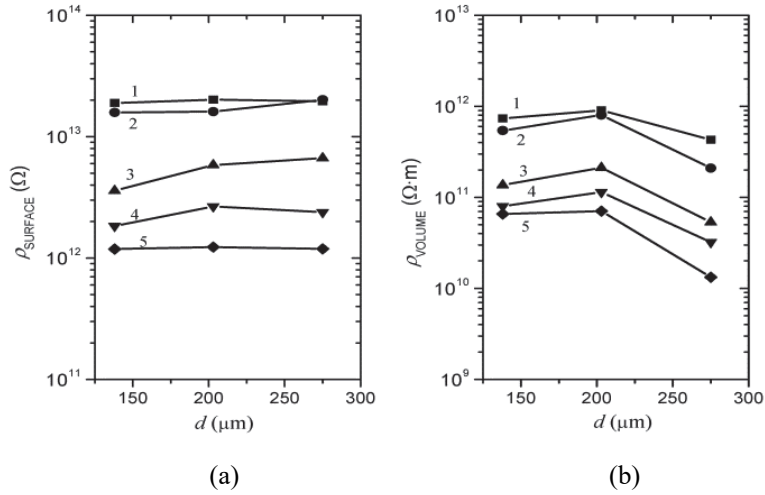


Figure 7. Surface (a) and volume (b) resistivity versus paper thickness at different applied voltages: 1 – 10 V, 2 – 100 V, 3 – 500 V, 4 – 750 V, 5 – 1000 V. [Publication II]

The surface resistivity is almost independent of the paper density, *Figure 8*. The volume resistivity at low electric fields grew with increasing paper density, which contradicts Lim’s result [26] with commercial papers, which can be ascribed to differences in moisture and ion contents; yet the volume resistivity was independent of the density at high electric fields (above 10^4 V/cm). Regarding calendering, the influence of electrode contact needs to be considered, since improved contact decreases resistivity. The results show that surface resistivity does not depend on roughness in the smoothness range investigated, and volume resistivity at low electric fields increases with density. The latter effect can be partially influenced by the lower equilibrium moisture content of papers with higher density. It can be concluded that contact effects do not have any significant role that would cause differences to the resistivity readings in this smoothness range, although the results do not allow full exclusion of the influence of electrode contacts.

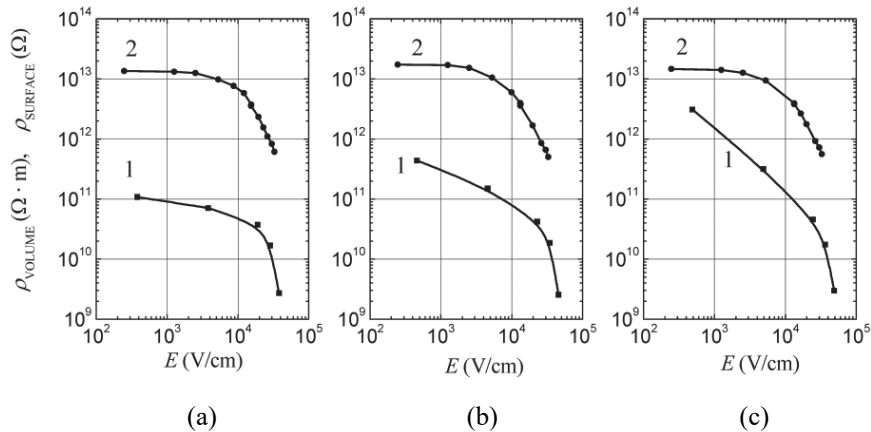


Figure 8. Volume (1) and surface (2) resistivity dependence on calendering level of paper. Paper density, g/cm³: a – 0.60, b – 0.72, c – 0.77, Print-Surf roughness, μm: a – 7.2, b – 5.0, c – 4.2. [Publication II]

The fact that paper resistivity is dependent on electric field strength to a phenomenal level is mentioned in several publications [28, 45, 60]. This dependence is attributed mostly to the mechanism of the drift of ions, i.e., the hopping of ions from one site to another. The sites are separated in space and consequently by an energetic barrier. Such a mechanism is well known for the drift of holes or electrons in organic semiconductors [61]. In general, the hopping drift mobility of charge carriers is dependent on the electric field. This dependence is often attributed to the reduction in the barrier between the sites (the Poole-Frenkel effect), and the dependence of the charge carrier mobility on the electric field is expressed as

$$\mu = \mu_0 \exp(\alpha\sqrt{E}) \quad (3)$$

where μ_0 is the charge carrier mobility at zero electric field and α is the Poole-Frenkel parameter.

As a phenomenon, the electrical conductivity σ of any material may be expressed in terms of the density of charge carriers n , the electronic charge of the carrier e , and the mobility μ of the carrier, as $\sigma = en\mu$. Conductivity in paper is an ionic phenomenon and we can assume that for a constant paper composition, constant temperature, and humidity, and assuming that the dissociation of ionogenic species in paper does not depend on the electric field, the density of charge carriers (ions) is constant. The conductivity will then be dependent only on the mobility of the ions, and the dependence of $\log \mu$ on $E^{0.5}$ will be linear.

This dependence for the surface conductivity of pilot papers was linear over a wide range of electric field strengths, *Figure 9*, up to the limit of electrical breakdown. However, the volume conductivity shows two linear regions. The hopping mechanism of ionic movement in the paper is quite reasonable for surface conductivity. In the case of volume conductivity, the situation is more complicated, as the mechanism of ionic movement is overlapped by changes in thickness and density due to the electrostatic pressure. The exponential dependence of conductivity on the electric field can be explained by the Schottky effect, i.e., the influence of the electric field on the charge carrier injection from the electrodes. The intensity of the charge carrier injection affects the electrical current through the paper and hence the “apparent” resistivity. The injection, in turn, depends on the contact condition at the paper-electrode interface. This phenomenon was investigated theoretically by Chen and Tse [62]. The role of the Schottky effect was considered by Hanneson et al. [40] but they assumed, based on their experimental results, that this contact effect is improbable in papers. The independence of surface resistivity on paper roughness, as shown in *Figure 8*, confirms indirectly the assumption made by Hanneson et al. On the other hand, the slope of dependence of $\log \mu$ on $E^{0.5}$ depends on the PCC filler content (*Figure 9*) and on the salt (NaCl) content. Therefore, contact effects cannot be totally excluded.

As shown in *Figure 9*, the Poole-Frenkel approach of the hopping drift of ions can be applied to paper, although the electric field dependence is overlapped by paper compression effects in the case of volume conductivity.

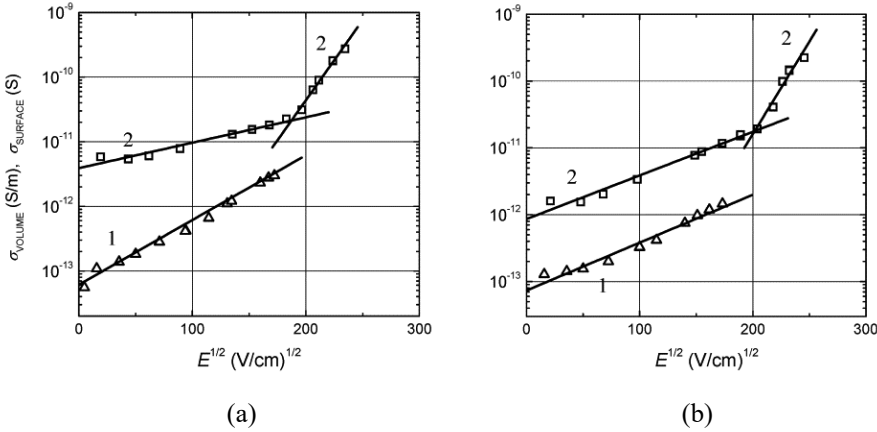


Figure 9. Log conductivity plotted as a function of the square root of the electric field (Poole-Frenkel effect). 1 – surface conductivity, 2 – volume conductivity. a – no filler, b – 15 % filler. [Publication II]

5.2.2 Effect of NaCl and moisture content

The surface and volume resistivities of 160 g/m² pilot paper at 100 V are shown in *Figure 10* on a logarithmic scale as a function of RH. The exponential dependence of resistivity on RH or paper moisture content is well known, but different mathematical expressions for this dependence have been proposed [30, 31]. The dependence of surface and volume resistivity on RH acquired with the controlled set of pilot papers can be expressed as

$$\rho = \rho_0 e^{\frac{-RH}{B}} \quad (4)$$

where ρ_0 is the resistivity at zero moisture content and B is the factor which reflects the sensitivity of the paper resistivity to the moisture content or to the RH at which the paper was conditioned. This exponential dependence on moisture is in line with the results of Simula et al. [31] and confirms the effect of moisture on paper conductivity.

The addition of salt slightly increases the ability of paper to absorb moisture and the equilibrium moisture content at a given RH, but the hygroscopic effect itself is responsible for only a small part of the resistivity decrease with the increase of NaCl content in this data: the hygroscopic effects on absolute moisture due to the addition of NaCl are minimal compared to the effects of RH-level changes in this experiment design. Furthermore, the hygroscopic effects on resistivity are greatly overlapped by the changes in ion content due to variation in NaCl dosing, as evident in *Figures 10 and 11*. This conclusion contradicts the conclusion drawn by Soetanto et al. [35] on the importance of the hygroscopic effects on paper resistivity.

When the RH was 60 % or lower, the range in which the electrical and dielectric measurement results were obtained, the relationship between absolute and relative moisture content was almost linear, tested with 160 g/m² pilot paper (correlation coefficients > 0.99), *Figure 11*.

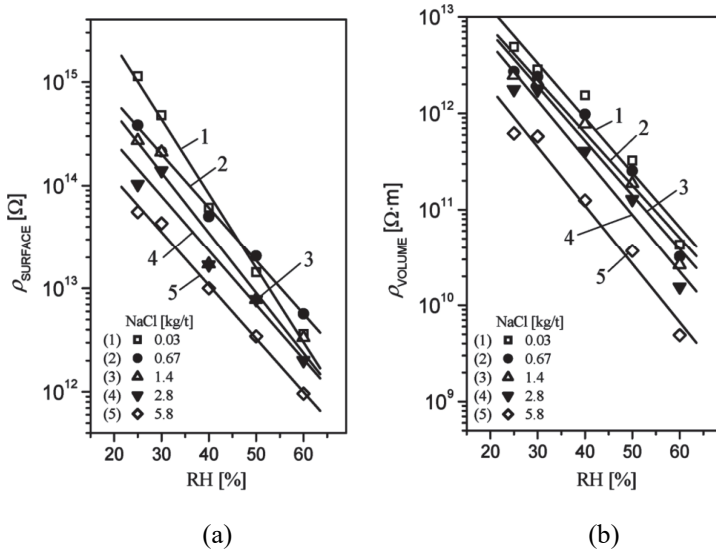


Figure 10. Surface (a) and volume (b) resistivity with 100 V test voltage as a function of RH. 160 g/m² paper with different amounts of NaCl. [Publication IV]

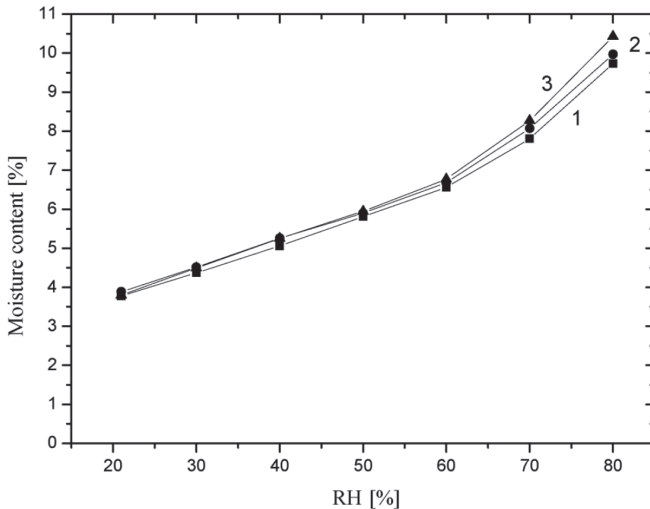


Figure 11. Moisture content dependence on RH and on NaCl addition with 160 g/m² paper: 1- no salt added, 2 – 1.4 kg/tonne, 3 – 5.8 kg/tonne. [Publication IV]

At high voltages (500 - 1000 V), the resistivity decreased as a function of NaCl content, particularly at low dosages for paper conditioned at 50 % RH [Publication IV]. At lower test voltages (10 or 100 V), the effect was smaller, and this was also the case at high voltages when the paper was drier (30 % RH), where the log resistivity decreased more linearly with increasing NaCl content. This behavior was independent of paper thickness (grammage). It can be assumed that when water molecules are available in hydroxylic groups for ion conduction and the field strength is high, increasing mobility, the resistivity is very sensitive to the ion content of paper at low concentrations. Above a certain concentration limit, the resistivity decreases at a slower rate when more salt is added. These results are in agreement with the analysis of Lowe et al. on conductivity in single fibers [63].

5.2.3 Effect of temperature

Both surface (Figure 12) and volume conductivities depend strongly on the temperature, for all the paper grammages and NaCl content investigated. The electrical conductivity of paper increases with increasing temperature. The temperature dependence of volume conductivity is similar to that of surface conductivity, but the data scatter is larger because of the influence of temperature on the thickness and mechanical deformation of paper due to electrostatic pressure [Publication V].

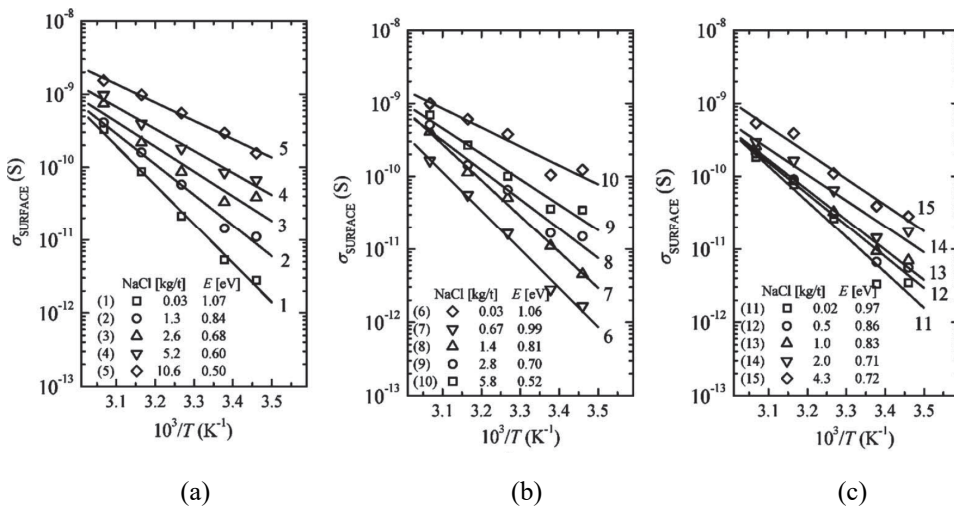


Figure 12. Surface conductivity of papers with different amounts of NaCl as a function of the inverse of temperature on a logarithmic scale. Grammage: a - 90 g/m², b - 160 g/m², c - 230 g/m². [Publication V]

It is known from measurements with cotton cellulose [64, 65] that the absolute moisture content decreases with increasing temperature at a certain relative humidity. For example, in the data presented by Eklund and Lindström [64], at 50 % RH, cotton cellulose had approximately 1.5 % lower absolute moisture content at 50 °C, compared to 10 °C. Even when this impact is presumably smaller with paper that also contains materials other than fibers, this emphasises and verifies the importance of temperature on conductivity. Conductivity increases with temperature, Figure 12, despite the decrease in absolute moisture content which reduces conductivity. This also applies to the

dielectric and charging properties discussed in 5.3.2 and 5.4.3, in the results of which the effect of temperature dominates over the effects of a moderate decrease in moisture with temperature.

Analogous with the results of Murphy [66] for dry cellulose and those of Simula and Niskanen [31] for viscose-kraft fiber mixtures, the conductivity-temperature dependence of fine papers containing different amounts of NaCl is exponential. The dependences plotted in a semi-logarithmic diagram are approximately linear with substrate-specific slopes:

$$\sigma = \sigma_0 e^{\frac{-E_a}{kT}} \quad (5)$$

where σ_0 is a constant, E_a is the thermal activation energy of conductivity, k is Boltzmann's constant, and T is the temperature in Kelvin. For surface conductivity, E_a depends on the NaCl content (Figure 13a). It decreases with increasing NaCl content and does not seem to be dependent on grammage. The volume conductivity behaves in a similar manner to the surface conductivity, but the activation energy is dependent on grammage, although the data scatter is larger (Figure 13b). The activation energy augments with increasing grammage and this effect can be attributed to the temperature-dependence of paper thickness and to compressibility. It must be noted that the activation energy for conductivity is significantly higher than the analogous coefficient for potential decay, B_t (see 5.4.3). This indicates that the temperature-dependence of potential decay cannot be defined only by the dependence of conductivity on temperature. Other factors must also be taken into account; these include, for example, the formation of space charges, the dependence of conductivity on electric field strength [Publication II], and polarization effects [Publication III], all of which are temperature-dependent [Publication V].

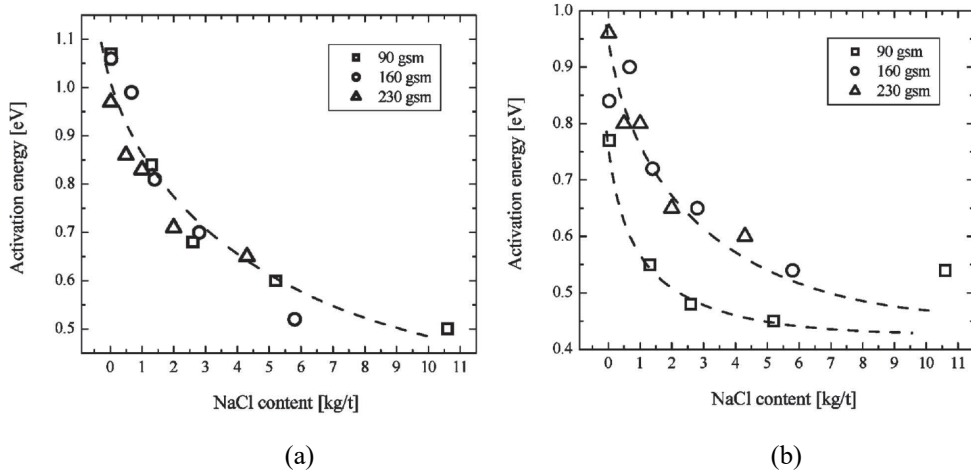


Figure 13. Activation energy of surface (a) and volume (b) conductivity of papers with different grammages as a function of NaCl content. [Publication V]

5.2.4 Effects of coat weight and base paper on conductivity

Pigment coating changes the electrical conductivity of paper. With increasing mineral coating weight on the 160 g/m² low-conductivity (no NaCl) base paper, the surface conductivity increased, whereas the volume conductivity remained unchanged (*Figure 14a*). In this arrangement, the mineral coating layer had an essentially higher conductivity than base paper, and the surface and volume conductivities of the coating layer material (dry coating film measured on Al. foil) were strongly dependent on the electric field. The dependence of the surface conductivity on coat weight with 160 g/m² paper can thus be attributed to the fact that the surface electrical conductivity becomes higher with increasing mineral coating and ion content on the paper surface, whereas the volume conductivity is much less affected, because of the greater impact of the lower electrical conductivity of the base paper, *Figure 14a*.

During the coating process, a proportion of the liquid phase from the coating dispersion penetrates into the base paper, and this liquid phase influences the conductivity gradient in the base paper top layer. The depth of penetration of the pigment particles, on the other hand, is limited and the coating penetration can be expected to have a greater effect on the volume conductivity of the lower grammage paper. Indeed, the volume conductivity dependence on the coat weight was stronger in the case of the 90 g/m² paper (*Figure 14b*) compared to 160 g/m² paper (*Figure 14a*).

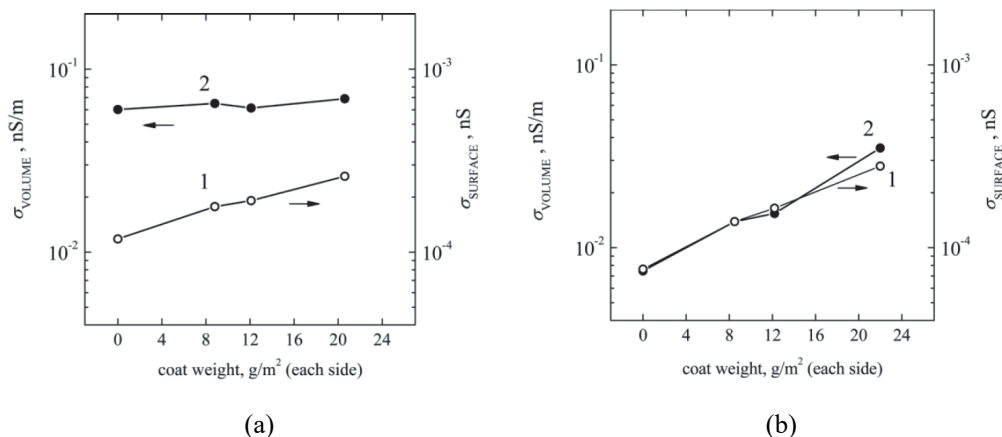


Figure 14. Influence of coat weight on surface (1) and volume (2) conductivity at $3 \cdot 10^4 - 5 \cdot 10^4$ V/cm electric field for 160 g/m² (a) and 90 g/m² (b) base papers without NaCl addition. [Publication VI]

The surface conductivity of the coating material without base paper was higher than that of the coated paper, because the conductive coating components partly penetrate into the base paper. In general, the surface conductivity is not equal to the conductivity of the surface layer, but the surface conductivity values measured in fact reflect the conductivity of this layer together with some of the bulk material adjacent to it. The surface conductivity of the coated paper measured is therefore the conductivity of a mixture of coating and base paper components, *i.e.* cellulose fibers, sizing chemicals, starch, retention aids, filler, and water. The electrical conductivity of this mixed layer is therefore higher than that of the uncoated paper, but lower than that of the coating material itself, and the thickness of this mixed layer is dependent on coat weight (the amount of liquid in the blade coating). With increasing coat weight, both the thickness of the coating and mixed layers increase,

augmenting the surface conductivity. The magnitude of the volume conductivity change with increasing coat weight depends on the paper thickness (grammage), i.e., the depth of coating penetration vs. paper thickness; with low grammage paper, the coating can penetrate throughout the bulk of paper causing a dependence between surface and volume resistivity, as seen in *Figure 14b*.

The penetration of the coating color soluble in water (mono- and multivalent ions, surface-active agents, monomers, and polyelectrolytes) needs to be recognized when analyzing the differences in base paper conductivity and the conductivity of coated paper. However, the amounts of these coating compounds are relatively small compared to the effects of base papers with NaCl, and they are not expected to cause any change in the volume conductivity comparable to the effects of NaCl addition on base paper [Publications IV and VI].

The influence of coat weight on the electrical conductivity of papers is dependent on the base paper properties, *Figure 15*. A higher dosage of sodium chloride in the base paper increased both the surface and volume conductivities of the coated paper. At low NaCl concentrations, the surface conductivity did not substantially change with increasing coat weight. The volume conductivity decreased when the coat weight was increased (*Figure 15*), whereas the volume conductivity was fairly independent of coat weight on the salt-free base paper (*Figure 14a*). Appropriate combinations of coat weight and base paper ion content can be utilized when optimizing the surface and volume conductivities of paper.

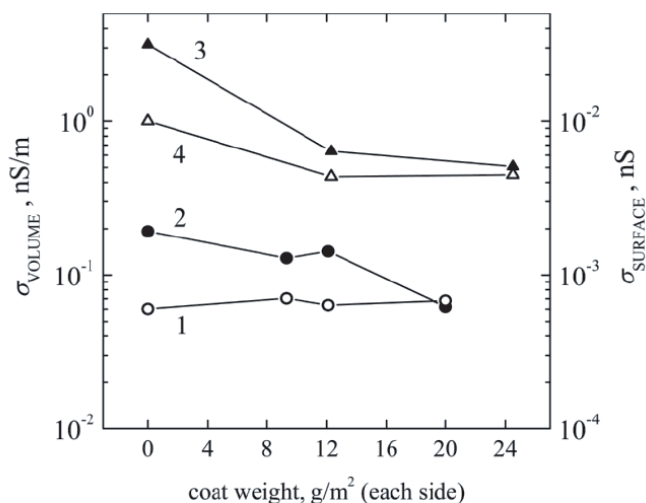


Figure 15. Influence of coat weight and salt concentration on surface (1,4) and volume (2,3) conductivity at $3...5 \cdot 10^4$ V/cm electric field. 160 g/m² paper with 0.75 kg/t NaCl (1, 2) and 6 kg/t NaCl (3, 4). [Publication VI]

A summary of the main results of this section:

- The electric field applied has a great influence on the resistivity of paper.
- The Poole-Frenkel approach can be applied to paper substrates.
- The dependencies between paper resistivity or conductivity vs. relative humidity of paper and temperature of paper are exponential. The slope of the dependency of conductivity vs. inverse of temperature of paper in a semi-logarithmic scale is influenced by the ion (NaCl) content of paper.

5.3 Dielectric properties of paper

The dielectric constant (permittivity), $\epsilon(\omega)$, consists of real and imaginary parts: $\epsilon(\omega) = \epsilon'(\omega) + i\epsilon''(\omega)$, where $\omega = 2\pi f$. The real part of the dielectric constant ϵ' describes the ability of a material to polarize and store energy in a capacitor with a DC voltage applied. The relative dielectric constant (relative permittivity) is the ratio of the real part of the dielectric constant of the material and that of the vacuum. Polarization of a material takes time and is, therefore, frequency-dependent if the capacitor is connected to an AC voltage. The imaginary part of the dielectric constant ϵ'' describes the energy loss within the material in the resulting alternating electric field and includes the effects of dielectric loss and conductivity. The loss tangent or the ratio between the imaginary and real parts of the dielectric constant, $\text{tg}(\omega) = \epsilon''(\omega) / \epsilon'(\omega)$, is often utilized to characterize the dielectric properties of a material in an alternating electric field.

5.3.1 Influence of moisture and ion contents

The real part of the dielectric constant (across the paper) was found to be strongly dependent on frequency, as expected, *Figure 16*. More interestingly, the dependency of ϵ' on frequency was stronger at higher NaCl contents and there was no flattening of the ϵ' frequency dependence at low frequencies in the investigated frequency range. When the frequency increased, the real part of the dielectric constant gradually decreased, but no step drop was observed. This must correspond to a comparatively large-scale polarization phenomenon with a long relaxation time.

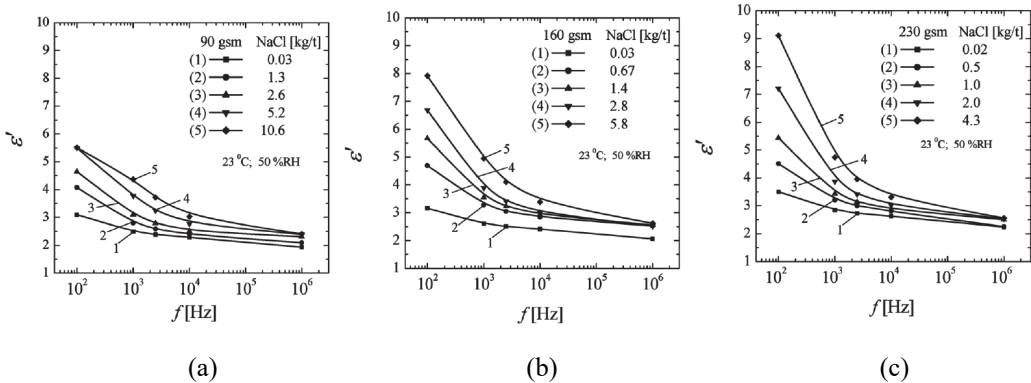


Figure 16. Real part of the dielectric constant ϵ' of 90 (a), 160 (b) and 230 g/m^2 (c) papers with different NaCl amounts as a function of frequency at 50 % RH and 23 °C. [Publication IV]

The effect of NaCl on ϵ' was more pronounced at low frequencies, while at 1 MHz frequency, the differences were quite small. The increase of ϵ' at low frequencies was not linearly proportional to the increase in NaCl content, meaning that all the ionic species added were not available for polarization. It is evident in *Figure 17* that the increase in ϵ' depends not only on NaCl content but also on RH, i.e., on the moisture content, water contributing to the polarization via its own polarization and by influencing the number of ionic and other polarizable groups in the paper structure.

It is considered that the amount of water and the possibility of the water molecules to orientate in the paper structure have a significant impact on the polarization phenomenon in paper [31, 32]. Water in

paper can be localized (adsorbed) and firmly bound to the cellulose structure as water molecules that cannot rotate, adsorbed but appearing as relatively free molecules, and molecules as free as in liquid water. Also, fibers, on which water is adsorbed, are orientated in different directions with respect to the applied electric field [31], causing the amount of types of water having different orientation possibilities with respect to the electric field direction to become significantly larger. These types of water have different polarization properties and different relaxation times and this contributes to the observed dependence of the real part of the dielectric constant on the frequency. The increase in the real part of the dielectric constant ϵ' cannot be explained by simple summation of the dielectric properties of NaCl and H₂O.

The real part of the dielectric constant ϵ' also increased with grammage and consequently with thickness at all frequencies, and the influence of NaCl on ϵ' was greater in paper with higher grammage (Figure 17). A possible cause of the change in dielectric properties could be the difference in paper density [31, 37], but in the case of these pilot-made paper samples, the density varied within a small range and was not influenced by the addition of NaCl [Publication IV]. The results do not therefore need to be corrected in this case to constant density or polarizability per mole, using the Clausius-Mossotti relation [44], and the very small density differences cannot explain the significant increase in the real part of the dielectric constant ϵ' . The addition of NaCl can be assumed to change the microstructure of paper, resulting in changes in the polarizability of water and in the amount and type of polarizable groups in the paper constituents. It is also possible, due to the method of introducing NaCl into the paper (size press), that the surface and bulk of the paper containing NaCl have significantly different conductivities. If so, space charges which influence the dielectric properties can be formed in paper in the presence of an electric field, causing a dependency between ϵ' and the thickness of paper.

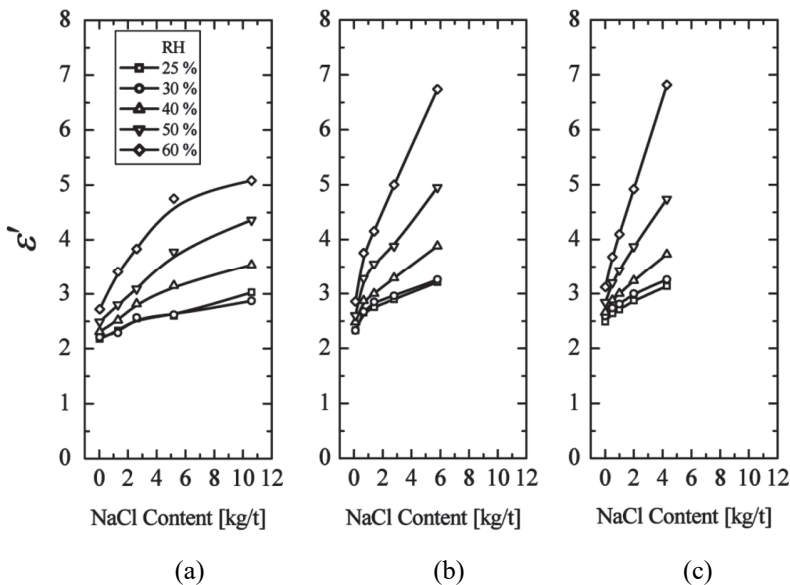


Figure 17. Real part of the dielectric constant ϵ' of 90 (a), 160 (b), and 230 g/m² (c) papers at 1000 Hz frequency and 23 °C as a function of NaCl content at different RH values. [Publication IV]

The addition of NaCl had a significant impact on the dielectric loss (Figures 18 and 19). The dielectric loss factor $\tan \delta$ increased with NaCl content at 30 % and 40 % RH in all investigated cases and with

NaCl content up to ~ 3 kg/t, the dielectric losses increased at all RHs. However, at higher NaCl content and RH levels, the dielectric loss factor $tg \delta$ decreased. Increasing the dosing of NaCl in paper naturally increased the electrical conductivity and this caused the $tg \delta$ to increase up to a limit, but this cannot explain the unusual $tg \delta$ dependence on NaCl content and RH in the ranges where $tg \delta$ decreased.

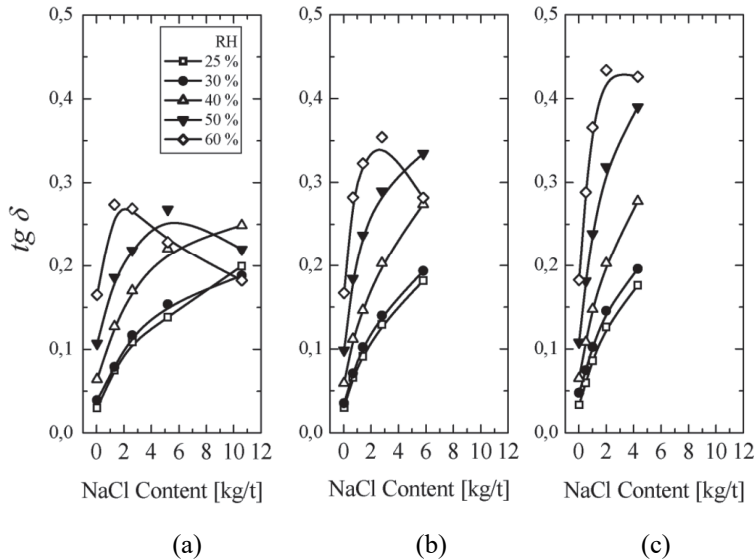


Figure 18. Dielectric loss factor $tg \delta$ of 90 (a), 160 (b) and 230 g/m² (c) papers at 1000 Hz frequency as a function of NaCl content at different RH values. [Publication IV]

The frequency dependence of $tg \delta$ provides additional information. Dielectric losses depend on frequency and the addition of NaCl changes this dependency, as can be seen in *Figure 19*. At low NaCl content, $tg \delta$ gradually decreased with frequency, which is in agreement with the results of Simula et al. [31] and Morton et al. [32]. However, at higher NaCl contents, the maximum appeared at a frequency of about 1 kHz. This could mean that at higher NaCl contents new structures are formed that have a relaxation time of the order of 10^{-3} s. If so, then this is additional evidence that salt addition changes the microstructure of paper, which then influences its electrical and dielectric properties. A similar effect on cotton fibers was observed by Morton et al. [32]: at a relatively low RH (up to 56 - 60 %), the power factor gradually decreased with frequency, but at higher RH (68 - 95 %) a maximum appeared at 0.1 - 1.0 kHz, attributed to some element with a relaxation time of the order of 10^{-2} - 10^{-3} s.

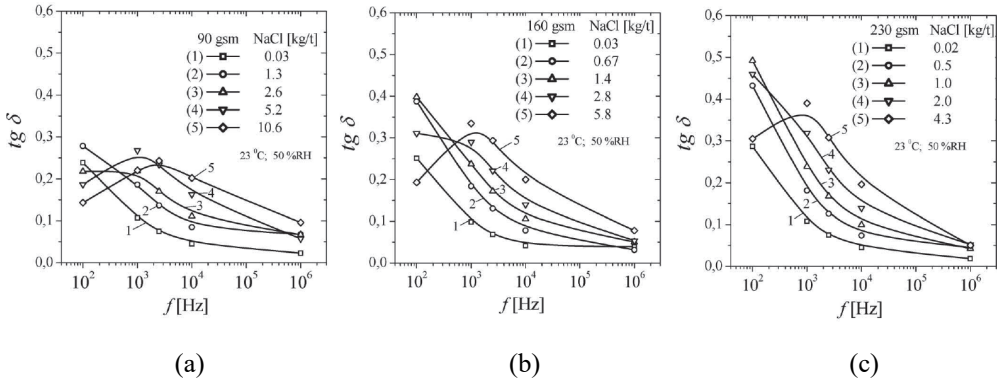


Figure 19. Dielectric loss factor $tg \delta$ of 90 (a), 160 (b), and 230 g/m^2 (c) papers with different NaCl contents as a function of frequency at 50 % RH and 23°C . [Publication IV]

The assumption that NaCl leads to the formation of new microstructures that influence the electrical properties of paper is supported by the dependence of the dielectric losses on RH. As was shown by Simula et al. [31], dielectric losses increase with RH and this increase is related to water in cellulose fibers. *Figure 18* shows that the dielectric loss factor $tg \delta$ does not always increase with RH: in many cases, at high NaCl contents, $tg \delta$ is lower at higher RH. Thus, the polarization freedom of at least part of the water is changed in the presence of NaCl. It can also be supposed that NaCl forms new structures in the presence of water in paper, which have specific dielectric properties and influence the ion movement and paper conductivity.

5.3.2 Influence of temperature

The real part of the dielectric constant and the thickness determine the paper electrical capacity and consequently the charging potential at a given surface charge. The dielectric constant at a frequency of 1 kHz depends significantly on temperature and on NaCl content, *Figure 20*. ϵ' increases with temperature and NaCl content. At a frequency of 1 MHz, the temperature effect was similar but weakly expressed.

The temperature effect can be explained by temperature reducing the restraints of the dipoles [32, 42, 43], resulting in an increase of permittivity. Some of the water molecules in cellulose fibers can act as if they are restrained in a manner similar to the restraints in ice [42], but, at higher temperatures, the immobilization of such water molecules becomes weaker and the dielectric constant increases. The addition of ionic salt, NaCl, increases the concentration of polarizable elements in paper. The interaction of NaCl with water and cellulose hydroxyl groups can form new elements, which polarize in an electric field; these elements will also then lead to an increase in permittivity.

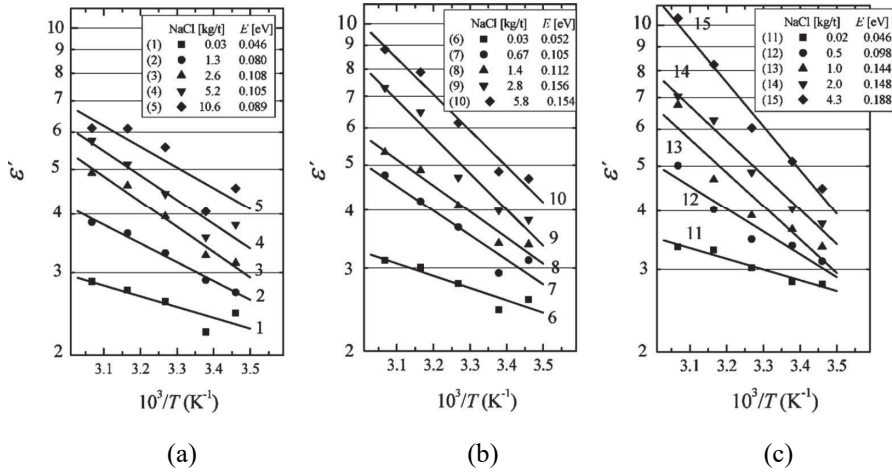


Figure 20. Real part of dielectric constant at 1 kHz frequency of paper with different amounts of NaCl as a function of inverse of temperature on a logarithmic scale. Grammage: a - 90 g/m², b - 160 g/m², c - 230 g/m². [Publication V]

The relationship between the dielectric constant ϵ' and temperature in a semi-logarithmic diagram is approximately linear and can be expressed as an exponent with an activation energy. This activation energy at a frequency of 1 kHz increases with increasing NaCl content, *Figure 21*; at a frequency of 1 MHz such dependence of ϵ' on NaCl content was weak but still evident. This nature of the activation energy of ϵ' may indicate that salt induces formation of new elements in paper, which require additional energy for polarization.

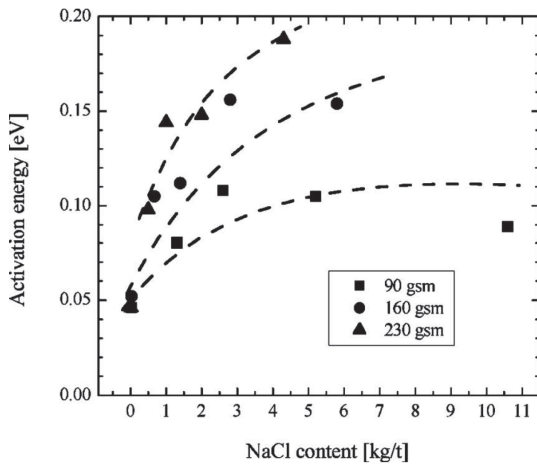


Figure 21. Activation energy of the real part of the dielectric constant at a frequency of 1 kHz for papers with different grammages as a function of NaCl content. [Publication V]

The temperature and frequency dependences of the dielectric loss factor $tg\delta$ provide additional information on the polarizable elements: $tg\delta$ depends greatly on temperature and this dependence is clearly influenced by the presence of NaCl, *Figure 22*. At the interesting frequency of 1 kHz, the addition of NaCl even changes the sign of the slope in a logarithmic scale of $tg\delta$ vs. the inverse of T . At higher grammages, the effect of slope change is weaker, but the lower grammages present a wider range of NaCl dosage levels [kg/t] applied in surface sizing; a more detailed analysis shows that the amount of NaCl indeed has the greatest effect: papers with the same NaCl content exhibit the same temperature dependence regardless of their grammage and the change in the slope of the dependence of $tg\delta$ on temperature is caused only by NaCl. NaCl initiates the formation of new structures in the paper, which influences the dielectric constant and conductivity of paper.

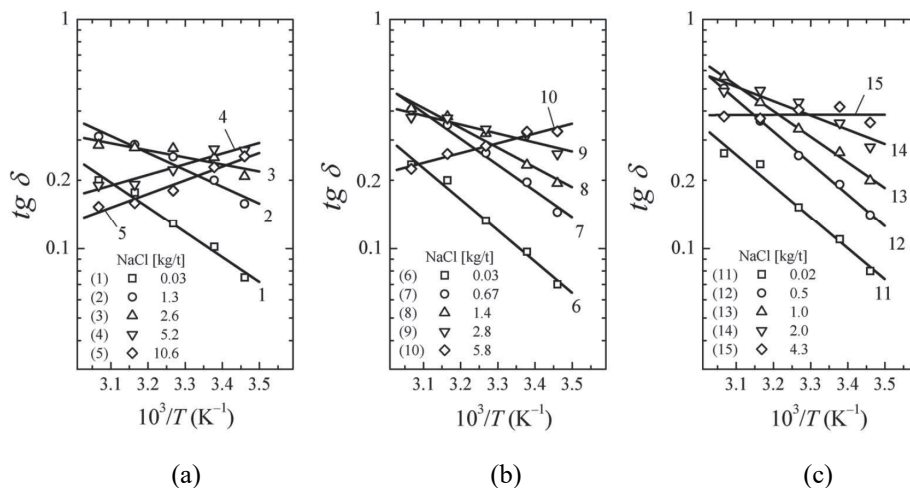


Figure 22. Dielectric loss factor at 1 kHz frequency of paper with different amounts of NaCl as a function of inverse of temperature on a logarithmic scale. Grammage: a - 90 g/m², b - 160 g/m², c - 230 g/m². [Publication V]

These structures can also be expected to affect the frequency dependence of $tg\delta$; indeed, the addition of salt essentially changes this dependence, causing a new maximum appearing at a frequency $10^3 - 10^4$ Hz (*Figure 23*). The position of the maximum depends on temperature and salt content; with increasing NaCl content and temperature the maximum moves towards a higher frequency. Similar changes occurred at a frequency of 1 MHz, although the changes were weak.

Dependencies between the dielectric constant and dielectric loss factor and NaCl content, temperature, and frequency confirm the changes in the paper microstructure due to the formation of new structures with specific dielectric properties in the presence of NaCl and water.

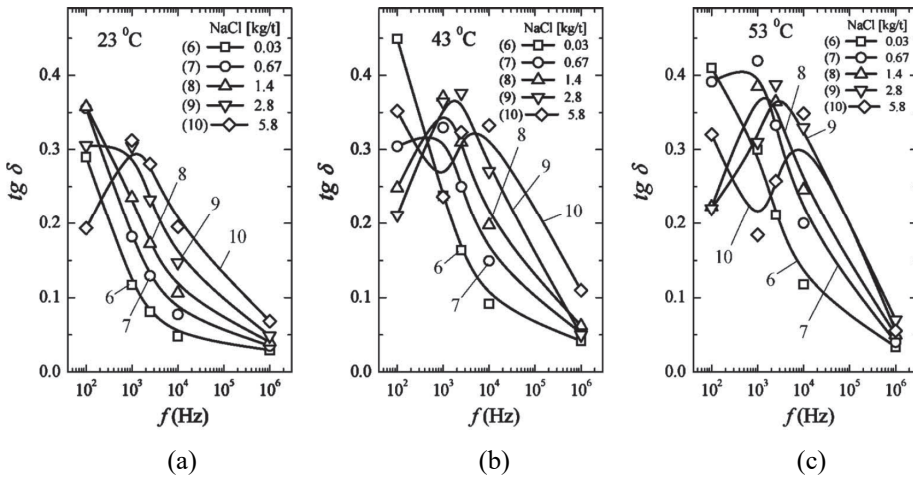


Figure 23. Dielectric loss factor of 160 g/m² paper with different amounts of NaCl as a function of frequency at different temperatures. [Publication V]

5.3.3 Influence of coating

When the papers were pigment-coated, the increase in the coat weight increased the ϵ' of the paper over the whole frequency range of 10² - 10⁶ Hz, and ϵ' decreased with increasing frequency, this dependency being the same, independent of the coat weight, *Figure 24*. The density of the paper increases with coating and calendering but the density change cannot explain the increase in ϵ' ; when the density increases by about 15 %, the ϵ' increases in most cases by 30 % or more. This increase is attributed to the dielectric constant of the coating mixture including the pigments. The value of the real part of the dielectric constant of the base papers, ranging from 2.6 - 7.0 due to the NaCl addition of 0 - 6 kg/t does not significantly affect the input of the coating on the ϵ' [Publication VI].

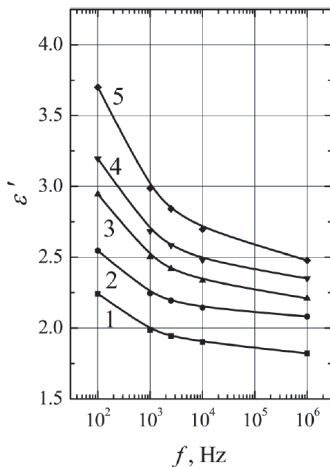


Figure 24. The influence of coat weight on dielectric constant ϵ' for 90 g/m² paper in frequency range 10² - 10⁶ Hz: 1) uncalendered base paper, 2) calendered base paper, 3) with a coat weight of 9.3 g/m² per side, 4) with a coat weight of 12.1 g/m² per side, and 5) with a coat weight of 22 g/m² per side. [Publication VI]

A summary of the main results of this section:

- The real part of the dielectric constant at low frequencies is strongly dependent on the ion and moisture contents of paper. In the presence of a sufficient amount of moisture and NaCl, there is a maximum of dielectric loss, indicating structures with a relaxation time in the order of 10^{-3} s forming in paper in these conditions.
- Dielectric constant and loss are highly dependent on temperature. The slope of the relation between the real part of the dielectric constant vs. inverse of temperature on a semi-logarithmic scale is dependent on the NaCl content. The activation energy depends on the NaCl content and also on paper grammage (thickness).
- The increase in the real part of the dielectric constant with paper calendering and coating is due to the combined effects of increased density and the dielectric properties of the coating mixture.

5.4 Charging and charge decay of paper

5.4.1 Charge acceptance

In dosed charging, the surface potential of paper increases and reaches a maximum value, as in *Figure 25* for a 250 g/m^2 coated paper, after continuously applying charge doses on the paper with a corona. The maximal potential depends on the corona wire voltage and is significantly dependent on the magnitude of charge doses, i.e., the corona potential, although the charging transients in the initial stage of charging are almost independent of the corona voltage. In the initial stage of charging, i.e., at low electric fields, the charge leakage is relatively low and the deposited charge in the particular charging cycle is significantly greater than the dissipated charge in the same cycle. In contrast, at higher surface potentials, i.e., at stronger electric fields, leakage increases and more intensive charging is needed to reach a given surface potential. The maximum or equilibrium potential at a given corona voltage is reached when the charge deposition intensity and the leakage become equal.

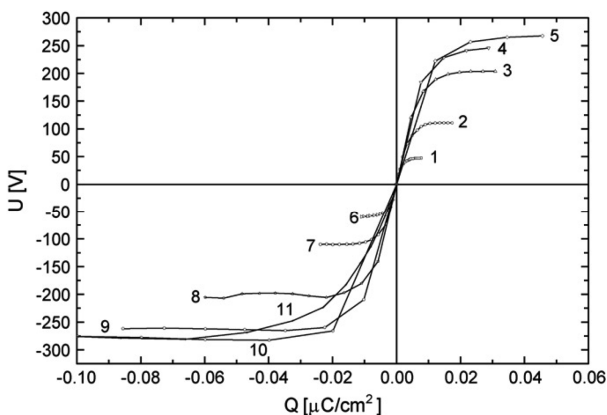


Figure 25. Dosed charging under different charging conditions. Q – charge deposited onto paper, U – surface potential of paper. Corona charging voltages: 1 and 6: 5.0 kV; 2 and 7: 5.5 kV; 3 and 8: 6.5 kV; 4 and 9: 7.5 kV; 5 and 10: 8.5 kV; 11: the voltage was changed from 5.0 to 7.5 kV; 1 to 5 positive charging and 6 to 11 negative charging. Coated paper, 250 g/m^2 . [Publication III]

The maximum charging potentials of commercial uncoated and coated papers within a grammage range of 90 - 300 g/m² are on different levels since the products differ in materials – and, thus, the maximal charge acceptance levels are not comparable. However, more interesting is the tendency for coated papers to exhibit charge acceptance saturation at high corona voltages, *Figure 26*. The fact that the saturation level is not dependent on paper thickness at 62 % RH shows that the electrical capacitance does not (alone) determine the maximum charging potential. The effect of polarization was further evaluated with pilot paper having varying coat weight and base paper NaCl content. Mineral coating and NaCl addition to the pilot-made coated papers increase the ϵ' which, if that were the dominating effect, should lead to a decrease in the paper surface potential at a given charge on its surface; yet no decrease in the charging potential with increasing ϵ' was observed [Publication VI]. Especially with high NaCl contents of base paper, the charging potential was higher at higher ϵ' induced by an increase in coat weight. The conductivity or charge carrier mobility through different material layers influences the charge decay and therefore the maximum charging potential of such a paper more than the polarization, in an arrangement where the potential is measured shortly (within 0.1 - 0.2 s.) after applying the surface charge.

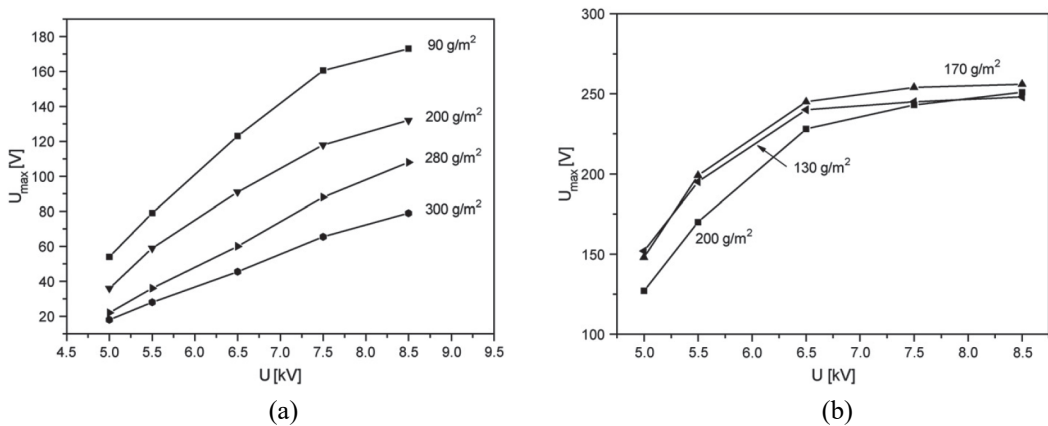


Figure 26. Maximum charging potential of paper with different grammages versus corona voltage at 62 % RH, a - uncoated, b - coated. [Publication III]

5.4.2 Charge decay

The potential decay of paper is often characterized by a single value of the decay curve, e.g., with a time $t_{0.5}$ (s) at which the potential has decreased to half ($V_{0.5}$) of its initial value. This does not describe the discharge process of paper accurately, as there can be several mechanisms influencing the charging-discharging processes, including charge carrier movement in the electric field, polarization, and space charges. The impact of these mechanisms vary depending on paper materials and structure. The decay of charge in paper is not an exponential function of time, but regions with different characteristics can be distinguished, thus, the charge decay curve requires two or more exponential functions similar to the pilot paper types presented in *Figure 27*. The time constants of the exponential functions depend on the thickness (capacitance), conductivity (RH), and on the dielectric properties, indicating that the charge dissipation is governed by several mechanisms [Publication III].

In [28], it was suggested that several decay constants correspond to different charge carriers, but also other mechanisms including the field dependency of the charge carrier mobility, polarization effects, space charges, and thickness changes due to the electrostatic pressure can give rise to decay phenomena with differing time constants.

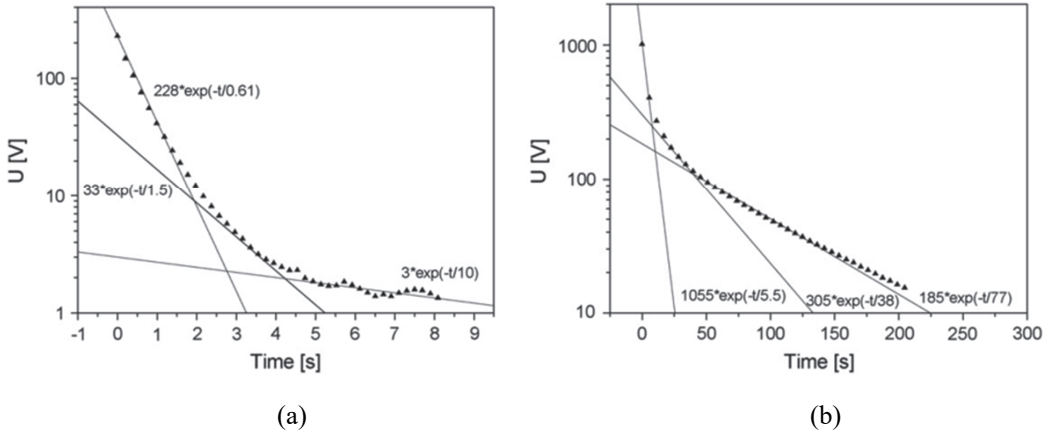


Figure 27. Discharge kinetics and fitting with several exponential functions, a - uncoated paper, 90 g/m², RH 50 %, b - coated paper, 250 g/m², RH 30 %. [Publication III]

The effect of polarization can be investigated by discharging with the opposite polarity. *Figure 28* compares the discharge kinetics of a normal discharge and a forced discharge with opposite corona charging to approximately 0 V. After forced discharge to zero volts by opposite charges, the potential rises again and, after reaching the second maximum, decays again with time. This is observed for both charging polarities, and is ascribed to the orientation-polarization of paper constituents with relatively long depolarization times and by the formation of space charges. Paper is charged as an electrical capacitor in which polarization also occurs. After the charging is switched off, the potential decays due to conductivity mechanisms, which are in part overlapped by the effect of depolarization influencing in the opposite direction. Depending on the paper type and on the relative input of these mechanisms, different charge decay laws may be observed.

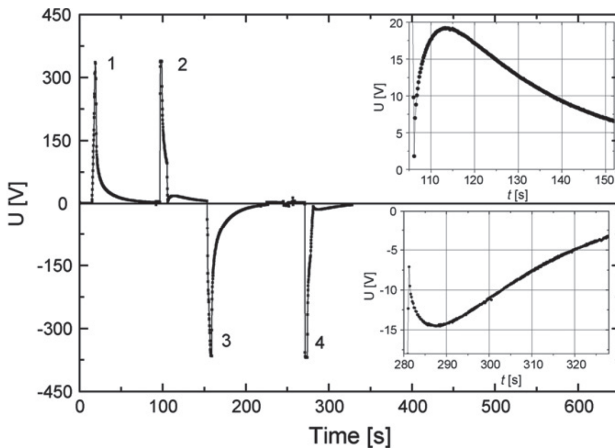


Figure 28. Polarization effect. Conventional discharge kinetics (1,3) and kinetics after paper discharge by opposite charge (2,4). Uncoated pilot paper, 160 g/m². Inserts are enlargements of curves 2 and 4 after discharge with opposite polarity. [Publication III]

5.4.3 Temperature vs. charging and charge decay

The charging potential U_0 depends greatly on temperature and salt content (Figure 29). It is dependent on grammage (increases with grammage) and decreases with temperature for all the grammages and NaCl contents examined. The decrease in charging potential with temperature can be considered exponential. This is seen when U_0 and the inverse of temperature are presented in a semi-logarithmic diagram, giving an almost linear relationship. Therefore, the charging potential U_0 can be expressed as a function of temperature as

$$U_0 = A_u e^{\frac{-B_u}{kT}} \quad (6)$$

where A_u is the proportionality coefficient influenced by the grammage (thickness) of the substrate, B_u is the coefficient reflecting the sensitivity of U_0 to the temperature changes, k is Boltzmann's constant, and T is the temperature in Kelvin. B_u is expressed in eV and can be formally considered as an activation energy, but its real physical meaning is not clear.

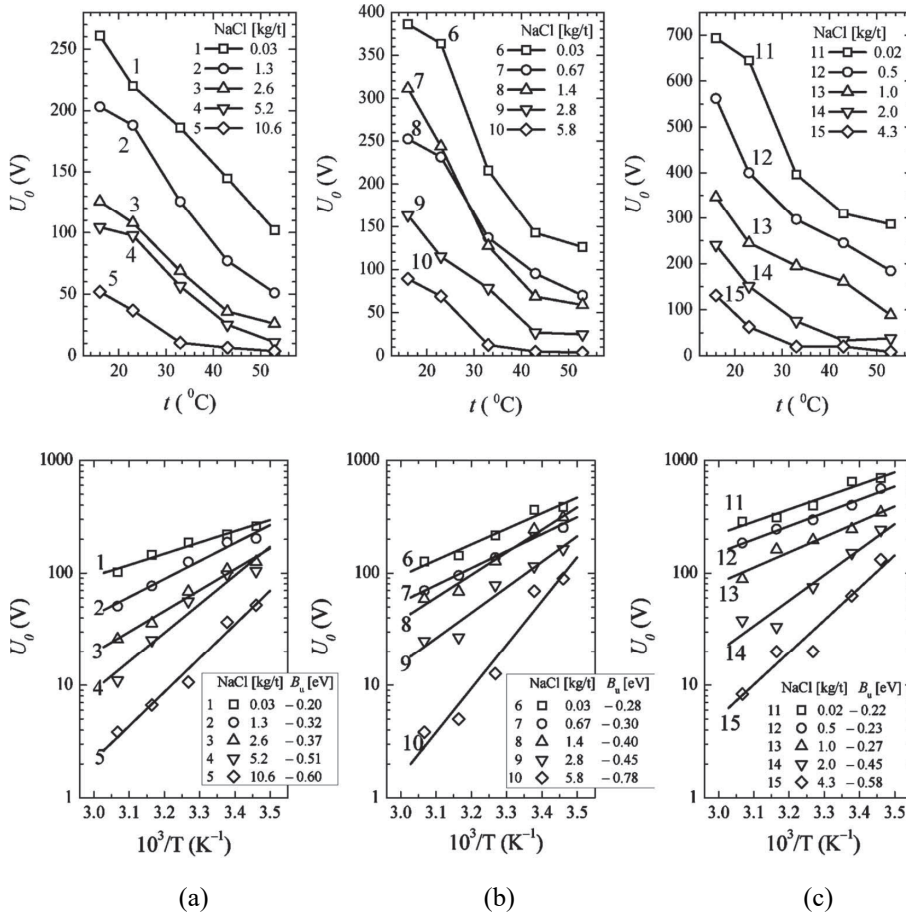


Figure 29. Charging potential of paper with different amounts of NaCl as a function of temperature (top) and as a function of the inverse of temperature on a logarithmic scale (bottom). Grammage: a = 90 g/m^2 , b = 160 g/m^2 , c = 230 g/m^2 . [Publication V]

The absolute value of coefficient B_u increases with increasing NaCl content (*Figure 30*). The charging potential depends (inversely) on the electrical capacity of the paper and (directly) on the charge amount on the paper surface at the time of measurement. This charge depends on the amount of deposited charge and on the leakage of this charge measured as the potential decay rate after charging is stopped. The electrical capacity of a paper type depends on its thickness and dielectric constant and possibly on the formation of space charges during charging. Since all these electrical properties are temperature-dependent, B_u is influenced by a combination of the specific temperature-dependences of potential decay, electrical conductivity, and dielectric constant.

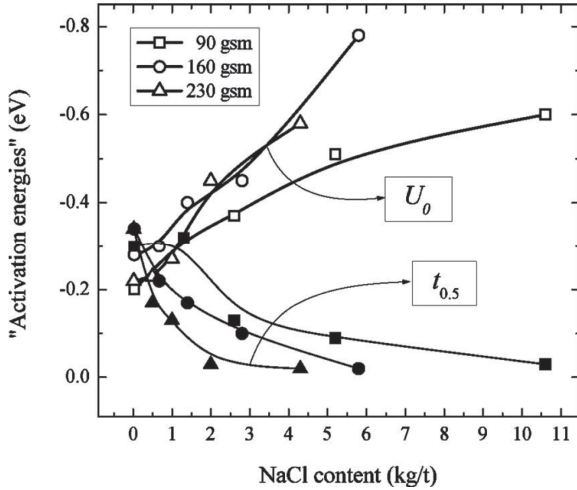


Figure 30. Coefficients (“activation energies”) B_u of charging potential U and B_t of potential half decay time $t_{0.5}$ of paper with different grammages as a function of NaCl content. [Publication V]

As in the case with the charging potential, the potential decay rate also depends on the temperature and the dependence strongly decreases with increasing NaCl content. The temperature dependence of the potential half decay time can be considered exponential, as seen in *Figure 31*: in a semi-logarithmic diagram, the relationships are approximately linear and can be expressed as

$$t_{0.5} = A_t e^{\frac{-B_t}{kT}} \quad (7)$$

where A_t is the proportionality coefficient, B_t is the coefficient which reflects the $t_{0.5}$ sensitivity to the temperature changes, k is Boltzmann’s constant, and T is the temperature in Kelvin. As in the case of charging potential, B_t (expressed in eV) can be formally considered an activation energy but its physical meaning is not clear, especially bearing in mind the character of its change with NaCl content. B_t depends on the NaCl content (*Figure 30*) but, in contrast to the charging potential coefficient B_u , the absolute value of B_t decreases with increasing NaCl content and the decrease is significant: for a 230 g/m² paper, B_t changes from 0.34 with no salt to 0.02 at a NaCl content of 4.3 kg/t.

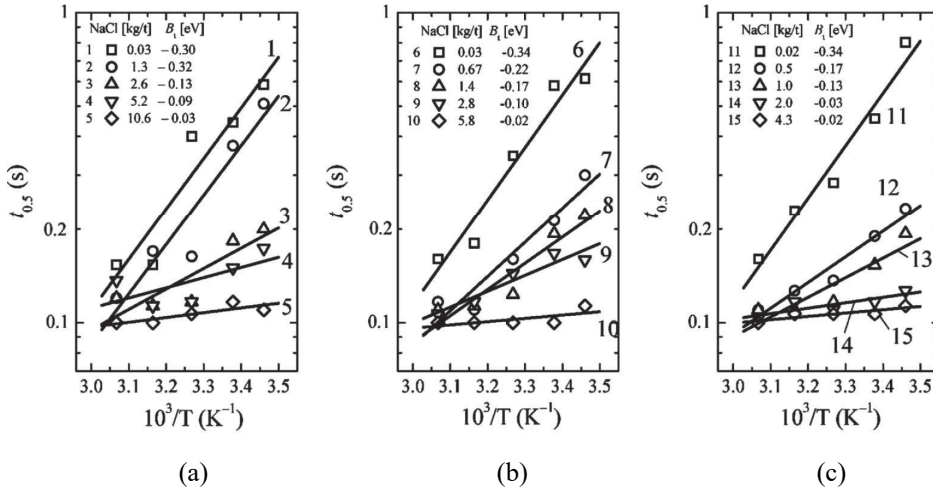


Figure 31. Potential half decay time of papers with different amounts of NaCl as a function of inverse of temperature on a logarithmic scale. Grammage: a = 90 g/m², b = 160 g/m², c = 230 g/m². [Publication V]

A summary of the main results of this section:

- Charge decay is controlled by several overlapping processes and cannot be precisely described with a single exponential function.
- The polarization of paper in charging can be observed experimentally by forced discharging, after which the voltage increases again.
- The dependencies of maximum voltage and charge half-time vs. temperature are exponential, slopes vs. inverse of temperature on a semi-logarithmic scale depending on the NaCl content of the paper.

6. CONCLUSIONS

The aim of this investigation was to analyze the relationships between the traditional paper properties normally utilized as control parameters in papermaking or reported as paper specifications and the electrical and dielectric properties of papers affecting the print quality and runnability in dry toner electrophotography. The former include surface roughness, density, thickness, basis weight, NaCl content, and coat weight and the latter the impact of electric field, surface and volume electrical resistivities, charge acceptance, charge decay, dielectric constant, and dielectric loss. Utilization of pilot-made papers enabled several dependencies between the above-mentioned parameters to be established and verified.

Roughness of low-grammage coated papers impacts the transfer of charged toner particles. Coating and surface parameters with local influence on the electric field become increasingly important with increasing toner coverage, whereas local discharging did not inhibit transfer with the paper roughness levels normal in electrophotographic printing.

Paper charging is dependent on paper thickness, moisture content, base paper ion content, and coat weight. Forced discharging of uncoated paper revealed the impact of polarization. In the potential decay of paper, the depolarization and decay due to conductivity overlap during the different stages of potential decrease, and the charge decay of paper cannot be accurately described with a single exponential function as there are several decay regions with overlapping charge decay mechanisms.

The surface and volume electrical resistivities of paper not only depend on the NaCl content and exponentially on the moisture content, but also on the electric field applied. These effects are related to the amount of free ions and the field influencing the mobility of ions, i.e., conductivity. The surface and volume resistivities of paper are strongly dependent on the electric field strength and this dependence is influenced by the thickness, density, and filler content of the paper. The relationship between the logarithm of surface conductivity and the square root of electric field is linear and the Poole-Frenkel model can be applied to uncoated paper. With volume conductivity, there is a step change in the slope due to the effects of electrostatic compression.

The real part of the dielectric constant ϵ' of paper decreases with increasing frequency and the rate of decrease is strongest at low frequencies, which can be explained by the different binding states of water and other materials in the fiber network with respect to the direction of the electric field. ϵ' increases with increasing NaCl content and increasing relative humidity. However, this increase cannot be explained by summing the effects of the separate dielectric constants when these materials cannot polarize freely. The real part of the dielectric constant of coated papers is influenced both by the addition of pigments and other coating additives with polarizable groups and by the increase in density. The character of the changes in dielectric properties with varying NaCl contents at different relative humidities suggests that salt addition also influences the structure of paper in the presence of water. A maximum was observed in the dependence of the dielectric loss vs. NaCl content and RH, indicating that distinctive structures with a relaxation time in the order of 10^{-3} s are formed in paper in the presence of water and NaCl.

Temperature has a significant impact on the electrical and dielectric properties of paper. The charging potential decreases whereas the electrical conductivity, potential decay rate, and dielectric constant of paper increase with increasing temperature. The dependencies are exponential and these processes can be considered to be dependent on thermal energy. However, addition of NaCl changes the temperature dependence of these properties. In these dependencies, the decrease in activation energy of the conductivity and the increase in the potential decay rate with increasing NaCl content can be explained by a lowering of the barriers for ion movement. The increase in the absolute values of

activation energies of charging potential and dielectric constant were not expected and the cause of these phenomena may be changes in the paper microstructure that alter the physical processes in papers during charging and polarizing. This assumption is supported by the changes in the temperature and frequency dependencies of the dielectric loss.

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