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Effect of NaCl and Moisture Content on Electrical and Dielectric Properties of Paper

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Abstract. *The electrical and dielectric properties of paper are important parameters in electrophotography, influencing both toner transfer and runnability in laser printers and in digital printing machines. These parameters are dependent on the ionic content and moisture content of the substrate. The effects of NaCl content of the substrate and the ambient relative humidity (RH) on the direct current resistivity in different electric fields and on the real part of the dielectric constant and dielectric loss factor at different frequencies have been investigated with experimental papers of different grammages. A physical model of electrical conductivity in papers with ionic addition is proposed, based on the hypothesis that ionic addition not only increases the number of available free ions but also changes the water state and the paper structure, which influence the ionic motion in an electric field. This model is supported by the dependencies of the real part of the dielectric constant and the dielectric loss factor on the NaCl content, RH, and frequency. © 2009 Society for Imaging Science and Technology.*
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INTRODUCTION

The ionic and moisture contents of paper influence the electrical and dielectric properties and thus the toner transfer in dry toner electrophotographic processes. Salts like NaCl are used in papermaking, in addition to the moisture content of the paper, to control the properties of electrophotographic papers and to ensure that the paper responds to the requirements placed on the correct level of static electricity and toner transfer. Toner particles are transferred using an electric field in the printing process, which means that the resistivity and polarization characteristics of paper influence the toner transfer efficiency and the resulting print quality, insofar as these paper properties influence the electric field in the toner layer. The optimal electrical and dielectric properties are also partly dependent on the speed of the printing process as this influences the time available for the electric field development in the transfer nip. The results of direct current (DC) resistivity measurements, which are used to measure paper resistivity, are time dependent,[1] which seriously complicates any absolute comparison of the DC resistivity values and the toner amount transferred in a dynamic printing process. Even with these limitations, DC resistivity can be, and is, widely used in the paper industry to predict the behavior of substrates in electrophotographic processes.[2–5] Chen et al.[6] found, however, that toner transfer efficiency does not depend on paper moisture content or resistivity alone but can be better correlated with paper charge relaxation time constant (product of dielectric constant and resistivity). It is therefore important to have information concerning both the resistivity and the dielectric properties of paper, and the dependencies of these on relative humidity (RH) and ion content of the paper.

The mechanism of electrical conduction in paper is assumed to be ionic, with charge carriers passing through the paper along fibers.[1,7,8] According to Murphy,[7] the charge carriers are cations which move through hydroxyl groups of the polysaccharide chains, but they can move only when the hydroxyl group is bound to a water molecule,[7] 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 num-

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ber of carboxyl groups able to supply cations in an electric field.[8] In addition, any model assuming zero conductivity at zero moisture content does not agree with experimental results. Simula and Niskanen proposed a model in which resistivity is linearly proportional to the fraction of hydroxyl groups without a water molecule since, as in Murphy's model, such sites would stop the path of conduction. The exponential dependence of DC resistivity on moisture content has been clearly demonstrated with various substrates, but different mathematical equations for this dependence have been proposed.[7,8]

Smith[9] found an exponential dependence between conductivity and charge carrier (ion) concentration in kraft pulp handsheets. A mechanism was suggested 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[10] 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 salt content is clearly below this limit, as is the case described in this paper, salt content also becomes an important factor for the level of conductivity.

The salt content also changes the conductivity through hygroscopic effects. Results demonstrating this effect have been presented by Soetanto et al.[11]. Due to the importance of the moisture content for the resistivity of paper, the magnitude of this mechanism must be evaluated when analyzing the relationship between salt addition and the resistivity of paper.

The impact of moisture and ion content on the electrical and dielectric properties of pulp and paper has been extensively investigated. Most of the published data relate to the dielectric properties of capacitor papers,[12–14] but some more recent work has also referred to the polarization of paper from the aspect of electrophotography, using commercial coated and copy papers[8] and viscose-kraft laboratory handsheets.[15] In the case of paper, the most important mechanisms of dielectric polarization are electronic, atomic, and molecular polarization,[1] but space charges in paper,[16] and thus their polarization, cannot be ruled out. The effect of changes in paper moisture content on the dielectric properties is strongly frequency dependent, since the frequency determines which polarization processes can contribute to the polarization. At low frequencies, all these mechanisms can operate. In paper made of wood-free pulp, main components influencing the polarization are carboxyl and hydroxyl groups on the fibers as well as water molecules and free ions. In the electrophotographic imaging process, it is of interest to make measurements at low frequencies, but testing the dielectric properties at higher frequencies can give additional information on the mechanisms and materials contributing to the polarization.

In general, the literature contains few reports on the role of the electrical and dielectric properties of paper specific to electrophotography, and it is difficult to compare data from different sources, especially if the experimental conditions (mainly voltage in DC measurements and properties of papers) are not reported. To be able to optimize the substrate properties for the electrophotographic process, it is necessary to acquire more information on these properties and related phenomena. In this report we describe the effects of ion and moisture content of experimental papers of different grammage on direct current conductivity in different electric fields and on the polarization of the substrates at different frequencies.

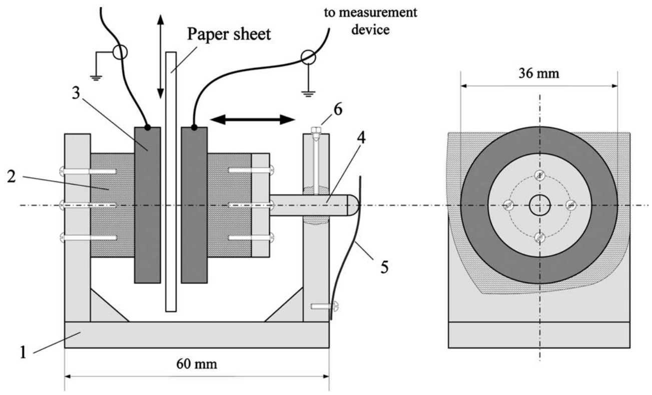


Figure 1. The holder for the measurement of paper dielectric properties: 1–holder base; 2–dielectric; 3–contact electrode; 4–axis of contact electrode; 5–spring, 6–screwbolt.

EXPERIMENTAL

The DC volume resistivity of paper was evaluated in accordance with ASTM D257, and the DC surface resistivity was evaluated using raker-type electrodes because a small distance between the electrodes makes it possible to measure surface resistivity at high electrical field strengths before electrical breakdown. The volume resistivity was calculated from the resistance values using paper thickness values obtained during dielectric measurements (1.5 kPa pressure). The mechanical pressure was maintained constant in all the measurements. The geometry and formulae used in the resistivity measurement are described in detail in a previous publication.[17]

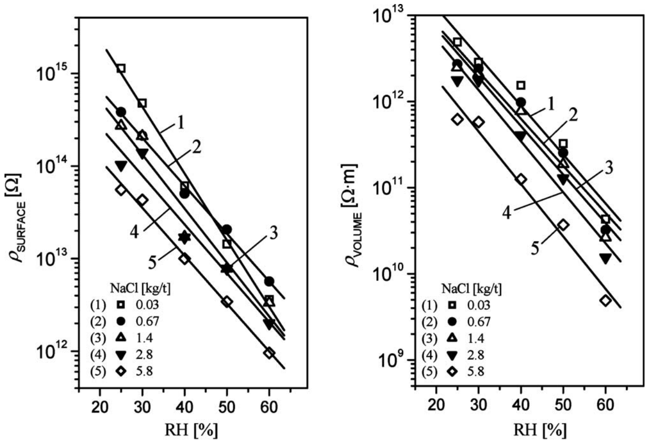
Dielectric properties were evaluated by measuring the electrical capacitance C and the dielectric loss factor $tg\delta$. The capacitance was measured using a special holder (Figure 1) and commercially available bridges: Wayne Kerr Automatic Precision Bridge B905 for measurement in the frequencies range from 100 Hz to 10 kHz, and digital LCR-meter E7-12 for a frequency of 1 MHz.

The capacitance C was first measured with paper inserted into the holder, where electrodes are pressed against the paper by the spring (pressure 1.5 kPa). The distance between the electrodes is fixed by a screw bolt. 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 $\varepsilon = C/C_0$. This procedure also makes it possible to determine the paper thickness $h = \varepsilon_0 S/C_0$, where ε_0 is permittivity of vacuum and S is the area of electrodes. This thickness was used in the calculation of the electric field strength. The measurement of C and $tg\delta$

Table I. Properties of pilot-made papers.

Grammage [g/m2]	Thickness[a] [um]	Density [g/cm^3]	Salt content calculated by means of Cl^- [kg/ton]
89	122	0.73	0.03
90	123	0.73	1.3
89	121	0.74	2.6
91	124	0.73	5.2
91	125	0.73	10.6
158	209	0.76	0.03
169	202	0.84	0.67
167	205	0.81	1.4
168	209	0.80	2.8
164	203	0.81	5.8
234	294	0.80	0.02
236	292	0.81	0.5
238	296	0.80	1.0
238	295	0.81	2.0
237	295	0.80	4.3

[a] Determined from dielectric measurement, pressure 1.5 kPa.



(a) (b)

Figure 2. Surface (a) and volume (b) resistivity with 100 V test voltage as a function of RH. 160 g/m^2 paper with different amounts of NaCl.

was performed at frequencies from 100 Hz to 1 MHz. The dielectric and electrical properties were measured in five different relative humidities (RH): 25%, 30%, 40%, 50%, and 60% at 23°C, after the papers had been conditioned for at least 8 h in the humidity concerned.

The papers investigated (Table I) were made on a pilot paper machine to three different grammages: 90, 160, and 230 g/m^2. The papers contained 15% precipitated calcium carbonate as filler, an alkyl ketene dimer internal sizing agent (1.5 kg/ton), cationic starch (8 kg/ton), and a two-component retention system containing bentonite and polyacrylamide, 1.7 and 0.2 kg/ton, respectively. The pulp mixture contained 80% hardwood and 20% softwood.

Sodium chloride was dosed on pilot-machine base papers by applying the salt at the size press as an aqueous solution on both sides of the web. Target dosage levels were 0, 0.75, 1.5, 3, and 6 kg/ton with the 160 g/m^2 paper, and the same amounts per unit area on the 90 and 230 g/m^2 base papers. The actual NaCl contents of the trial papers were then determined by ion chromatography of the water-soluble chloride, where the sample pretreatment procedure and determination of water-soluble chloride complied with ISO 9197. The NaCl content [kg/ton] of the 160 g/m^2 paper was in good agreement with the target dosage (Table I). The NaCl content of the 90 and 230 g/m^2 papers was 0–10.6 kg/ton and 0–4.3 kg/ton, respectively. It can be seen in Table I that the 90 and 160 g/m^2 base papers took up similar amounts of NaCl per square meter, but that uptake by the 230 g/m^2 paper was greater, which may be linked to the higher pore volume available with the high grammage.

Samples were laboratory calendered both sides twice at a nip pressure of 50 kN/m and a temperature of 100° C, resulting in a Print-surf roughness value between 3.2 to 5.0 μm (measured in accordance with ISO 8791-4 with 1.0 MPa clamping pressure) and a density between 0.73 and 0.84 g/cm^3.

The moisture isotherms of papers containing different amounts of NaCl were obtained in order to assess 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 papers 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 h, after which the A4-size sheets were weighed on a balance with a resolution of 0.001 g (Mettler PC440). The dry weight of each sheet was determined according to SCAN-P4. The reported moisture content values are average of three samples.

RESULTS AND DISCUSSION

DC Resistivity

Surface and volume resistivities of 160 g/m^2 papers, determined at a voltage of 100 V, are shown in Figure 2 on a logarithmic scale as a function of RH. The resistivity of paper decreased exponentially when the atmospheric relative humidity was increased. Since the moisture content of the paper in the investigated RH range was linearly dependent on RH (Figure 3), the resistivity of the papers is exponentially dependent on the moisture content. The exponential dependence of resistivity on paper moisture content is well known, but different mathematical expressions for this dependence have been proposed.[7,8] The surface and volume resistivity dependence on RH can here be expressed as

$$R = Ae^{-\mathrm{RH}/B}, \quad (1)$$

where A is the resistivity at zero moisture content and B is a factor which reflects the sensitivity of the paper resisitivity to the moisture content or to the RH at which the paper was

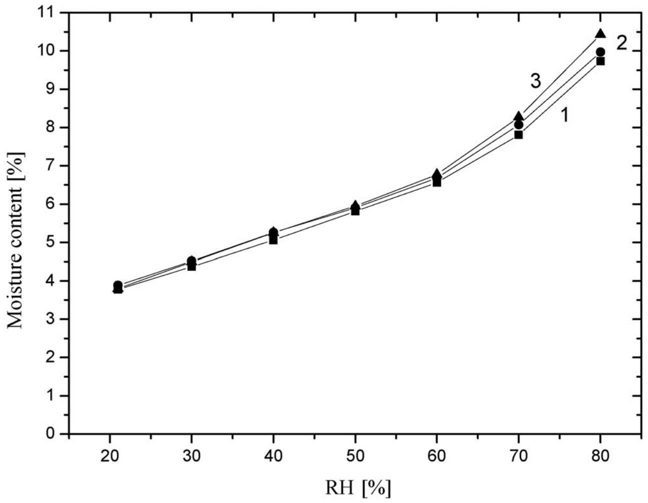


Figure 3. Moisture content dependence on RH and on NaCl addition with 160 g/m^2 papers: 1–no salt added; 2–1.4 kg/ton; 3–5.8 kg/ton.

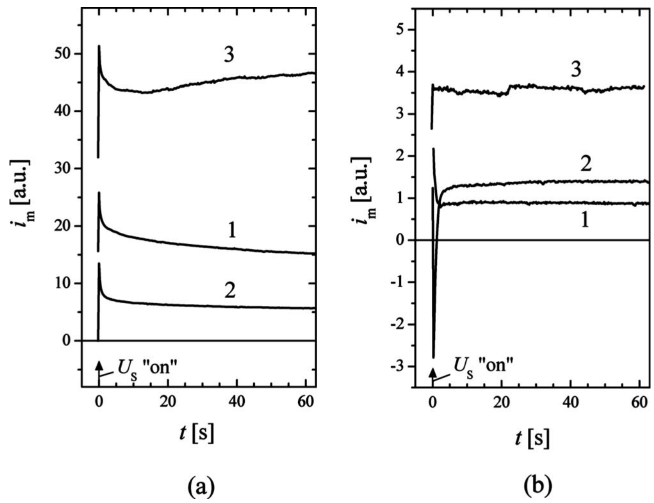


Figure 4. Kinetics of electrical current i_m: a–surface current; b–volume current. 160 g/m^2 papers: 1–no salt; 2–0.67 kg/ton; 3–5.8 kg/ton.

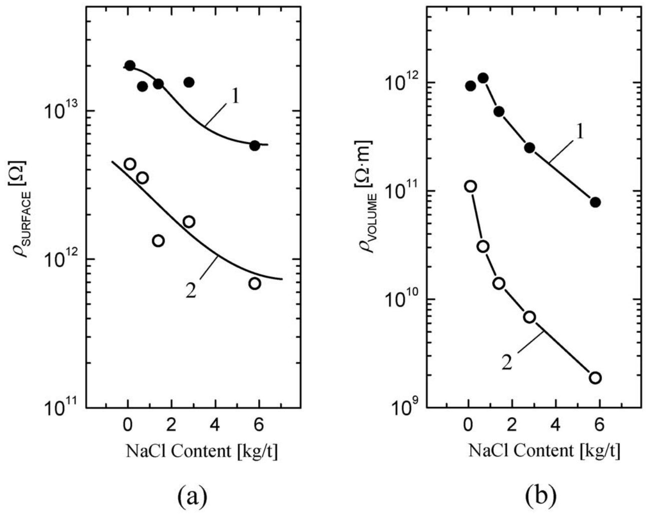


Figure 5. Surface (a) and volume (b) resistivity of 160 g/m^2 papers as a function of NaCl content at 50% RH with test voltages 10 V (1) and 750 V (2).

conditioned. This exponential dependence conforms with the results of Simula and Niskanen.[8] Our results confirm that moisture plays a dominant role in paper conductivity, but the role of the salt content is not clear. Salt addition increases the ability of paper to absorb moisture, and the equilibrium moisture content at a given RH is slightly increased (Fig. 3), but this hygroscopic effect of the salt addition cannot explain the decrease in resistivity when salt is added. Combining data of Figs. 2 and 3, it can be seen that the volume resistivity of 160 g/m^2 paper without salt addition decreases about fivefold when the RH is raised from 40% to 50%, while the moisture content increases by 0.75%. In 160 g/m^2 papers, the difference in moisture content between papers without salt and with the highest salt addition (5.8 kg/ton) at RH 50% was only 0.1%–0.2%, while the volume resistivity was reduced by a factor of about 10 by addition of 5.8 kg/ton NaCl. The hygroscopic effect is responsible only for a small part of resistivity decrease with NaCl addition. The addition of NaCl increases the concentration of mobile ions and this reduces the resistivity of the paper. The minor effect of the increased hygroscopicity suggests that the effect of NaCl on the paper resistivity may also be influenced by other mechanisms. One possible cause is the change in the paper's "electrical structure," the conditions for the formation of space charges in the paper being different in papers with and without salt. The possibility of such effects is seen in the current kinetics shown in Figure 4 after voltage is applied. The nature of such differences is not clear, but addition of salt may not only change the character of formation of space charges but may also affect the paper microstructure responsible for the movement of ions in an electric field. The change in the slope of the dependence of the resistivity on RH (Fig. 2) can be considered as some evidence of this phenomenon. Addition of NaCl decreases the sensitivity of paper surface resistivity to changes in RH. The change in the volume resistivity dependence on RH due to salt addition is not clear, but it must be noted that in this case there are two simultaneous effects: the change in resistivity with RH and changes in the paper structure due to the pressure arising from the electrostatic forces. If this phenomenon takes place, some changes in polarizing behavior should be detected. It was therefore of interest to investigate in detail the influence of salt addition on the dielectric properties.

It is known that DC resistivity depends on the electric field or voltage applied.[1,17] At high voltages (500–1000 V) resistivity decreased as a function of NaCl content, in particular at low dosages, as shown in Figure 5 for papers conditioned at 50% RH. At lower test voltages (10 or 100 V) the effect was smaller, which was also the case at high voltages, when the paper was drier (30% RH); in this case 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 bound to hydroxyl groups for ion conduction and

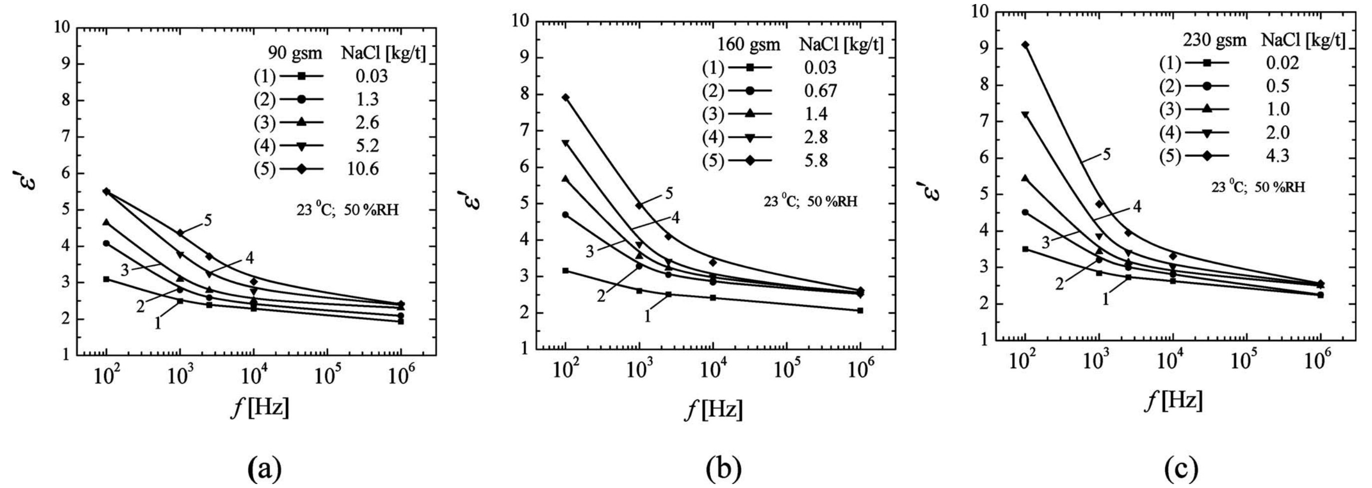


Figure 6. Real part of the dielectric constant ε' of 90 (a), 160 (b) and 230 g/m^2 (c) papers with different NaCl amounts as a function of frequency at 50% RH and 23°C.

the field strength is high, thereby increasing ionic mobility, resistivity becomes very sensitive to the ion content of paper, at low ionic 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 the conductivity in single fibers.[18]

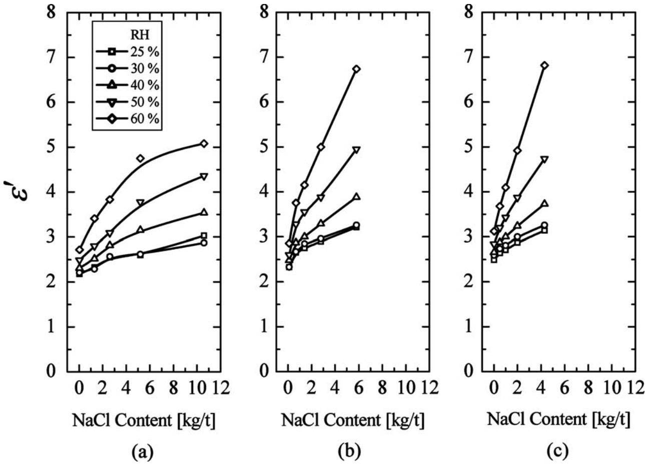


Figure 7. Real part of the dielectric constant ε' of 90 (a), 160 (b), and 230 g/m^2 (c) papers at 1000 Hz frequency and 23° C as a function of NaCl content at different RHs.

Dielectric Properties

Figures 6 and 7 show the real part ε' of the complex dielectric constant (direction across the paper sheet) as a function of frequency and as a function of NaCl content, respectively. As expected, the results indicate a significant influence of frequency on the dielectric properties. This influence was more marked at higher NaCl contents. In the investigated frequency range, there was no flattening of the ε' frequency dependence at low frequencies. As the frequency increased, the real part of the dielectric constant gradually decreased, and no steep drop was observed. This must correspond to a comparatively large-scale polarization phenomenon with a long range of relaxation time. It is usually considered[8,19] that the amount of water, and its state in the paper structure, play a significant role in the polarization phenomenon in paper. Water in paper can be in several states: localized (adsorbed) and firmly bound to the cellulose structure as water molecules which cannot rotate; absorbed but relatively free molecules; and molecules as free as in liquid water. It must also be taken into account that fibers on which water is adsorbed are oriented in different directions with respect to the electric field,[8] and the number of the different water states with respect to the electric field direction therefore becomes significantly larger. These water states have different polarization properties and different relaxation times, which can explain the observed dependence of the real part of the dielectric constant on the frequency.

The real part of the dielectric constant ε' increased markedly with NaCl content. The effect of NaCl was more pronounced at low frequencies, while at 1 MHz, frequency differences were quite small. The real part of the dielectric constant ε' depended on paper grammage, increasing with grammage and consequently with thickness at all frequencies. The influence of NaCl on the real part of dielectric constant ε' was greater in papers with higher grammage (Fig. 7). It is evident in Fig. 7 that the increase in ε' depends not only on NaCl content but also on RH; i.e., on the moisture content. The addition of NaCl led to only a small increase in moisture content (Fig. 3). In 160 g/m^2 paper, the moisture content at 50% RH increased from 5.81% (no salt) to 5.95% (5.8 kg/ton of NaCl). If it is assumed that this additional water is free (dielectric constant is about 80), then the input of additional water due to NaCl addition would increase ε' by about 0.1. The input due to NaCl also would be very small, because the maximum content of NaCl in paper is 1% or less. The increase in the real part of the dielectric constant ε' cannot therefore be explained by simple summation of the dielectric properties of the constituents.

Another possible cause of the change of dielectric properties could be a difference in paper density.[8,13] The density of the papers in our trial was varied within a limited range and was not influenced by the NaCl addition (Table I). The results do not therefore need to be corrected to constant density using the Clausius–Mossotti relation, and the density differences cannot explain the significant increase in the real part of the dielectric constant ε'.

Two possible explanations of the experimental results remain. One possibility is that the addition of NaCl changes the microstructure of paper, resulting in changes in the state of water and in changes 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 (the salt was applied in the 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. The difference in conductivity of the paper surface and bulk should be more significant in thicker papers, and the influence of space charges on dielectric properties should then be more prominent.

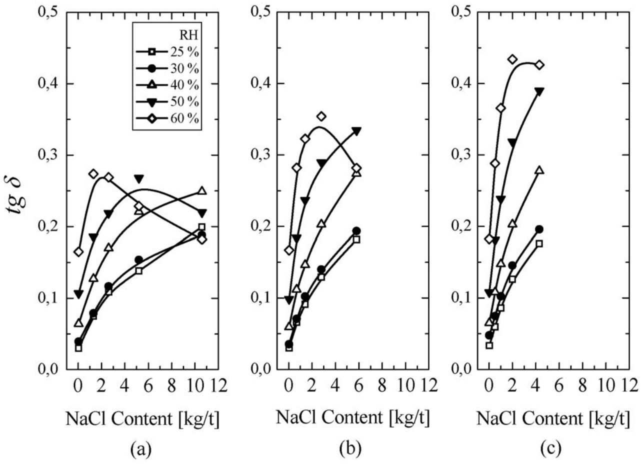


Figure 8. Dielectric loss factor *tgδ* of 90 (a), 160 (b), and 230 g/m^2 (c) papers at 1000 Hz frequency as a function of NaCl content at different RHs.

The addition of NaCl significantly affected dielectric loss (Figures 8 and 9). The dielectric loss factor *tgδ* increased with NaCl content at 30% and 40% RH in all investigated cases. At a low NaCl content (up to approximately 3 kg/ton), the dielectric loss increased at all RHs. At higher NaCl content and higher RH, a decrease in the dielectric loss factor *tgδ* was observed. NaCl increases electrical conductivity, which causes *tgδ* to increase, but the conductivity increase cannot explain the unusual *tgδ* dependence on NaCl content and RH. The frequency dependence of *tgδ* provides additional information. Dielectric loss depends on frequency, and the addition of NaCl changes the dependence of *tgδ* on frequency (Fig. 9). At low NaCl content, *tgδ* gradually decreased with frequency, in agreement with results of Simula and Niskanen[8] and of Morton and Hearle.[19] At higher NaCl concentrations, however, a maximum appeared at a frequency of about 1 kHz. We interpret this result to mean that at higher NaCl contents new microstructures are formed with a relaxation time of the order of 10^{-3} s. If so, then this is additional evidence that salt changes the microstructure, which, in turn, changes the electrical and dielectric properties of the papers described in this article. A similar effect on cotton fibers (pure cellulose) was observed by Morton and Hearle;[19] at 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.

Our assumption that NaCl leads to the formation of new microstructures which influence the electrical properties of paper is supported by the dependence of dielectric loss on RH. As was shown by Simula and Niskanen,[8] dielectric loss increases with RH and this increase is related to the amount of water in the cellulose fibers. Fig. 8 shows that the dielectric loss factor *tgδ* does not always increase with RH: in many cases at high NaCl content, *tgδ* is lower at higher RH. This can mean that the state of at least part of the water is changed in the presence of NaCl. It can also be supposed

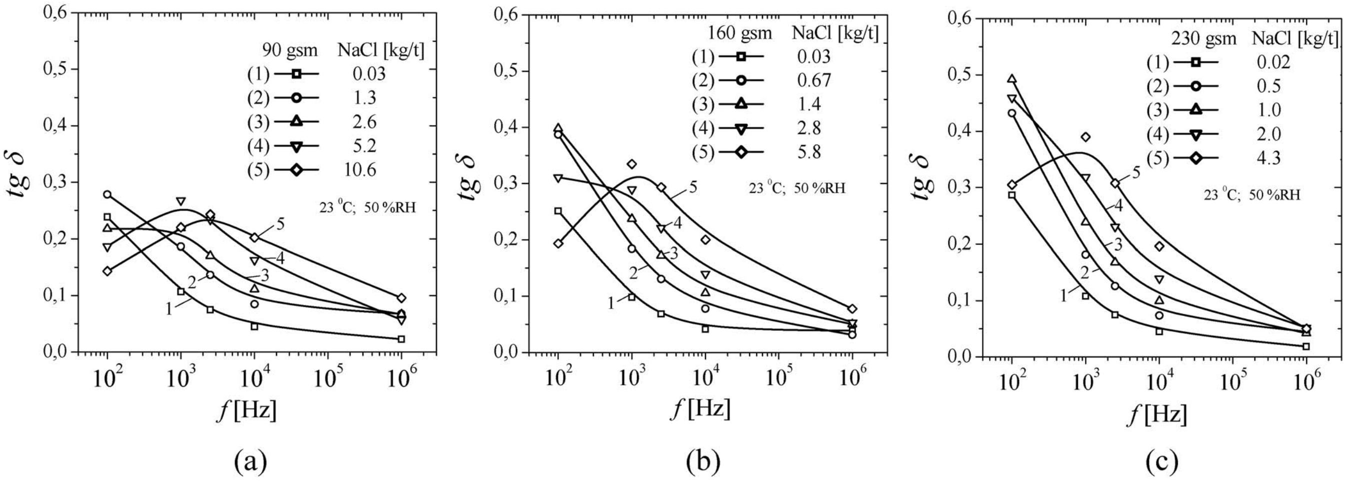


Figure 9. Dielectric loss factor *tgδ* of 90 (a), 160 (b), and 230 g/m^2 (c) papers with different NaCl contents as function of frequency at 50% RH and 23° C.

that NaCl forms new structures with the water in paper—clusters which may have specific dielectric properties and thereby influence the ion movement and paper conductivity.

CONCLUSIONS

The addition of NaCl results in an essential decrease in the surface and volume resistivity of paper, as expected. The rate of decrease of both surface and volume resistivity depends on the salt content, and also on the voltage applied in the resistivity measurement. The dependence of resistivity on relative humidity and consequently on the moisture content of paper is exponential. The addition of NaCl slightly increases the moisture content of paper but this small hygroscopic effect cannot explain the decrease in resistivity. The change in resistivity of paper with changing salt content must be controlled by other mechanisms. The effects are presumably related to the amount of free ions, which, according to current theories, enable ions to pass through the hydroxyl groups that are occupied by water molecules. The time dependence of the conductivity suggests, however, that changes in salt addition also influence the character of formation of space charges. It can also be assumed that salt changes the microstructure of paper, and thus influences its electrical properties. Evidence for this possibility includes change in the dependence of resistivity on RH on salt addition, changes in current kinetics after the voltage is applied, and the character of the changes in dielectric properties with changing NaCl content at different relative humidities.

The real part of dielectric constant ε' decreased with increasing frequency in all the investigated papers without any flattening at low frequencies. This must correspond to a comparatively large-scale polarization phenomenon with a long relaxation time range, and thus it is related to the different states of water in the cellulose fiber network, with respect to the direction of the electric field; ε' increases with increasing salt content and increasing relative humidity. The increase cannot be explained solely by the addition of moisture and NaCl. It is supposed, as in the interpretation of the resistivity data, that space charges can be formed in the papers with salts and that some changes in the microstructure of papers occur, especially related to the state of water. These assumptions are supported by the observed maxima in the frequency dependence of the dielectric loss factor on the NaCl content and on the RH. These maxima are attributed to the formation of new structure with a relaxation time of the order of $10^{-2}-10^{-3}$ s.

Despite the evidence for the formation of new structures in paper and the assumed effects of space charges, both influencing the resistivity and dielectric properties of paper when moisture and NaCl are added, these hypotheses require more experimental data. A study of the dependence of the electrical and dielectric properties on temperature may provide such additional information.

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