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The Physical Basis of Dielectric Moisture Sensing

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Abstract: Moisture content sensors for hygroscopic solids below saturation most commonly detect low frequency (<10 kHz) conductivity, or permittivity at microwave frequencies (0.1 – 10 GHz), with calibration being done empirically. Two physical processes are responsible for the moisture-dependent admittivity in these frequency ranges. At low frequencies ionic hopping between the absorbed water molecules gives rise to the “logarithmic” dependence of conductivity on moisture content that is a generic characteristic of hygroscopic solids. At higher frequencies the admittivity is dominated by the dipole response of the absorbed water molecules. *Copyright © 2008 IFSA.*

Keywords: Moisture content, Proton hopping, Dipole rotation

1. Introduction

Due to its inherent simplicity and versatility, moisture sensing based on dielectric measurement is commonly employed [2-4]. However despite its widespread exploitation, the origin of the dielectric response and of its dependence on moisture content are not well understood. It is axiomatic that improved understanding of the underlying physics of the interaction between water-absorbing materials and applied electric fields will facilitate improvements in sensor designs.

The accumulation of experimental data over many decades [5] shows that the electrical properties of hygroscopic solids are determined primarily by the concentration of absorbed water. In practice moisture content sensing is most commonly attempted by measuring dielectric properties in the frequency range 0 to ~10 GHz. At low frequencies the dielectric parameters have a “logarithmic” dependence on moisture content (M, % dry basis) and exhibit a constant phase angle response, i.e. dielectric loss and permittivity have an identical frequency dependence of the form $\omega^{-\beta}$, where ω is

the radial frequency and β is a constant. The magnitude of the specific admittance (admittivity) decreases linearly in logarithmic representation to a change of slope at intermediate frequencies ($\sim 10^{-1} - 10^5$ Hz). The characteristics of the intermediate frequency response vary between different materials and the nature of the underlying process is unclear. At higher frequencies classical dipole dispersion is observed with a relaxation frequency $\sim 10^9$ Hz and magnitude linearly dependent on M .

Two variables commonly determine the minimum signal frequency that can be used: the first is the time constant of the process to be controlled, which in the case of industrial drying for example can be on the order of minutes, allowing frequencies as low as 0.01 Hz to be used. The second and usually controlling variable is the maximum rate of change of moisture content in the target material. Since the relationship between low frequency dielectric data and moisture content is non-linear, the minimum measurement frequency should be between 1 and 2 orders of magnitude higher than the maximum frequency of moisture content variation. However experimental measurement of dielectric properties should be made over as wide range as possible as an aid to correct interpretation of the data; this is the approach that has been taken here.

In the following sections we present experimental data illustrating the generic characteristics of hygroscopic solids, and then develop an electrical model for the entire frequency range over which the presence of absorbed water can significantly affect their properties.

2. Experimental

The 600 Hz relative conductance of commercial samples of rubber coir, chocolate crumb and gelatine were measured at room temperature using the Streat Instruments Ltd Drycom Moisture Meter. Measurements on a single sheet of chemically cleaned cellophane were performed in a vacuum chamber in which water vapour pressure could be varied and temperature controlled to $\pm 0.1^\circ\text{C}$. The sample was mounted in a guarded cell with aluminium foil electrodes. A second sample mounted on a Mettler Toledo SAG204 weighing balance in the chamber served to verify stability and to determine M . The dry weight was obtained by placing anhydrous P_2O_5 in the chamber. The voltage source was an Agilent 33120A Function Generator. Voltage and current were detected with a purpose-built amplifier. Frequencies from 1×10^{-3} to 3×10^1 Hz were measured digitally, while from 3×10^0 to 3×10^5 Hz amplitude was measured with an HP 34401A multimeter and phase angle with an analogue circuit.

3. Results and Discussion

3.1. Low Frequency Region - Moisture Content Dependence

Fig. 1 shows typical conductance versus moisture content data measured with the Drycom moisture meter. Similar data were observed for many other industrial products, including for example bread crumbs, olive pomace, wool, cotton, viscose, nylon and possum fur. Fig. 2 shows typical data taken from the literature.

The implication of these data and of many others that can be found in the literature is that "logarithmic" dependence of low frequency conductivity on M is a universal characteristic of hygroscopic solids.

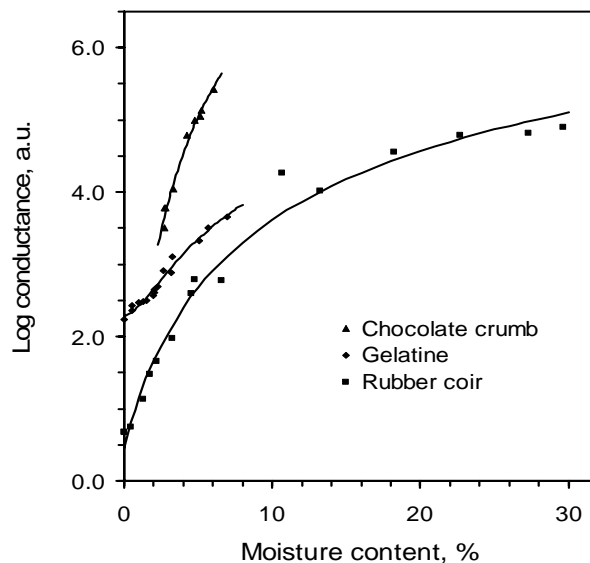


Fig. 1. The room temperature conductance of rubber coir, gelatine and chocolate crumb at 600 Hz, a.u. = arbitrary units. The latter two data sets have been offset by +1 for clarity.

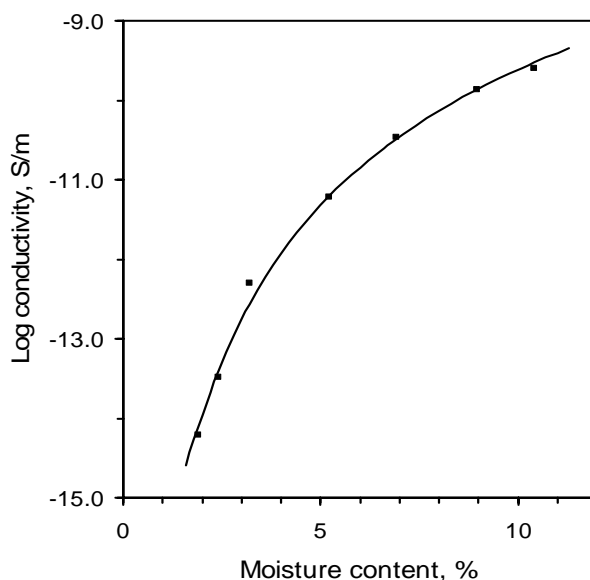


Fig. 2. The low frequency conductivity of nylon at 25°C [6]. Similar data were obtained by Hearle [7] for a large number of textile fibres.

As a step towards theoretical interpretation many investigators have attempted to identify the charge carrier [8-15]. Whereas the evidence in favour of electrons is weak, conduction by impurity ions has been demonstrated directly [16-18] and the evidence for protonic conduction is strong [14, 15, 19, 20].

Fig. 3 shows the conductivity of two samples of deuterated lysozyme measured in a high ($>10^6$ V/m) electric field [18]. One sample contained mobile impurity ions and in the other these ions had been removed electrically (“Uncleaned” and “Cleaned” respectively). Conductivity was calculated from the electrical measurements and from the quantities of deuterium gas collected (“Deuterium”), the latter being found to be independent of the impurity ion concentration.

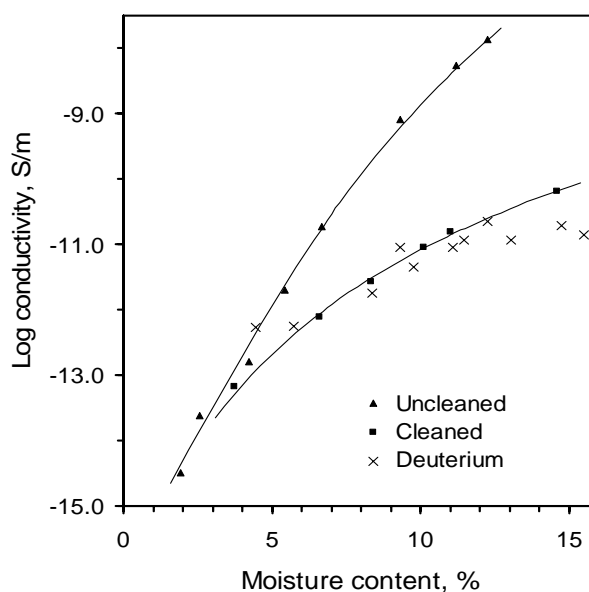


Fig. 3. The low frequency, high field conductivity of lysozyme at 294 K [18].

Protonic conduction was found also to be dominant in uncleaned samples with electric fields less than 8×10^5 V/m. We conclude that both protonic and impurity ionic conduction can occur in hygroscopic solids that both are dependent on water concentration and that in low electric fields protonic conduction are the dominant charge transport process.

By analogy with liquid water, we have proposed that dominant conduction process in hygroscopic solids is proton hopping between absorbed water molecules [21]. In a disordered distribution of the hopping sites, the macroscopic conductivity σ is given by

$$\sigma \propto e^{-\frac{2R_C}{\alpha}}, \quad (1)$$

where α is the decay length of the charge carrier wave function and R_C is the critical distance for percolation. We have found in numerical calculation [21] that $R_C \approx 0.87R_{AV}$ with a weak dependence on M , where R_{AV} is the average distance between water molecules. Writing $R_C = a_C R_{AV}$ we get:

$$\sigma \propto e^{-\frac{2a_C}{\alpha} \left(\frac{1.8}{\rho N_A M} \right)^{1/3}}. \quad (2)$$

Here ρ is the density of the dry absorbent and N_A is Avogadro's number. We allow for the existence of a concentration N_0 of non-aqueous hopping sites and for non-hopping and non-aqueous conduction by writing

$$\sigma = \sigma_L + \sigma'_0(\omega, T) e^{-\frac{2a_C}{\alpha} \left(\frac{1.8}{\rho N_A (M+M_0)} \right)^{1/3}}. \quad (3)$$

$M_0 = 1.8N_0/\rho_0N_A$ is the equivalent water concentration contributed by hopping sites in the dry absorbent and represents low level non-aqueous protonic conductivity such as that attributed to conduction via

hydroxyl groups in cyclodextrin [20]. Possible origins for conductivity σ_L include leakage current in the measurement apparatus and other weak conduction processes in the sample. $\sigma'_0(\omega, T)$ is the proportionality factor (T = temperature).

The results of fitting equation (3) to experimental data are shown in Figs. 1-3 (solids lines) and support our contention that the characteristic shape of conductivity – moisture content curves in hygroscopic solids has the mathematical form $e^{-AM^{-1/3}}$ (where A is a constant). In Fig. 3 the “Uncleaned” curve is the sum of the “Cleaned” protonic conductivity and a fitted ionic conductivity, the result suggesting that non-protonic ionic conduction too may be a hopping process.

Further insight into the influence of impurity ions is obtained from Fig. 4 showing equation (3) fitted to conductance data for two samples of cotton thread with different concentrations of ionic impurities [5]. The Fig. 4 fitting constants are identical except for the $\sigma'_0(\omega, T)$ which differ by a factor of 76. Since both samples have the same α and the electric field strength was small ($<8 \times 10^3$ V/m) we deduce that conduction was protonic only in both samples. Writing the conductivity as $\sigma = zeN\mu$ where ze , N and μ are the electric charge, concentration and mobility of the charge carriers, we identify μ with the exponential term in equation (3) and zeN with $\sigma'_0(\omega, T)$. The difference between the two curves arises therefore from a difference in the concentration of mobile protons. The ash content difference is typical of the difference between raw and washed cotton [22]. The data therefore suggest that the ionic salts catalyze the production of mobile protons in the absorbent and also that the charge carrier concentration is independent of M . This conclusion was also reached by Lederer *et al.* for bovine serum albumin (BSA) [11] and by Pethig in a review of conduction in biological polymers [23].

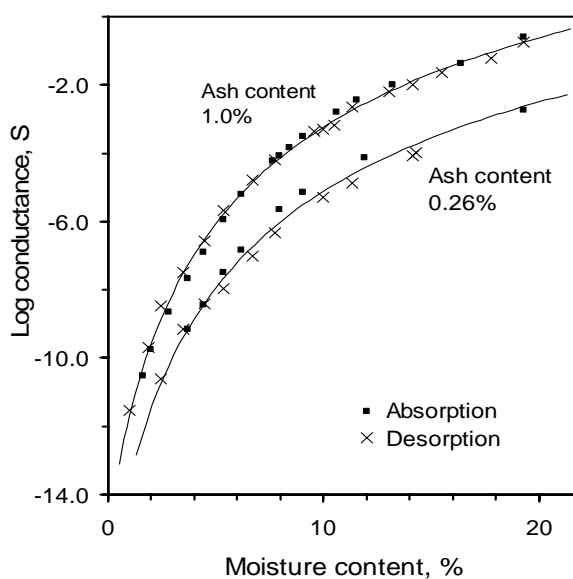


Fig. 4. The low frequency conductance of cotton thread at 25°C [5].

We can therefore rewrite equation (3) as

$$\sigma - \sigma_L = zeN\mu'_0(\omega, T)e^{-\frac{2a_c}{\alpha} \left(\frac{1.8}{\rho N_A (M + M_0)} \right)^{1/3}}, \quad (4)$$

An implication of the model is that the dependence of conductance on moisture content will change at saturation, i.e. the equilibrium moisture content at 100% relative humidity, M_{SAT} . Above M_{SAT} any

further increases in M occur by condensation on external surfaces. R_C therefore remains constant, and the shape of the theoretical curve will change. Fig. 5 shows equation (3) fitted to data for viscose ($33\% \leq M_{SAT} \leq 45\%$ [24]). A change from a “logarithmic” to approximately linear characteristic occurs at $M \approx 35\%$.

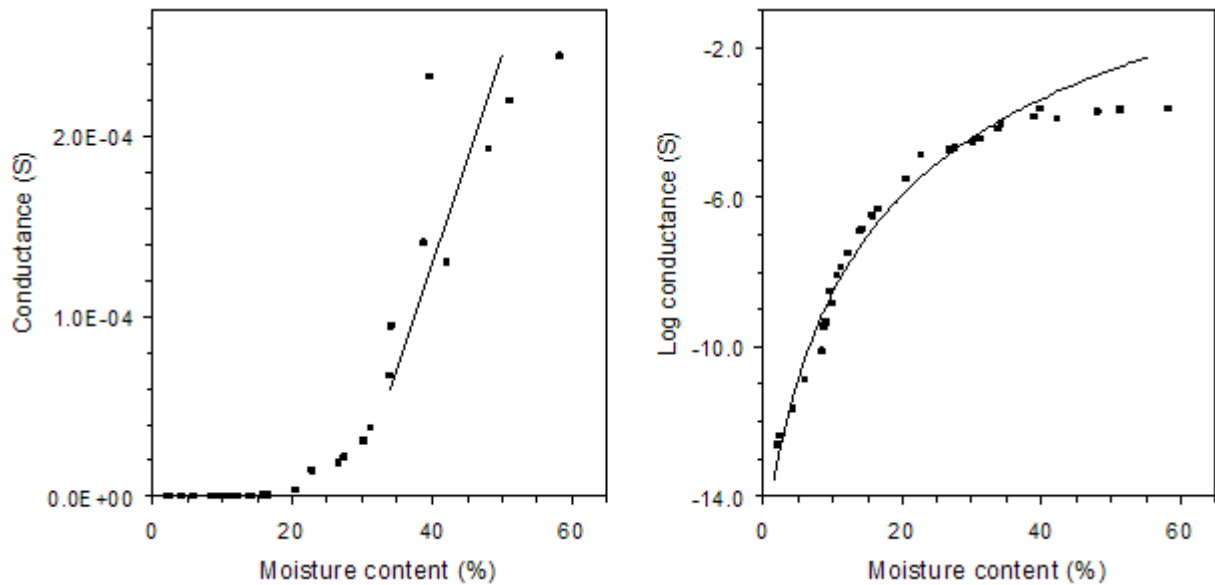


Fig. 5. The low frequency conductance of viscose at 20°C [7] plotted in log and linear representation.

3.2. Low Frequency Region - Frequency Dependence

Figs. 6 and 7 show the dielectric spectra of cellophane from $10^{-3} - 10^5$ Hz for three different M .

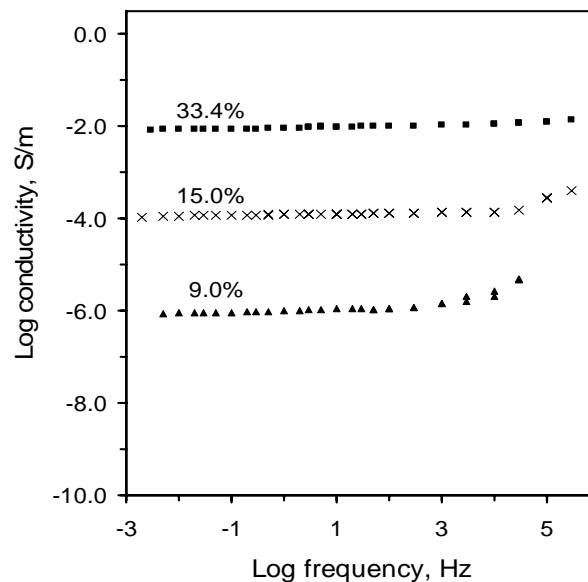


Fig. 6. The conductivity of cellophane at 30.9°C for $M = 9.0\%$, 15.0% and 33.4% .

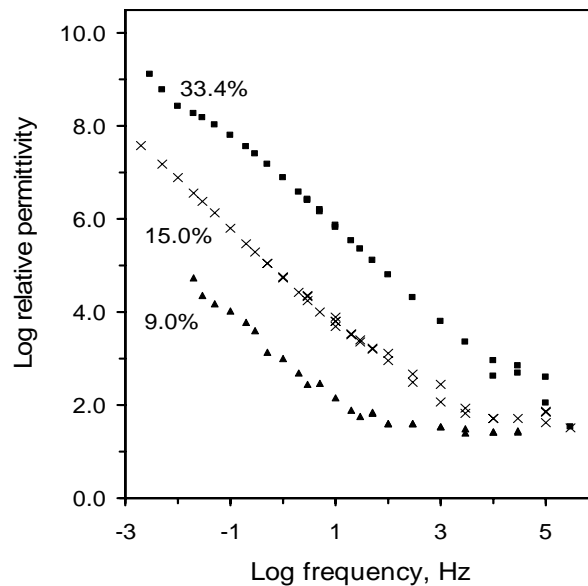


Fig. 7. The permittivity of cellophane at 30.9°C for $M = 9.0\%$, 15.0% and 33.4% .

Similar characteristics have been observed in many hygroscopic solids [25-31].

Starting with the same proton hopping model we have obtained for the low frequency admittivity γ_{LF} [21].

$$\gamma_{LF}(\omega) = \sigma_{DC}(T, M, N) + \gamma_{AC}(T, M, N)\Gamma(\beta)(j\omega)^{-(\beta-1)}, \quad (5)$$

where Γ is the gamma function and $j = \sqrt{-1}$. σ_{DC} is the dc conductivity at T , M and N , γ_{AC} the proportionality factor and β ($0 < \beta \leq 1$) represents the degree of disorder in the charge carrier energy levels and in the hopping site distribution, with $\beta = 1$ corresponding to no disorder. In the time domain equation (5) describes a system in which a step voltage produces a current decay proportional to $t^{\beta-1}$ ($t = \text{time}$). Introducing the dependence on M and N from equation (4) we get

$$\gamma_{LF}(\omega) = zeNe^{-\frac{2a_C}{\alpha} \left(\frac{1.8}{\rho N_A (M+M_0)} \right)^{1/3}} \left[\mu_0(T) + \mu_{AC}(T)\Gamma(\beta)(j\omega)^{-(\beta-1)} \right], \quad (6)$$

where μ_0 and μ_{AC} are T -dependent mobility factors. Equation (6) predicts that γ_{LF} will have a constant phase angle of $(1-\beta)\pi/2$.

3.3. Intermediate Frequency Region

Literature data show a wide variety of relaxations in the frequency range $\sim 10^{-1} - 10^5$ [20, 26, 28, 30-34].

It is likely that these features arise from polarizations associated with the substructures of experimental samples, which are commonly in the form of compressed powders, grains or fibrous sheets. Hygroscopic polymers typically contain mixtures of amorphous regions accessible to water molecules and crystalline regions which are not [35], which could be expected to give rise to interfacial

polarisations. We interpret the change of slope at the high frequency end of the cellophane data as arising from this cause.

The charge carrier transit time in sub-structure units is inversely proportional to the mobility which for constant N is proportional to the admittivity. From equation (5) and anticipating $\sigma_{DC} \approx 0$, $\beta \approx 1$, we see that the transit time is approximately proportional to $1/\gamma_{AC}(M)$ so relaxation frequency $\omega_0 \propto \gamma_{AC}(M)$. The loss peak amplitude is proportional to $\gamma_{AC}(M)\omega_0^{-(\beta-1)}/\omega_0$ (equation (5)) which is a constant. Such behaviour has been observed experimentally in BSA [33] and lysozyme [34], supporting our interpretation of intermediate frequency relaxations. A similar conclusion has been reached regarding intermediate frequency dispersion in ovalbumin [36].

To represent the contribution of the intermediate frequency relaxation to total admittance we add a general function γ_{IF} which could represent, for example, the Havriliak-Negami function [37] which was found to fit data for BSA [33] or the Davidson-Cole model which fitted ovalbumin data [36].

3.4. High Frequency Region

A classical Debye dipolar dispersion detected in lysozyme at 0.2 GHz and $M = 34\%$ by Harvey *et al.* [38], and a similar response has been obtained in BSA, with magnitude proportional to the amount of primary absorbed water [39]. A dipolar response at frequencies below that for bulk water would be expected in view of the stronger bonding experienced by absorbed water. We represent the high frequency response with a Debye function, since this was found to accurately fit experimental data for lysozyme [38]:

$$\gamma_{HF} = j\omega \left(\varepsilon_{\infty} + \rho M / 100 \frac{\varepsilon_D}{1 + j\omega\tau_D} \right), \quad (7)$$

Here ε_D is the magnitude of the dipole relaxation per kg of absorbed water, τ_D is its relaxation time, ρ is the density of the dry absorbent and ε_{∞} is the high frequency limiting value of the permittivity.

3.5. Application of the Model to Cellophane

The equation

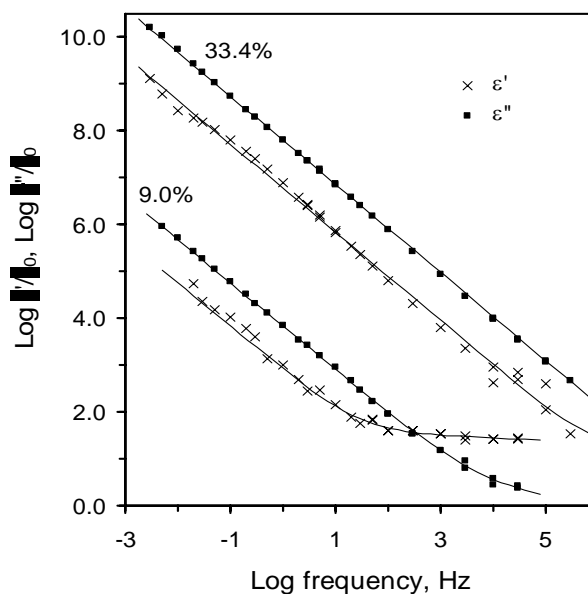
$$\gamma = \gamma_{LF} + \gamma_{IF} + \gamma_{HF}, \quad (8)$$

has been fitted to the cellophane data shown in Figs. 6 and 7. The data for the intermediate frequency response is too limited for any meaningful analysis so simple functions of the form $\omega^{-(n-1)}$ were used for the real and imaginary parts of γ_{IF} . We used $\varepsilon_{\infty}/\varepsilon_0 = 3$, $\rho\varepsilon_D/100\varepsilon_0 = 15/34$ (values for lysozyme [38] assumed, ε_0 = the permittivity of free space) and $\omega\tau_D = 0$ for γ_{HF} . The dc conductivity was treated as a fitting parameter. The results are shown in Fig. 8 for $M = 9.0\%$ and 33.4% , and in Table 1.

Since the gradients for both the ε' and ε'' lines at low frequencies and the magnitude difference (in logarithmic representation) are all determined by a single value of β , the fits obtained give good support to the model represented by equation (6).

Table 1. Values of σ_{DC} and β for cellophane at 30.9°C for $M = 9.0\%$, 15.0% and 33.4%.

	Moisture content, M , %		
	9.0	15.0	33.4
σ_{DC} , S/m	6.02E-07	7.53E-05	5.71E-03
β	0.92	0.95	0.94

**Fig. 8.** Equation (8) fitted to the relative permittivity and dielectric loss of cellophane at 30.9°C for $M = 9.0\%$ and 33.4%.

4. Conclusions

The universality observed in the low and intermediate frequency dielectric response of hygroscopic solids suggests the existence of a single underlying cause. A model of proton hopping within a disordered distribution of absorbed water molecules can explain the moisture content and frequency dependence of the admittivity in both the low frequency and intermediate frequency regions. The model also predicts that low frequency conductivity and permittivity will have the same water concentration dependence, a prediction supported by the data. Additional low frequency charge transport by impurity ions can occur provided a sufficiently high electric field is present.

The variable responses seen in the range $\sim 10^{-1} - 10^2$ Hz arise from interfacial polarization in sample sub-structures. At frequencies on the order of 1 GHz the response of the water molecule dipole becomes the dominating process.

The entire range from dc to the gigahertz frequencies could be exploited for moisture measurement purposes. The low frequency region has greatest sensitivity and the advantage that the dielectric response is directly dependent on M . Disadvantages include the slow measurement speed required, the weakness of the signal and sensitivity to uneven moisture distributions. Intermediate frequency signals also depend directly on M and are stronger, but are affected by variations in material structures which could reduce calibration accuracy. Signals are strongest in the high frequency region but are proportional to the total mass of water rather than to moisture content *per se*, and knowledge of total mass or density is therefore required as well.

Acknowledgements

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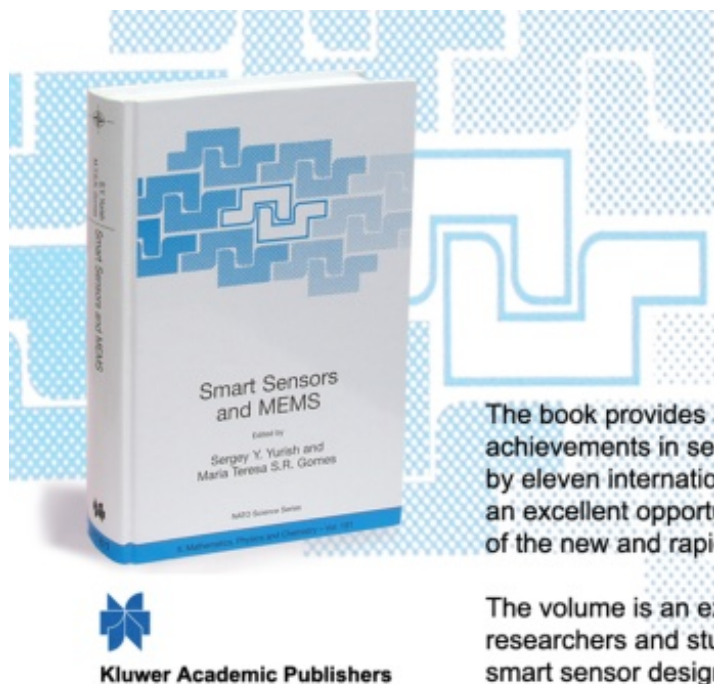
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