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Water activity and dielectric properties of gels in the frequency range 200 MHz–6 GHz

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Abstract

Dielectric spectroscopy gives information on a molecule's chemical relations with its surroundings while water activity (a_w) is the thermodynamic measure linked with water bonding to the food matrix. Thus, it should be possible to access information on a_w via dielectric measurements, which could be very interesting, in practice, regarding easiness of dielectric measurements. Forty-five model samples of animal gelatin gel of various water and NaCl contents (a_w from 0.91 to 1) were characterised in terms of a_w and dielectric properties. To analyse the variations of water activity with water and salt contents, over a larger moisture content range, isothermal sorption curves were determined. This investigation provides correlations between a_w and some dielectric properties, such as relaxation frequency. Results of this first investigation are encouraging but lead to some questions about influence of NaCl on dielectric measurements, the possibility of associating dielectric measurements with sorption isotherms and problems concerning measurements on real food products.

Keywords: Water activity; Sorption isotherms; Animal gelatine; Dielectric properties; Microwave; Coaxial line probe

1. Introduction

Food safety, quality and stability are major challenges for the food industry. One of the food properties that has to be controlled is water activity (a_w). The usual measurement techniques require bringing the material to a state of equilibrium with its surroundings, which takes a few hours (Timmermann & Chirife, 1991). Other experimental techniques, such as nuclear magnetic resonance (Chaland, Mariette, Marchal, & De Certaines, 2000; Traoré, Foucat and Renou, 2000) can also be used to investigate water activity, but not in situ.

Gabriel, Lau, and Gabriel (1996) and Kent and Jason (1974) show that dielectric properties are closely correlated with water content and state in a material. It has long been recognised (Smith, Duffy, Shen, & Olliff, 1995; Suzuki, Shigematsu, & Kodama, 1996) that water activity in food products depends on both bound and free water. Hence the water activity of a product could be derived from its dielectric properties (Grant, 1966; Kaatze, 1997). In particular, dielectric relaxation spectroscopy determines the response of molecular motion of polar molecules in the sample to a weak external alternating electric field. As the electric field frequency is increased, it reaches a frequency called the relaxation frequency (f_R) when the polar molecule can no longer rotate with the electric field. Dielectric properties change markedly around this relaxation frequency.

Dielectric properties not only depend on water binding in food material but also on food composition. For a given molecular composition only, the dielectric spectrum will change with molecular binding. In real material, complexity of molecular composition, presence of ions, electrical charges on proteins and pH variations lead to a complex dielectric spectrum with the contribution of several phenomena. Unfortunately, from the present knowledge, neither water activity nor dielectric properties can be theoretically predicted.

Foods have a complex composition on which water activity depends. On the other hand, dielectric parameters are also linked with food composition. So, a microwave sensor could be a good tool for fast a_w measurements.

The purpose of this work was to access the feasibility of a microwave sensor for a_w measurement. Since the

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measurement is very fast, this type of sensor could be used in-line.

2. Materials and methods

2.1. Materials

All the measurements were carried out on gelatine gels. The gelatin gel is an isotropic homogeneous material whose pH, moisture and NaCl contents can be accurately controlled. It is a good model of foods of animal origin because of its complex protein composition and behaviour when undergoing drying. Baucour (2000) has shown that the water sorption isotherm of pork lean meat is almost the same as that of gelatine samples. Moreover, its natural pH is about 5.2±0.2 in comparison with pH of meat, which reaches a value between 5 and 6 after rigor. It also allows a water content from 50 to about 90%. Meat and dehydrated or fermented meat products range from 33 to 75% in water content.

The samples were made up with a commercial animal gelatine powder (PS 60 Bloom, from SKW Biosystems SAS, Angoulême, France), de-ionised water and NaCl (99.5%, from ACROS) to depress a_w. All the components were mixed at 40 °C, heated from 40 to 80 °C and maintained at this temperature for one hour. The samples were then poured into tubes and left to cool at 18 °C for 1 day. Precautions were taken to avoid dehydration of samples.

Dielectric measurements, with a coaxial line reflecting probe, need to be performed on samples of reasonable size (at least a disk, 12 mm in diameter and 10 mm in thickness) within which water and salt are evenly distributed. This can only be achieved with native samples, while a gelatine gel having water content below 1 kg per kg of dry matter cannot be made up due to the high viscosity of the preparation before gelification. Thus, to extend the area of investigation and to have continuous relationships between water activity and gel composition, water sorption curves were determined on samples dehydrated in parallel to dielectric measurements (Table 1).

Forty-five native samples were prepared. Their water contents ranged from 0.9 to 10 kg per kg of dry matter (50–90%) and their salt contents ranged from 0 to 0.12 kg of NaCl per kg of dry matter. These ranges of water and salt contents also correspond to relevant water activity (0.908 to 1) (Table 2).

Five other samples were prepared for water sorption isotherm measurements, with initial water contents of 3 kg water/kg dry. NaCl contents were 0, 0.02, 0.04, 0.06 and 0.08 kg salt/kg dry matter, respectively. Each sample was cut into 10 slices of 2 mm in thickness.

2.2. Methods

2.2.1. Water activity measurements

The measurements were performed with a thermostatted fully automatic computer-controlled device, the aw Sprint from Novasina, Switzerland. It consisted of a fluid-tight low volume cell in which the sample was placed, and a humidity sensor. If the sample is left in the cell long enough, about three hours, it reaches equilibrium with the air in the cell. The humidity sensor measures water content in air, which gives water activity of the sample. It operates from 0 to 50 °C and can measure water activities from 0.06 to 1. Preliminary calibration was done with saturated salt solutions.

2.2.2. Sorption isotherm measurements

Isotherms were measured using an INRA-made device for fast measurement of water sorption isotherms in the high humidity range (Baucour, 2000; Baucour & Daudin, 2000).

The apparatus has two parts (Fig. 1): (1) a saturation column, which produces saturated air, and (2) a train of 10 cells where the samples are placed. Successive pressure drops from cell to cell allow to cover a desired range of air relative humidity.

(1) The saturation column (0.5×0.5×1 m) is insulated and filled with water to a height of 90 cm. A cryothermostat (LAUDA) controls the water temperature (T_{W1}) via a heat exchanger. Ambient air is injected at the bottom of the column through 10 porous tubular elements made of ceramic (10 mm in diameter and 300 mm in length, RENA Company). As the air bubbles rise, the relative humidity levels off to 100%. The temperature of the water and the total pressure set the dewpoint temperature of the outlet air (T_D).

Table 1
Water activity versus gelatine gel composition: two kinds of measurements

	Water activity measurements	Water sorption isotherms
Samples	45 not dehydrated (native) samples	5 series of 10 samples. Each series is obtained from a native sample ($X_{eau}=3$), and contains 10 final samples with different water contents
Device	NOVASINA	INRA Made
Water content range	1–10 (50–90%)	0.5–3 (33–75%)
Advantages	Sample size allows dielectric coaxial measurements	Continuous relationship on a wide range of water activity
Drawbacks	Limited low moisture	Final samples size not compatible with coaxial dielectric measurements. Need of cavity measurements for instance

Table 2
Salt content and water content of native samples and their water activities

X_{salt} (kg salt/kg DM)	X_{water} (kg water/kg DM)	a_w	X_{salt} (kg salt/kg DM)	X_{water} (kg water/kg DM)	a_w	X_{salt} (kg salt/kg DM)	X_{water} (kg water/kg DM)	a_w
0.12	0.94	0.908	0.08	3.10	0.977	0	2.82	0.991
0.08	0.97	0.937	0.04	2.27	0.978	0.04	3.10	0.991
0.08	1.34	0.942	0.06	3.22	0.98	0	3.43	0.992
0.08	1.61	0.955	0.04	2.76	0.981	0	2.21	0.995
0.04	1.06	0.958	0	1.09	0.981	0.04	4.23	0.996
0.12	2.00	0.96	0	1.72	0.983	0	4.12	0.996
0.06	1.66	0.963	0.02	2.26	0.983	0	4.92	0.997
0.04	1.63	0.967	0.04	2.09	0.983	0	5.93	0.997
0.08	2.10	0.967	0.08	2.91	0.983	0	3.17	0.998
0.08	2.32	0.971	0.02	2.72	0.984	0	6.65	0.998
0.08	1.99	0.972	0	2.26	0.985	0	4.26	0.999
0.06	2.18	0.973	0.12	3.80	0.985	0	7.78	0.999
0.12	2.91	0.976	0.02	3.11	0.986	0	8.68	1
0.02	1.69	0.977	0.04	3.39	0.988	0	9.95	1
0.06	2.99	0.977	0.08	3.91	0.989	0	10.74	1

(2) Downstream from the column, 10 cells are placed in series and connected by copper pipes fitted with diaphragms. Each cell contains a thin slice of the sample (approximate size 25×10×2 mm) placed in the flow, longitudinally, to facilitate the water transfer. The whole system is placed in a thermostatted water bath at the desired temperature (T_{W2}) for the sorption isotherm. The temperature is measured in each cell (T_i) by a type K thermocouple. Between successive cells, specially designed diaphragms cause pressure drops (ΔP). The air relative humidity in each cell is controlled from pressure measurements in each cell, at the top of the column and at the reference point where a dewpoint hygrometer (DEW 10 General Eastern) is placed.

A PC computer controls the whole system. It has been shown that the time of equilibrium between air and samples is less than 48 h. When equilibrium is reached the water content of each sample is determined by oven-drying at 106 °C for 24 h.

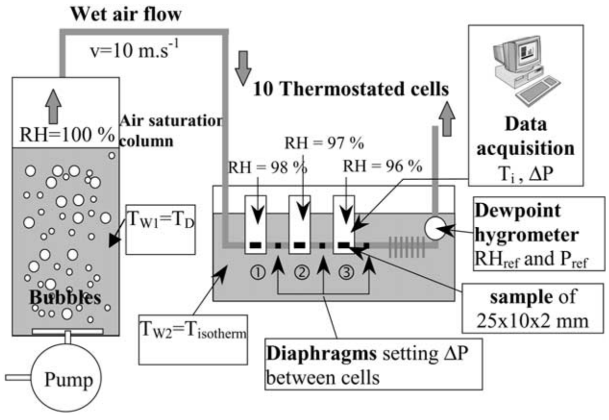

Fig. 1. General diagram of the experimental device for water sorption isotherm measurements.

To cover the range of a_w between 0.85 and 1 the pressure drop between two successive cells was set to about 1000 Pa from cell 1 to cell 5 and gradually increased to about 2500 Pa up to cell 10.

2.2.3. Dielectric measurements

Stuchly, Brady, Stuchly, and Gajda (1982) described the coaxial line reflection method used for dielectric measurements. The experimental system (Fig. 2) consisted of an open-ended coaxial line probe HP 87070 and an HP 8753 automatic network analyser (Hewlett Packard, CA, USA) equipped with the HP85047A S-parameter test set. All the instrumentation was computer-controlled via a GPIB (General Purpose Interface Bus).

The coaxial line reflecting probe consisted of a coaxial line with a flat end for contact between the sample and the probe. The complex permittivity ε* was calculated from the complex reflection coefficient Γ* as described below:

$$\varepsilon^* = \frac{1-\Gamma^*}{2\pi f C_0 Z_0(1-\Gamma^*)} - \frac{C_f}{C_0} \qquad (1)$$

where f is the frequency, C_0 the intrinsic capacitance of the probe, C_f the capacitance due to the fringing field in the material under test and Z_0 the characteristic impedance of the coaxial line.

This method is very useful and fast (a few seconds per measurement) but presents some difficulties: presence of air if contact between sample and probe is not carefully controlled, variations in pressure and problems due to poor homogeneity in samples. Accuracy for permittivity measurement is about 5%.

Dielectric spectroscopy was performed from 200 MHz to 6 GHz. Electrode polarisation effects (Kuang & Nelson, 1998), which can seriously perturb the measurements

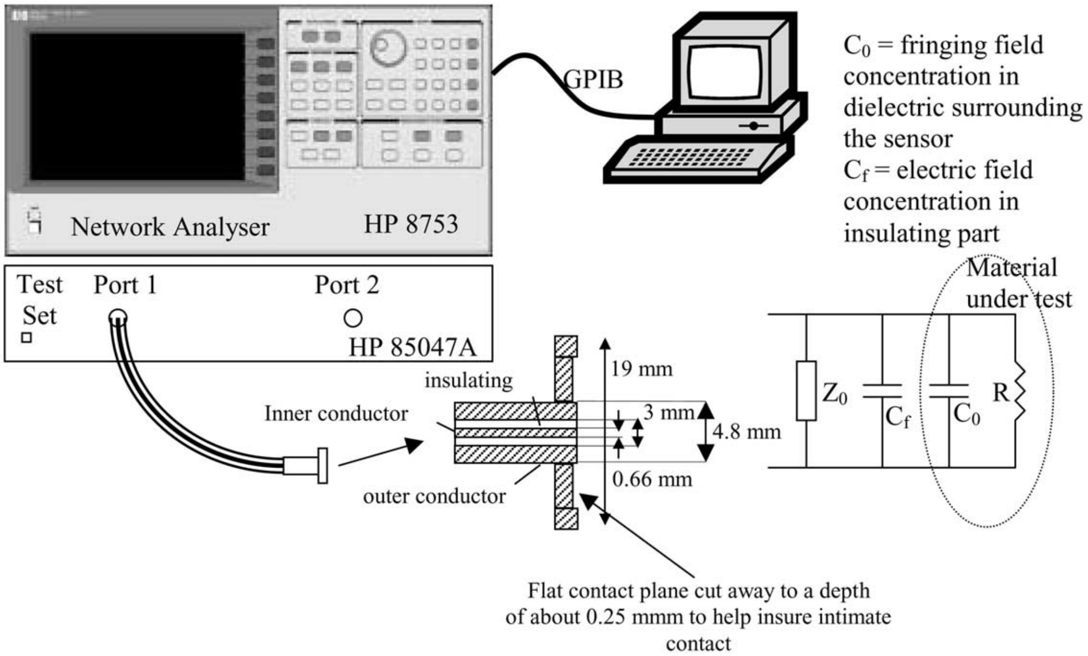

Fig. 2. Dielectric measurement experimental device.

below 100 kHz, were not significant in this frequency range.

Before sample measurements, an error correction was performed using standards as reference terminations. Water activity and dielectric measurements were both made at 18 °C.

3. Theoretical background for data analysis

When an electrical dipole, e.g. a polar molecule, is placed in an alternating electric field, it rotates to follow the electric field. The commonest polar molecule in food products is water.

As the electric field frequency (*f*) increases, it reaches a value at which the molecule can no longer follow the alternating field. This frequency is called the relaxation frequency (f_R) of the molecule and corresponds to large variations in dielectric properties (Fig. 3). The real and imaginary parts of permittivity change markedly with frequency, particularly around the relaxation frequency. There is a decrease in the real part, which corresponds to a decrease in energy storage (if the dipole can no longer rotate, it can no longer release energy), and a maximum in the imaginary part that corresponds to a maximum energy loss at relaxation frequency (f_R). The Debye relation describes this behaviour:

$$\varepsilon'(\omega) = \varepsilon'_\infty + \frac{\varepsilon'_s - \varepsilon'_\infty}{1+\omega^2\tau^2} \quad \text{and} \quad \varepsilon''(\omega) = \frac{(\varepsilon'_s - \varepsilon'_\infty)\omega\tau}{1+\omega^2\tau^2} \qquad (2)$$

where ω is the angular frequency, $\varepsilon_s{}'$ the real static permittivity (at low frequencies), $\varepsilon_\infty{}'$ the real high frequency permittivity and $\tau = 1/(2\pi f_R)$. For a given molecule, the relaxation frequency depends on its relations with its environment. Relation 2 is valid for a single monomolecular material while our samples contain various polar molecules, ions and polar clusters. Moreover, the state of each molecule corresponds to a relaxation frequency. However, between 200 MHz and 6 GHz, the water molecule is the main component that relaxes, and it can be assumed that dielectric values correspond to the average behaviour of the water molecules.

The higher the binding of water to food matrices such as protein chains, the lower the water activity and lower the relaxation frequency because this prevents water molecule from easily following the alternative electric field (Fig. 3). Hence a study of dielectric relaxation spectra, and in particular relaxation frequency, is likely to offer a solution to access water activity.

4. Results and discussion

4.1. *Water activity measurements and isothermal sorption curves*

All the water activity measurements on native samples were performed with the Novasina device and are presented in Table 2.

The water sorption isothermal curves are plotted in Fig. 4, using the common scales for three NaCl contents: 0, 0.04 and 0.08 kg/kg dry matter. The results

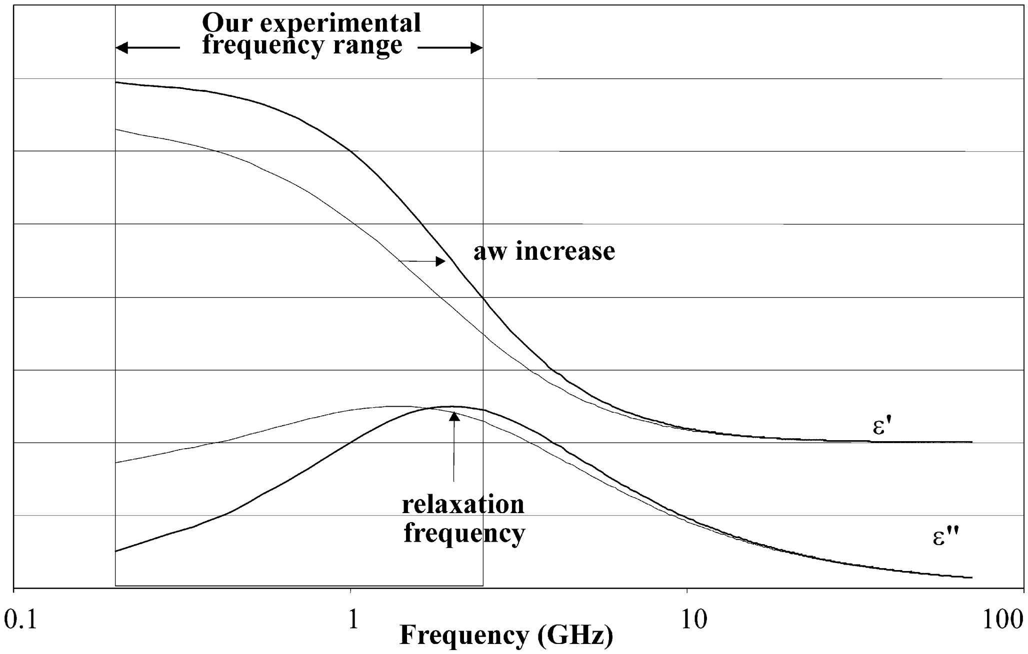

Fig. 3. Debye relation for two different water activities.

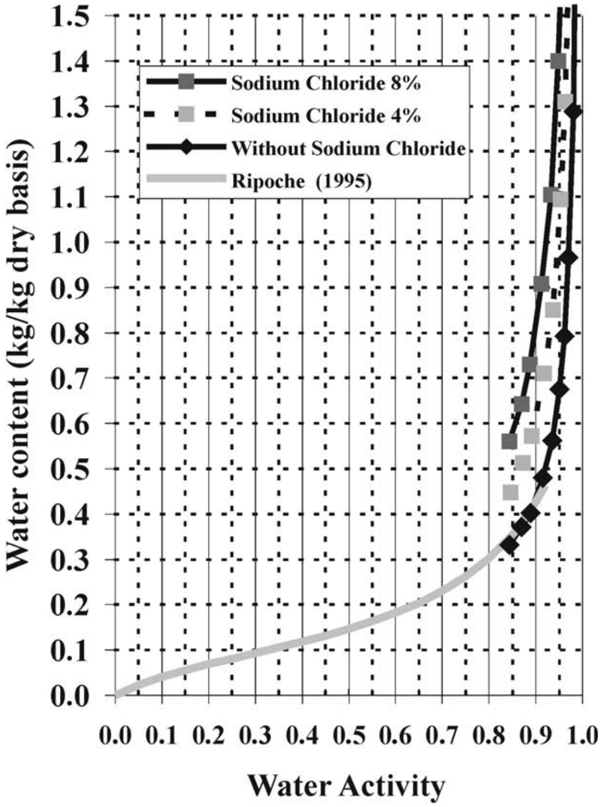

Fig. 4. Comparison of sorption isotherms of gelatine measured with the new method in the high humidity range to results obtained in the usual range with the standard saturated salt method.

of Ripoche (1995), who measured a gelatine water sorption isotherm at ambient temperature using the standard saturated salt method, have been fitted with the usual G.A.B. model up to $a_w = 0.92$ and plotted in the same figure for comparison. Our results in the high humidity range, for the samples without sodium chloride, are in the exact continuation of this curve. In agreement with theory and published results on the effect of NaCl addition in foods, the higher the NaCl content the higher the water content at the same water activity.

For the sake of clarity, all the results are grouped in Fig. 5, focussing on the high humidity area. Two vertical lines indicate the thresholds for the growth of the indicated bacteria; their growth rate is nil at the threshold and increase to a maximum at $a_w = 1$. Whatever the NaCl content, the sorption isotherms show an asymptotic behaviour. As regards the difficulties in measuring water activity above 0.99, there is a reasonable agreement between the measurements of a_w with the Novasina device (empty circles, diamonds, triangles and square dots for, respectively, 0, 4, 6 and 8% salt content) and the sorption isotherms. These results provide a set of data from which water activity can be derived by interpolation, for any value of the couple (water content, NaCl content). They can also be used to extrapolate the correlation between dielectric properties and a_w identified from the measurements for water contents higher than 1 kg water/kg dry matter.

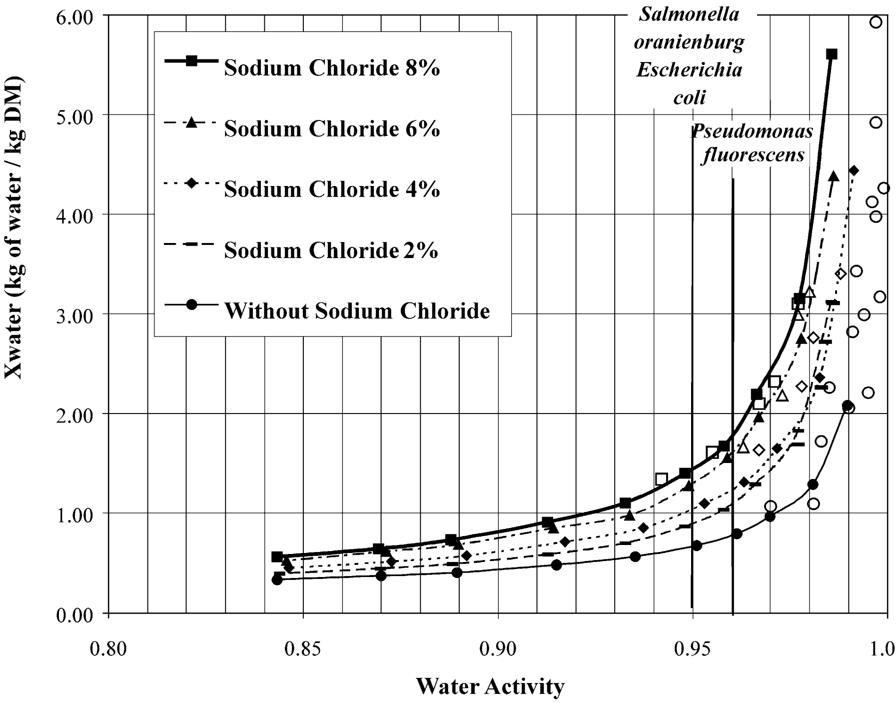

Fig. 5. All experimental results for a_w measurements.

4.2. Dielectric measurements

As shown in Fig. 3, water activity changes theoretically lead to dielectric spectrum translations. The framed part gives the limits of our experimental frequency range. The analysis of the experimental results seeks the best experimental parameter to describe water activity changes. In our frequency range, candidates are:

- Relaxation frequency which should increase as a_w.
- The slope of ε' between 5 and 6 GHz. This parameter should become more negative as a_w increases if relaxation frequency is below 6 GHz or less negative if it is above 6 GHz.
- The slope of ε'' between 5 and 6 GHz. This parameter should become less negative as a_w increases if relaxation frequency is below 6 GHz or less positive if it is above 6 GHz.

A preliminary analysis of the results suggested that three other parameters were tested:

- $\varepsilon'/\varepsilon''$ for low frequencies (200 MHz–1 GHz) versus water activity.
- Mean value of ε'' for low frequency (200 MHz–1 GHz) versus salt content.
- The average value of ε'' between 5 and 6 GHz versus water content.

Fig. 6 gives raw experimental data with real and imaginary parts of permittivity versus frequency between 200 MHz and 6 GHz. The small curves are the theoretical dielectric relaxation spectra for our experimental frequency range. Permittivities of five samples with various water activities are presented here. A zoom was performed on the imaginary part around relaxation frequency. The experimental data present some differences from the theoretical curves:

- Variations due to relaxation are smaller on experimental curves than on theoretical curves because our samples are complex materials with some polar molecules, and the relaxation observed is their total contribution. To obtain information on water activity, we assume a large contribution of water molecule in this frequency range.
- Behaviour at low frequency is very different. At these frequencies, polar molecules easily rotate in an alternating electric field, but ions also translate in the material, which leads to electromagnetic losses by a conduction effect. Losses appear as a large value of the imaginary part of the permittivity.

The main differences between samples are a shift in ε' value and a variation of ε'' slope at high frequencies.

These results can also be represented in a classical "Cole-Cole" diagram, which is a plot of permittivity in

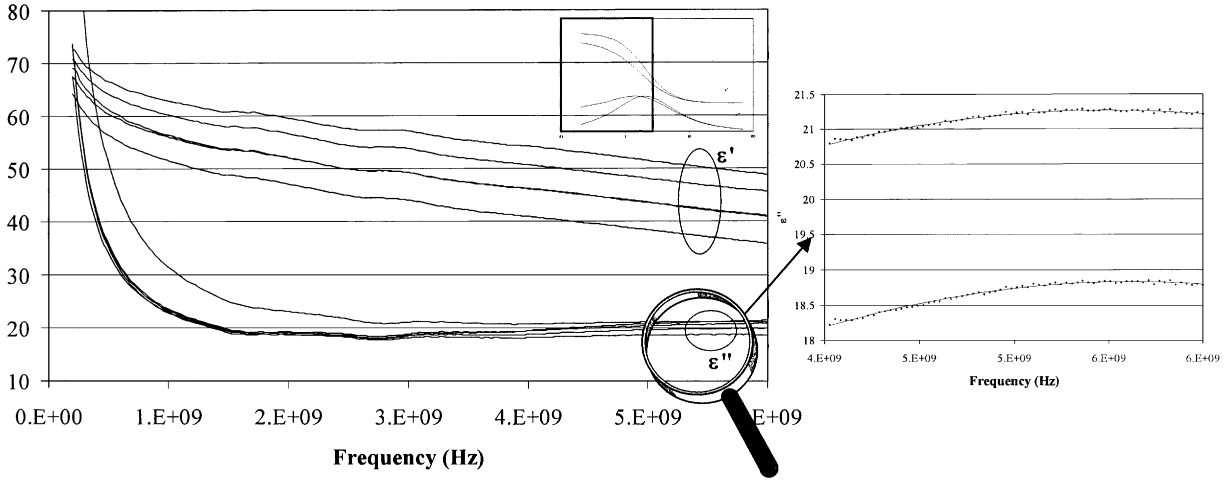

Fig. 6. Dielectric spectra for five samples and zoom on imaginary part.

the complex plane (Fig. 7). High values of real and imaginary parts correspond to low frequencies and frequency increases as permittivity decreases.

Nine samples with various a_w (from 0.942 to 0.992) are presented in Fig. 7. In the area of low frequencies (below 1 GHz), the real part of permittivity increases with a_w. This observation provides the following dielectric parameter for a_w evaluation:

$$\frac{\varepsilon'}{\varepsilon''}\Big|_{f \leqslant 1\mathrm{GHz}} \tag{3}$$

This parameter is presented in Fig. 8 versus a_w for the 45 native samples. An exponential fit of the results was obtained with a good correlation coefficient: $r^2 = 0.86$. This behaviour can be explained by phenomena that appear at low frequency: conduction rather than relaxation may explain it. However, the distribution of the experimental points around the fitting curve is too large and the variation of this dielectric parameter is too small for water activity below 0.96 to be used in practice.

This prompts us to correlate the dielectric properties independently of salt and water contents in samples.

Fig. 9 gives the low frequency dielectric losses ε'', averaged in the frequency range 200 MHz to 1 GHz, that correspond to conductivity and then salt content. This parameter grows linearly with salt content while water content varied from 1 to 10.5 kg of water per kg of dry matter for each value of salt content.

In Fig. 10, the mean value of ε' between 5 and 6 GHz is plotted against the water content for salt content varying from 0 to 0.12 kg of salt per kg of dry matter for each value of water content. A logarithmic type relationship fits these results rather well.

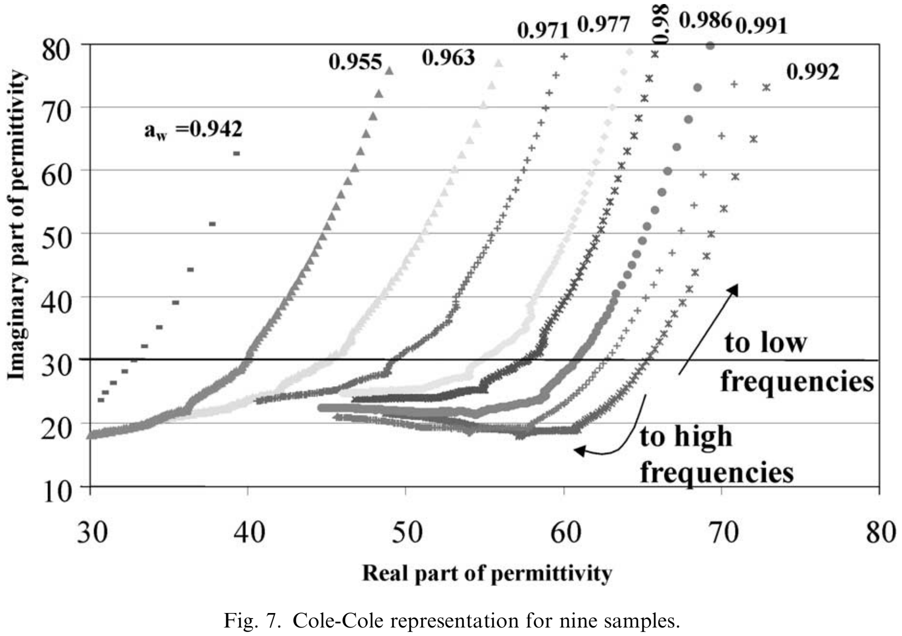

Fig. 7. Cole-Cole representation for nine samples.

Therefore, it seems that both salt and water content can be determined by non-destructive dielectric measurements. In materials where water activity is essentially linked with these two components, this information could be combined with the isothermal sorption curves shown in Fig. 5 to assess the water activity.

It is first necessary to investigate the range of water content below 1 kg of water per kg of dry matter to complete Fig. 10. This will be achieved using cavity dielectric measurements on the very small (size: 25×10×2 mm) dehydrated samples prepared with the INRA-made sorption isotherm device. In order to better-fit experimental data in Fig. 10, a measurement of ε'

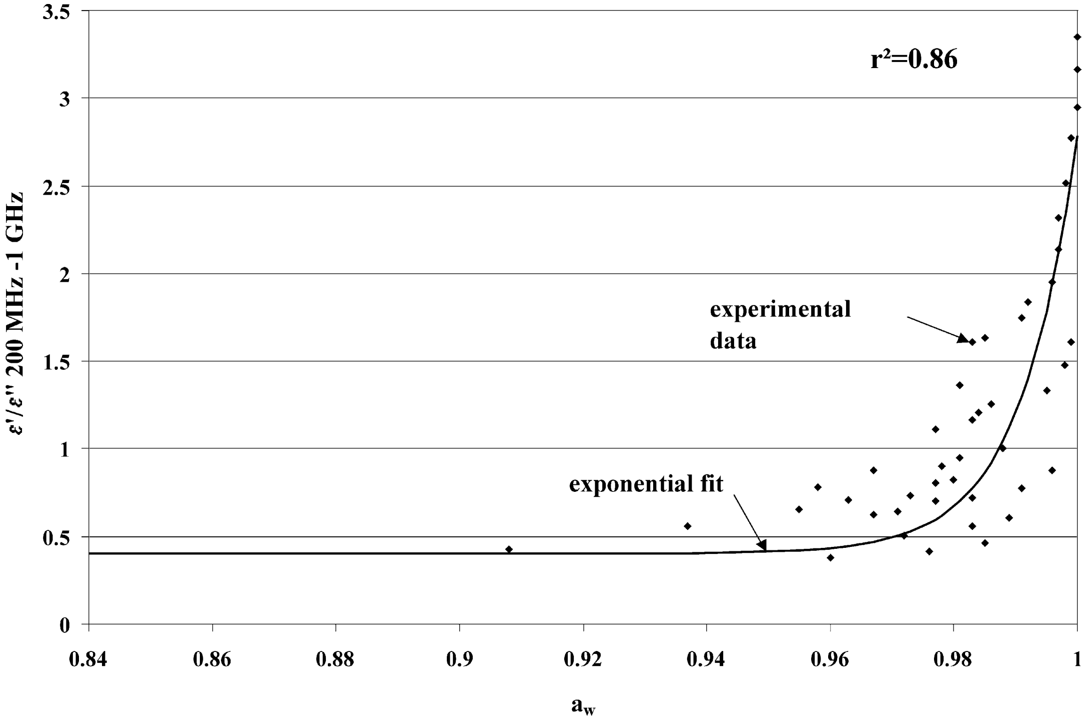

Fig. 8. $\varepsilon'/\varepsilon''$ for low frequency (averaged value between 200 MHz and 1 GHz) versus a_w. Experimental data and exponential fit.

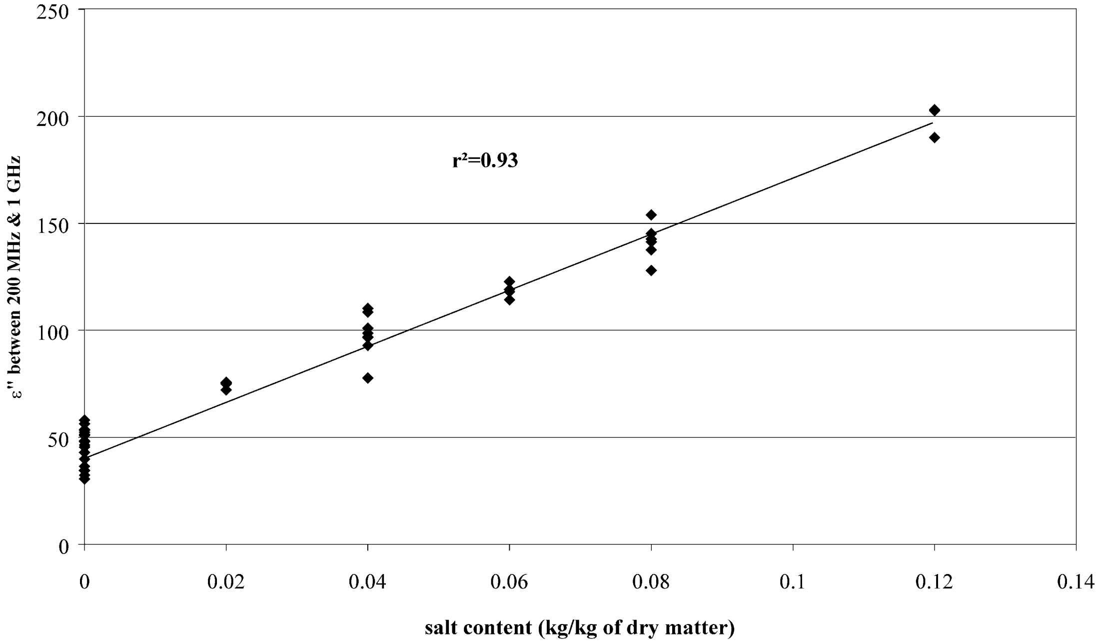

Fig. 9. ε'' at low frequency (averaged value between 200 MHz and 1 GHz) versus salt content.

for dry sample is planned. Measurements also need to be performed on all kinds of food components: fat, proteins, and on real material, such as meat fermented products or cheese.

Parameters connected with the shift in dielectric spectrum with a_w change are shown in Figs. 11 and 12.

The relaxation frequency (f_R) (Fig. 11) was estimated by fitting the experimental data with the Debye relation (2), which gives the theoretical variation of permittivity with frequency for a monomolecular material. When water activity increases, water molecules are less tightly bound and so can easily rotate, and the relaxation frequency logically increases. The curve has two parts: a linear increase for frequency above 2 GHz, and a flat part below it. This flat part is easily explained by the contribution of conductivity phenomena at low frequencies. For relaxation frequencies below 2 GHz, the maximum in ε'' curve is lost in the ε'' increase due to

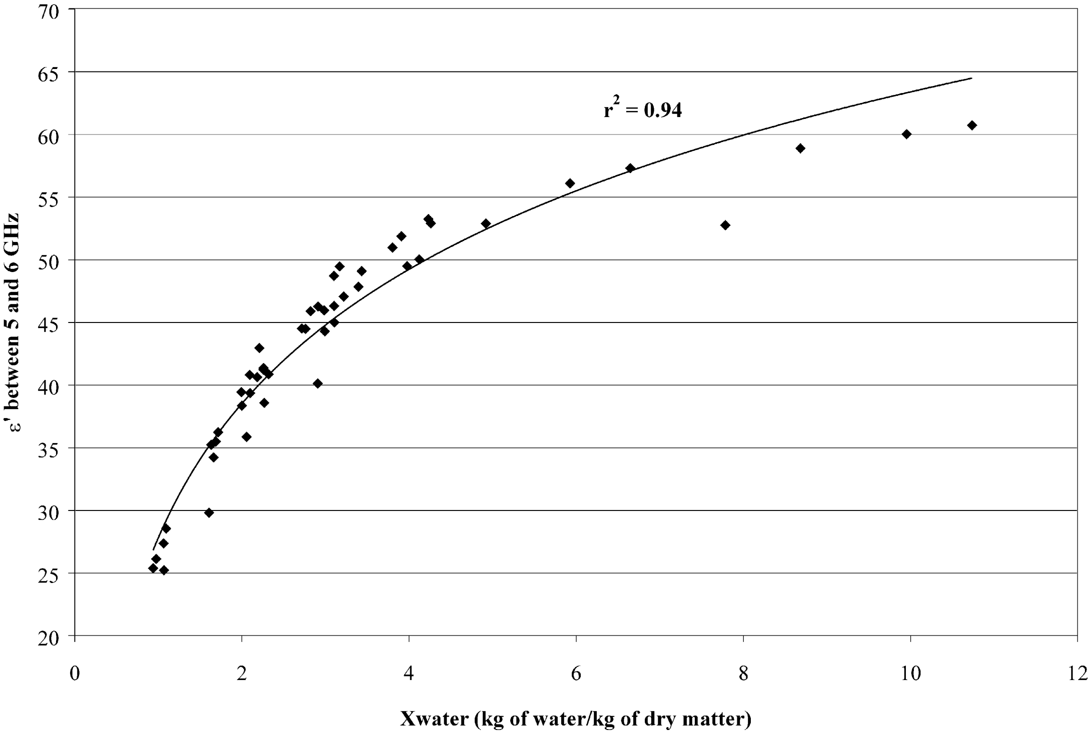

Fig. 10. ε' at high frequency (averaged value between 5 and 6 GHz) versus water content.

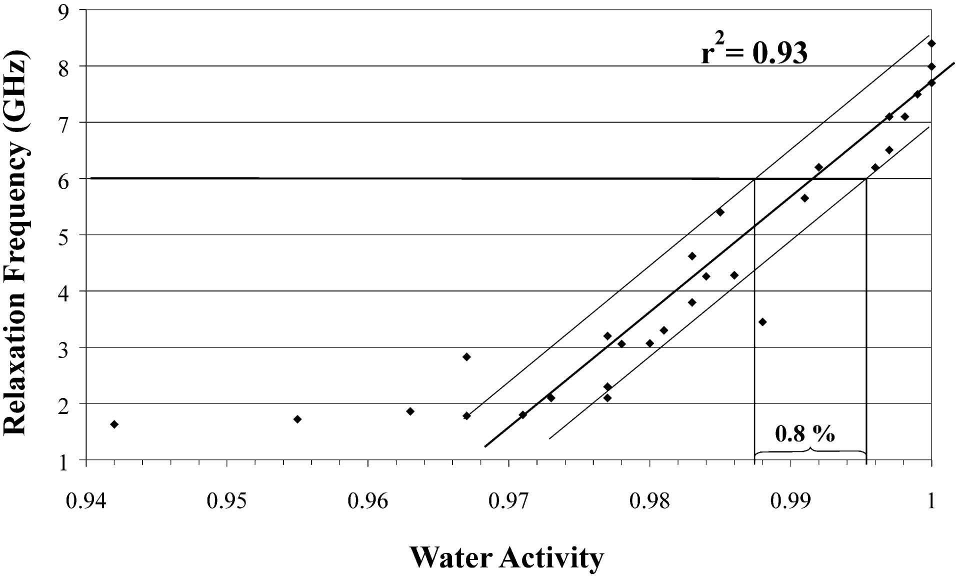

Fig. 11. Relaxation frequency versus water activity.

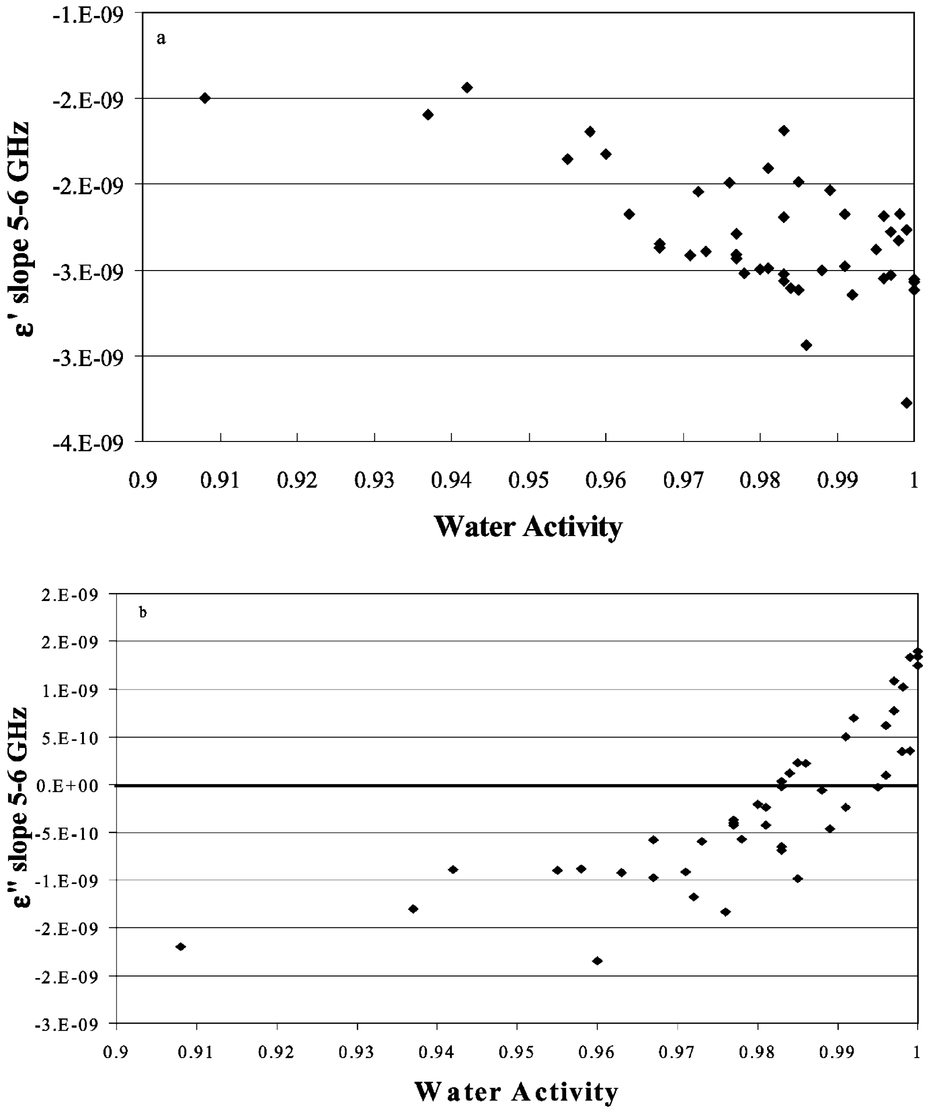

Fig. 12. Slope at high frequency versus a_w for (a) ε' and (b) ε''.

conductivity (see Fig. 6). Thus, it becomes wrong to fit the experimental data with a Debye relation. Results show that for a_w below 0.99, the relaxation frequency is in our frequency range ($f_R < 6$ GHz). These results are interesting for water activity control in meat since the growth rate of *pseudomonas*, which is the main spoiling bacterium that limits the shelf life in chilled storage, varies from nil at 0.96 to the maximum at 1 (Robles Olvera, 1999). Moreover, this dielectric measurement method favours the contribution of the first micrometres in depth.

The slope of real permittivity ε' at high frequencies (5–6 GHz) does not significantly change with water activity (Fig. 12a). The slope becomes slightly more negative, which corresponds to the increase in relaxation frequency. For a_w above 0.98, it remains steady because the relaxation frequency enters the frequency range 5–6 GHz where the slope is computed.

The imaginary permittivity slope at high frequencies (5–6 GHz) reverses from a negative to a positive slope when water activity increases (Fig. 12b). This slope reversal around $a_w = 0.99$ signals the relaxation frequency entering the 5–6 GHz frequency range (see Fig. 11). Sensitivity is better than for the ε' slope as the derivative of the ε' curve in the relaxation frequency range is fairly constant.

The variations of the two above parameters are consistent with the results on the relaxation frequency parameter (Fig. 11) but cannot be used for water activity control parameters because they have poor sensitivities.

5. Conclusions

Results concern six dielectric parameters that were correlated with a_w, salt content or water content. The first one: $\frac{\varepsilon'}{\varepsilon''}|_{f \leqslant 1\mathrm{GHz}}$ was more linked to low frequency conductivity effects. The average value of ε' at high frequencies (5–6 GHz), which theoretically varies with both conductivity and relaxation, was very well correlated with water content. The dielectric losses ε'', averaging between 200 MHz and 1 GHz, are linearly correlated with salt content in the samples.

Three parameters were directly connected to relaxation frequency shift, theoretically associated with a_w change. The relaxation frequency is well correlated with water activity above 0.97. In this range, it can predict water activity with a 0.8% error. Improvements will be caried out by using a complex Debye relation, which take into account the contribution of several relaxation phenomena in the samples. Below 0.97, the ε'' maximum associated with relaxation cannot be accessed, owing to conductivity phenomena. The high frequency (5–6 GHz) slopes of ε' and ε'' also vary with water activity in coherence with the previous parameter (f_R).

These first results confirm the theoretical relation between dielectric properties and water activity. But, we are still far from an industrial prototype. Dielectric measurements were only carried out on model samples with very high homogeneity and perfect contact surfaces. Real food material will of course be more difficult to handle.

It seems feasible to combine sorption isotherms with dielectric measurements: a sorption curve preliminary laboratory data bank, associated with in situ fast measurements of low frequency ε'' and high frequency ε' will give fast a_w control during processing of foodstuffs.

To continue this study, dielectric characterisation must be performed on samples dehydrated for sorption measurements which are sized (25×10×2 mm) for fast equilibrium in sorption measurements and can no longer be used in coaxial line dielectric spectroscopy measurements. Measurements in resonant cavity, which are compatible with such small samples, are planned.

To improve this experimental method, and adapt it to real materials, we also need to make more sample measurements, on different kinds of model materials (with other a_w depressors), but also on real food products. Although much work remains to be done to obtain an operational experimental method, both industrial applications and the performance of research tools stand to benefit.

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