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Study of the effect of microwave power coupled with hot air drying on orange peel by dielectric spectroscopy

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ABSTRACT

Monitoring moisture and water activity during drying is crucial for process optimization, avoiding inadequate uses of energy. The main objective of this work was to study the dielectric properties of orange peel during hot air drying at 55 °C (HAD) and microwave power coupled with hot air drying at different power intensities (2 W/g, 4 W/g and 6 W/g). At 5, 15, 40, 60 and 120 min mass, a_w, moisture, and permittivity were measured in fresh and dried samples. Results allowed developing a dielectric isotherm technique by adapting the GAB model to predict a_w in dried orange peel by using ε' (20 GHz). The physical meaning of the dielectric isotherm parameters (ε'_0 and C_d) was studied. The value of ε'_0 at 20 GHz (γ-dispersion) represents the induction effect of the minimum quantity of adsorbed water or the monomolecular moisture layer. The parameter C_d is related with isosteric heat, as well as the C parameter of the GAB model. The application of MW power produced an increase of isosteric heat or adsorption energy of the monomolecular layer, improving surface tension of samples and thus the hygroscopicity, explaining the reduction of the ε'_0 independently of the quantity of the water molecules adsorbed.

1. Introduction

Drying costs are a major issue in most valorisation processes, especially when the water content of the material is as high as citrus by-products (85%), being critical for their economic feasibility. Therefore, moisture monitoring plays a key role in food processing operations such as drying. However, water interaction with air and food depends more on water activity than on moisture, because the reactivity and the mobility of water are directly related with the water activity. The most common and faster tool to obtain the water activity from the moisture is the sorption isotherm. Sorption isotherms relate water activity to water content of a food product at a certain temperature and pressure. The sorption isotherms have an important role in the quantitative approach to the prediction of shelf-life of dried foods, due to their sensitivity to moisture and water activity changes.

On the other hand, the research of electrical properties of food systems has received tremendous attention in the recent years due to the increased development and application in the range of Hz to THz (radiofrequency, microwaves and infrared) for heating, drying or process monitoring in the food industry. The physical properties that define the heating capability of any material are permittivity for the photon electric interactions and permeability for the photon magnetic interactions (Pozar, 2012). They are affected by many different factors, depending on the range of frequency of the photon flux emitted (Castro-Giraldez, Balaguer, Hinarejos, & Fito, 2014), such as amount of water, temperature, structure (i.e. charge conformation of proteins) and chemical composition (i.e. electrolytes), especially on the presence of mobile ions (Castro-Giraldez, 2010). In the case of electric properties, the permittivity must be explained as a vector number, polar or complex. Permittivity explained as a complex number has two parameters, the dielectric constant ε′ and the loss factor ε″, being the real and imaginary terms of permittivity (ε), respectively. The dielectric constant is related to the material ability to absorb and store electric energy, and the loss factor is related to the dissipation of the electric energy in other energies such as thermal energy.

In the range of microwaves, the interaction of the photon flux with biological tissue produces two main dispersions, γ-dispersion and ionic conductivity. The γ-dispersion is due to the dipolar molecules orientation and induction, producing electric storage

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and dissipation of the electric energy in other energies such as mechanical and thermal energies. The main dipolar molecule of plant tissue is water (Castro-Giraldez, Fito, Chenoll, & Fito, 2010; Castro-Giraldez, Fito, Dalla Rosa, & Fito, 2011; Castro-Giraldez, Fito, & Fito, 2011). The other important effect in microwave range is ionic conductivity. It affects only to the loss factor, because it only produces a repulsion of charged molecules, transforming electric energy into others.

Dielectric spectroscopy has been used for many applications as non-destructive technique for monitoring different processes: pork meat salting (Castro-Giraldez, Fito, & Fito, 2010; Kent, Peymann, Gabriel, & Knight, 2002; Lyng, Zhang, & Brunton, 2005), brewing (Velázquez-Varela, Castro-Giraldez, & Fito, 2013), dehydration (Feng, Tang, & Cavalieri, 2002) and osmotic dehydration of apple (Castro-Giraldez, Fito, & Fito, 2011) and kiwi (Castro-Giraldez, Fito, Dalla Rosa, et al., 2011) and also for determination of apple maturity (Castro-Giraldez, Fito, Chenoll, et al., 2010). Traffano-Schiffo, Castro-Giraldez, Colom, and Fito (2015) analyze the viability of monitoring drying meat processes by using dielectric properties measurements at microwaves frequencies. The authors showed that there was a direct relationship between the dielectric loss factor with respect to sample surface and the number of water molecules at 20 GHz, obtaining also the desorption isotherm. This relationship can also be used for determining food product composition by applying dielectric spectroscopy. Iaccheri et al. (2015) studied water features in green and roasted coffee beans by dielectric spectroscopy.

Talens, Castro-Giraldez, and Fito (2015) analyzed the dielectric loss factor at 2.45 GHz (most commonly used MW frequency in Europe) of orange peel dried by hot air-microwave drying in order to quantify the amount of microwave energy that was absorbed and transformed into heating energy. This calorific energy was absorbed by the water molecules because at 2.45 GHz the relaxations phenomena were caused by the induction of polar molecules like water. Fava et al. (2013) also applied microwave drying to citrus by-products in order to dehydrate the final product for further conversion into dietary fiber with optimal microbial, sensory and technological properties.

In microwave assisted drying processes, the knowledge of dielectric properties and parameters that affect their values allows to predict moisture content, water activity and drying kinetics (Barba & d'Amore, 2012). The aim of this work was to develop and to determine dielectric tools to predict the moisture and water activity by using dielectric spectroscopy and sorption isotherms of orange peel dried by microwave power coupled with hot air drying.

2. Materials and methods

Oranges (*Citrus sinensis* (L.) Osbeck var Washington Navel) were bought from a local supermarket in Valencia (Spain). Orange peels were used for the experiments. 60 orange peel cylinders (20 mm diameter and 3 mm thickness) were cut with a core borer. A diagram of the experimental procedure is shown in Fig. 1.

Samples were subjected to hot air drying (HAD) and microwave assisted air drying (HAD + MW) (Fig. 2), using a specially designed MW-air drying oven (Martín, Martínez-Navarrete, Chiralt, & Fito, 2003) with maximum output 2000 W at 2450 MHz, connected to a computer where temperature of ambient air and hot air, relative humidity of ambient air and incident and reflected microwave energy could be registered. In order to measure incident and reflected energy a directional coupler with power meter was also connected to the computer. The modified microwave oven presents two parallel connected lines (diameter = 105 mm), one for the application of hot air and another for the generation and application of the microwaves. Drying chamber has a Teflon chamber (edge = 100 mm) and a mode stirrer to ensure a homogeneous microwave distribution. Different variables were measured in drying chamber for process control: hot air temperature by a Pt100

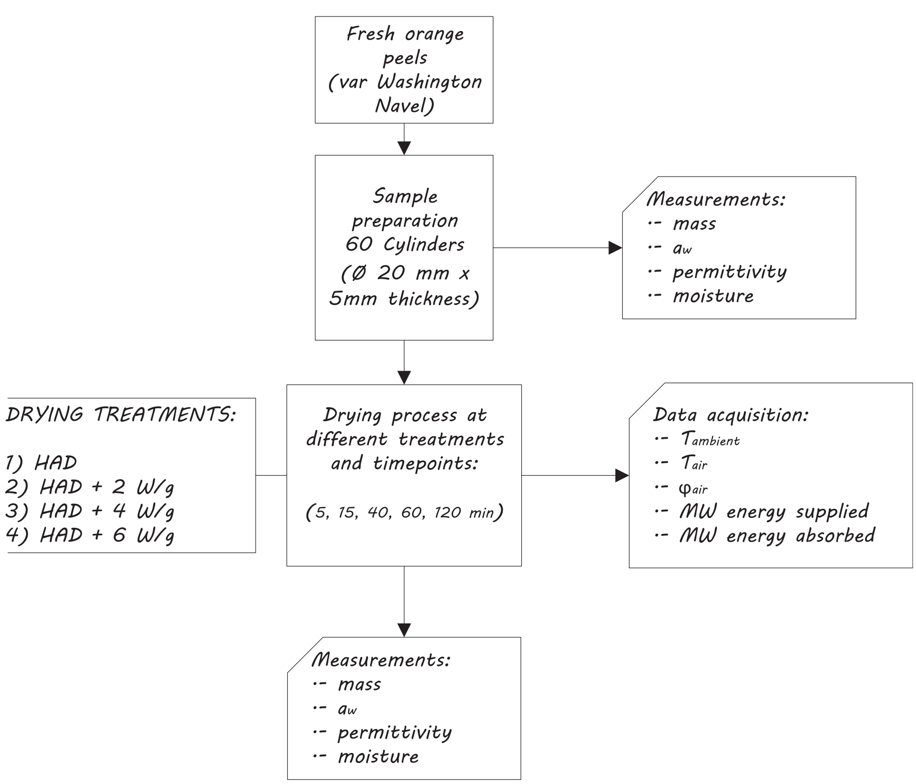

Fig. 1. Schematic diagram of the experimental procedure.

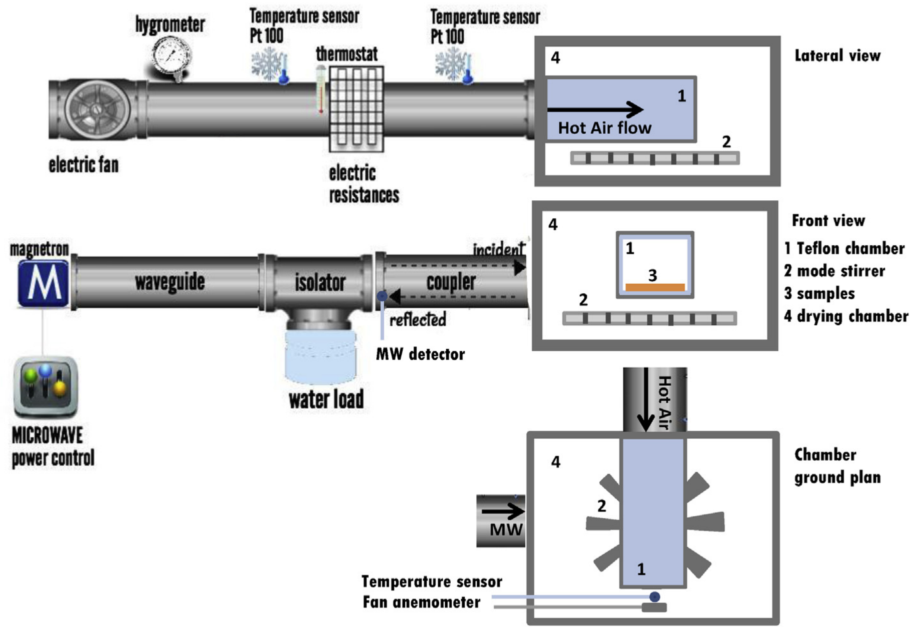

Fig. 2. Schematic description of the laboratory equipment used to dry samples.

thermocouple and air velocity by a fan anemometer.

For the experiments, air velocity was 2.5 m/s, air temperature 55 °C and the MW energy was 0, 2, 4 or 6 W/g. The MW power (W/g) was referred to the initial mass of sample. The microwave energy applied (determined by using the IEC-test) was selected such that samples did not burn during drying.

Orange peel samples were placed with flavedo side up on the dryer grid to facilitate mass transfer. Four drying experiments were carried out (HAD, HAD + 2 W/g, HAD + 4 W/g and HAD + 6 W/g). Three orange peels samples were used for each drying time (5, 15, 40, 60 and 120 min) per treatment. These 3 samples were removed at each time point and were equilibrated at 25 °C for 5 h in aqualab® disposable sample cups sealed with parafilm®, in order to eliminate the concentration profiles in samples. The next three samples were then placed in the drier.

Samples weight was determined by a precision balance Mettler Toledo AB304-S (precision ± 0.001 g). Surface water activity was determined by a dew point hygrometer Decagon Aqualab®, series 3 TE (precision ±0.003, dimensionless) (Decagon Devices Inc., Washington, USA). Measurements were done in structured samples (not minced), thus a_w obtained was considered surface a_w. Water content of representative fresh orange peels and 120 min dried samples was determined by drying in a vacuum oven at 60 °C until constant weight was reached (AOAC method 934.06, 2000). The moisture content of the sample at intermediate drying stages was calculated from the weigh lost during drying.

The permittivity was measured with an Agilent 85070E open-ended coaxial probe connected to an Agilent E8362B Vector Network Analyser. The system was calibrated by using three different types of loads: air, short-circuit and 25 °C ultra pure (Milli®-Q) water. Once the calibration was made, 25 °C ultra pure (Milli®-Q) water was measured again to check calibration suitability. Permittivity was measured by placing the probe on the surface of the samples. All determinations were made from 500 MHz to 20 GHz. The measurements were made in triplicate.

The desorption isotherm was fitted following the GAB model using Equation (1) (van den Berg & Bruin, 1981):

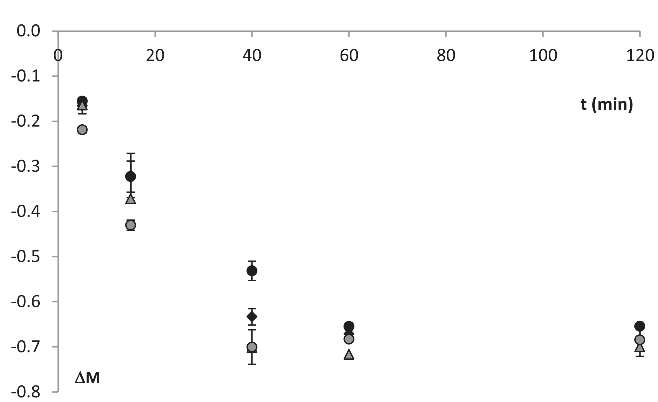

Fig. 3. Mass variation of orange peel treated by different drying treatments: ● HAD, ◆ HAD + 2 W/g, ▲ HAD + 4 W/g, ● HAD + 6 W/g. Data represent means and standard deviation of experiments performed in triplicate.

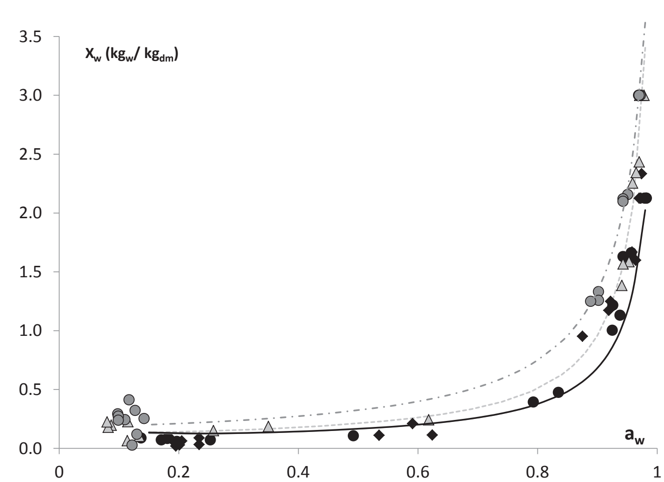

Fig. 4. Sorption isotherm of orange peel treated by different drying treatments: experimental points ● HAD, ◆ HAD + 2 W/g, ▲ HAD + 4 W/g, ● HAD + 6 W/g; and GAB model — HAD, — HAD + 2 W/g, - - - HAD + 4 W/g, - · - HAD + 6 W/g.

Table 1
Estimated GAB parameters (X_{W0}, C and K) and correlated coefficient (R^2) of GAB model for desorption isotherms of orange peel dried by hot air (HAD) and hot air coupled with microwaves (HAD + MW).

	GAB Parameters			R^2
Drying treatment	X_{W0} (kg_w/kg_{dm})	C	K	
HAD	0.080	22.33	0.98	0.9342
HAD + 2 W/g	0.080	18.90	0.98	0.9182
HAD + 4 W/g	0.105	70.33	0.99	0.9197
HAD + 6 W/g	0.165	153.04	0.97	0.8493

$$X_W = \frac{X_{W0}\, C\, a_w}{(1 - K\, a_w)(1 + (C - 1)a_w)} \tag{1}$$

Where: X_w corresponds to the orange peel moisture (kg_w/kg_{dm}), X_{W0} is the monomolecular moisture layer (kg_w/kg_{dm}), C is the energy constant and K is an empirical parameter, both dimensionless (Maroulis, Tsami, Marinos-Kouris, & Saravacos, 1988).

GAB model was fitted by using a non-linear regression with the Statgraphics Centurion XVI Software (Statgraphics, Virginia, U.S.A.).

3. Results and discussion

Mass variation of samples during the drying process can be obtained by the following equation:

$$\Delta \boldsymbol{M} = \frac{\boldsymbol{M}^t - \boldsymbol{M}^0}{\boldsymbol{M}^0} \tag{2}$$

Where M represents the mass of the sample (kg), the superscript t represents the process time, being 0 the initial time. The mass variation can be observed in Fig. 3.

A faster mass reduction can be observed in microwave drying treatments increasing with microwave energy. At 5 and 15 min, very significant differences ($p \leq 0.01$) were found between HAD + 6 W/g and the rest of the treatments. At 40 min, significant differences ($p \leq 0.05$) were appreciated among HAD and 2 W/g and the rest of the treatments. No differences between 4 and 6 W/g were appreciated mainly because a low level of mass was reached caused by water loss; the low level of moisture reached decreases the effect of MW power. After 60 min of drying, treatments converged to the threshold of the thermodynamic properties of dry air ($\Delta\mu_w|^i = 0$ or $a_w|^{sample} \approx \varphi|^{air}$).

Fig. 4 shows the sorption isotherms obtained for each drying treatment (experimental points) and the GAB model applied for each treatment. GAB parameters are shown in Table 1.

In order to explain the variation of the GAB parameters with physical sense in the different drying treatments, the permittivity in range of the microwaves was analyzed.

Permittivity in microwave range was measured in fresh and treated samples after 5, 15, 40, 60 and 120 min of drying. In Fig. 5 it can be observed the dielectric spectra of the fresh and dried orange peels treated at different microwave power energies.

In the range of microwaves, the interaction of the electric field with biological tissue produces two main dispersions, γ-dispersion and ionic conductivity. The first one mainly represents the induction and orientation phenomena of water, and the second one, represents the conductivity of the electrolytes and weak organic acids. In this figure it is possible to observe at high frequencies (above 1 GHz) the common shape of samples with liquid phase, decreasing the permittivity values with the water losses throughout the treatment. Below 1 GHz, in fresh samples, a negative slope of loss factor shows the low ionic strength of the weak organic acids. Ionic conductivity decreases with the drying treatment caused by the limitation of the movement of weak organic acids due to the loss of liquid phase.

Since there is a progressive loss of liquid phase during the drying process, the dielectric loss factor decreases with time. Therefore it is possible to relate the permittivity at high frequency (dipolar effect) with the quantity of water molecules in equilibrated samples (Traffano-Schiffo et al., 2015). The water molecules can be calculated with the following equation (Eq. (3)).

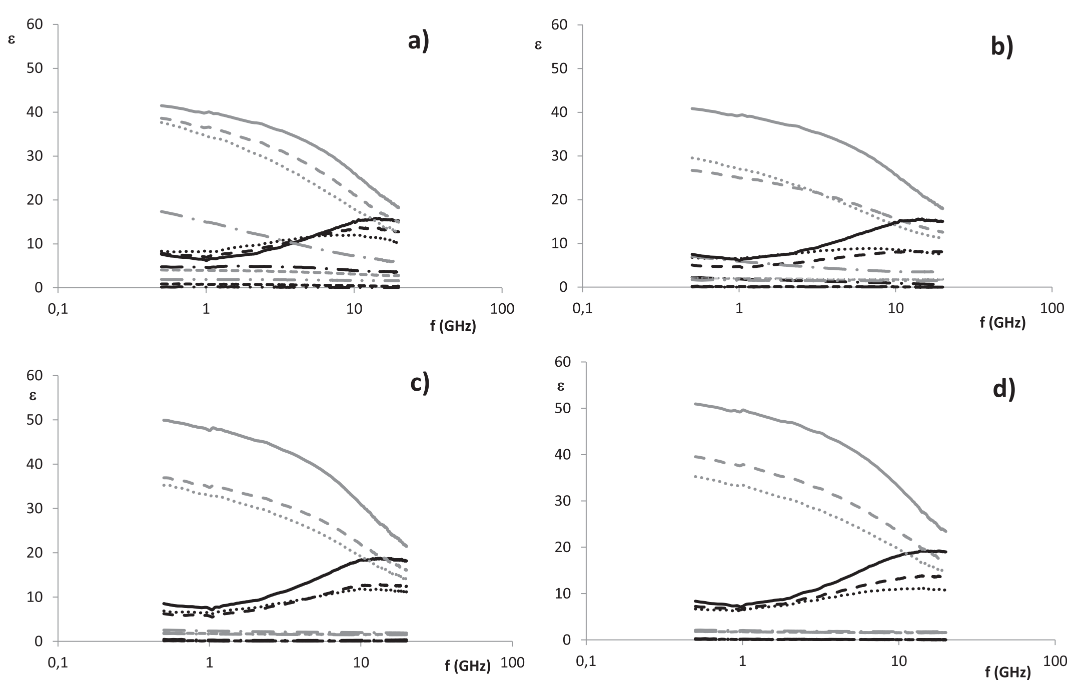

Fig. 5. Permittivity (ε) spectra of orange peels after 0 min ——, 5 min - - -, 15 min • • •, 40 min – ·–, 60 min □□□ and 120 min – ··– of drying by different treatments (a) HAD, (b) HAD + 2 W/g, (c) HAD + 4 W/g, (d) HAD + 6 W/g. Gray lines represent the dielectric constant (ε′) and black lines represent the loss factor (ε″).

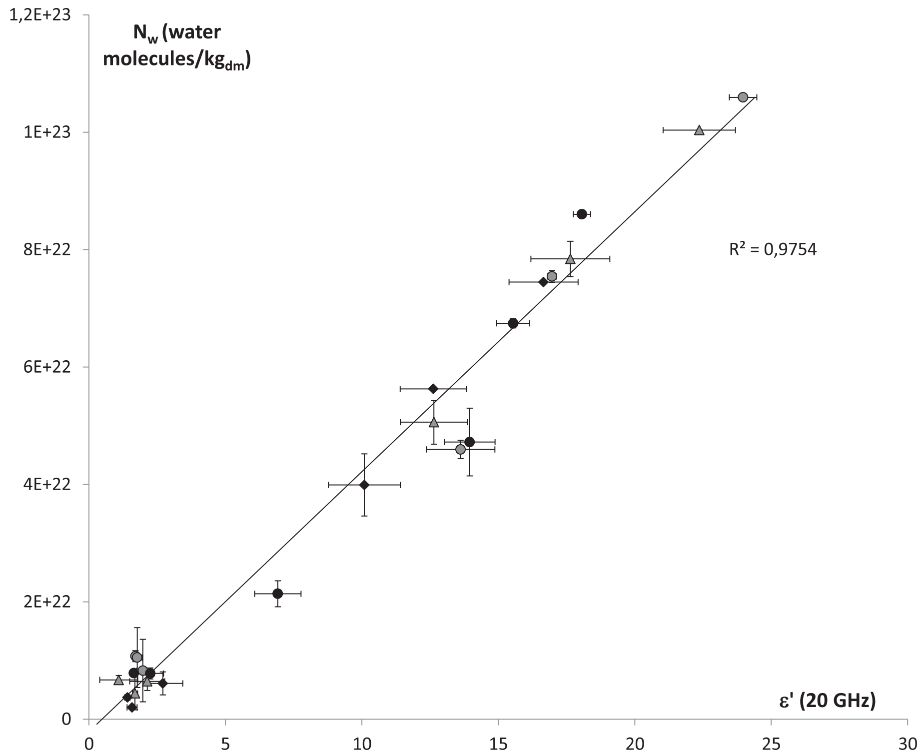

Fig. 6. Relation between number of water molecules (N_w) and the dielectric constant at 20 GHz of orange peels dried by different hot air-microwave treatments. Where: ● HAD, ◆ HAD + 2 W/g, ▲ HAD + 4 W/g, ● HAD + 6 W/g.

$$N_w = \frac{X_w\, N_A}{Mr_w} \tag{3}$$

Where N_w represents the water molecules (number of water molecules in dry basis), X_w is the moisture in dry basis (kg_w/kg_{dm}), N_A the Avogadro constant ($6.022 \cdot 10^{23}$ mol^{-1}) and Mr_W the molar mass of water (18 $kg_w/kmol_w$).

Fig. 6 shows the linear relation between number of water molecules related with the dry matter (water molecules/kg_{dm}) and the dielectric constant at 20 GHz, frequency close to the dipolar relaxation effect.

If the moisture in dry basis explains the dielectric constant, it is possible to develop a dielectric isotherm to predict the water activity in dried samples using equation (4) adapted from GAB model, where ε' (20 GHz) represents the dielectric constant at 20 GHz, close to the water relaxation frequency, ε'_0 is the minimum value of the dielectric constant and C_d and K_d are empirical constants (dimensionless).

$$\varepsilon'(20GHz) = \frac{\varepsilon'_0\, C_d\, a_w}{(1 - K_d\, a_w)(1 + (C_d - 1)a_w)} \tag{4}$$

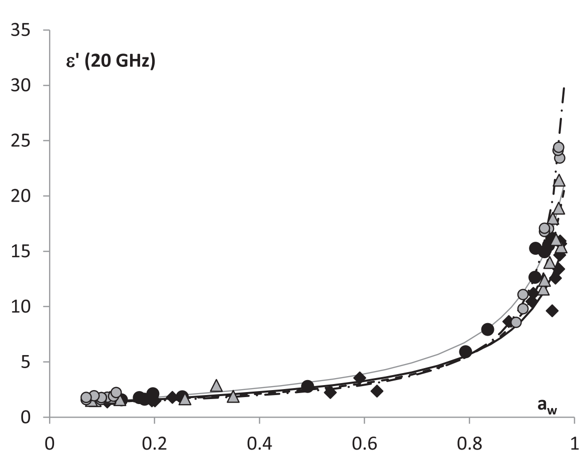

Fig. 7. Dielectric isotherm at 20 GHz of orange peel treated by different drying treatments: experimental timepoints ● HAD, ◆ HAD + 2 W/g, ▲ HAD + 4 W/g, ● HAD + 6 W/g and calculated model — HAD, — HAD + 2 W/g, - - - HAD + 4 W/g, - · - HAD + 6 W/g.

Fig. 7 shows the dielectric isotherms obtained by adapting the GAB model to dielectric approach previously explained. This figure shows that the dielectric isotherm of HAD treatment is above the rest of treatments. The dielectric isotherm equation (equation (4)) applied to the different treatments obtained well fittings (for HAD a R^2 0.8960 for HAD + 2 W/g a R^2 0.9494, for HAD + 4 W/g a R^2 0.8833 and for HAD + 6 W/g a R^2 0.9988).

The K_d parameters were similar in all treatments and the average value was 0.95 ± 0.02. In order to compare the dielectric

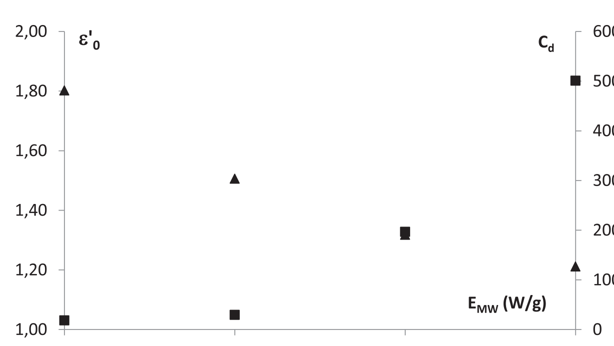

Fig. 8. Dielectric isotherm parameters (adapted to GAB model) of orange peels dried by different hot air-microwave treatments: ε'_0 ▲ and C_d.■.

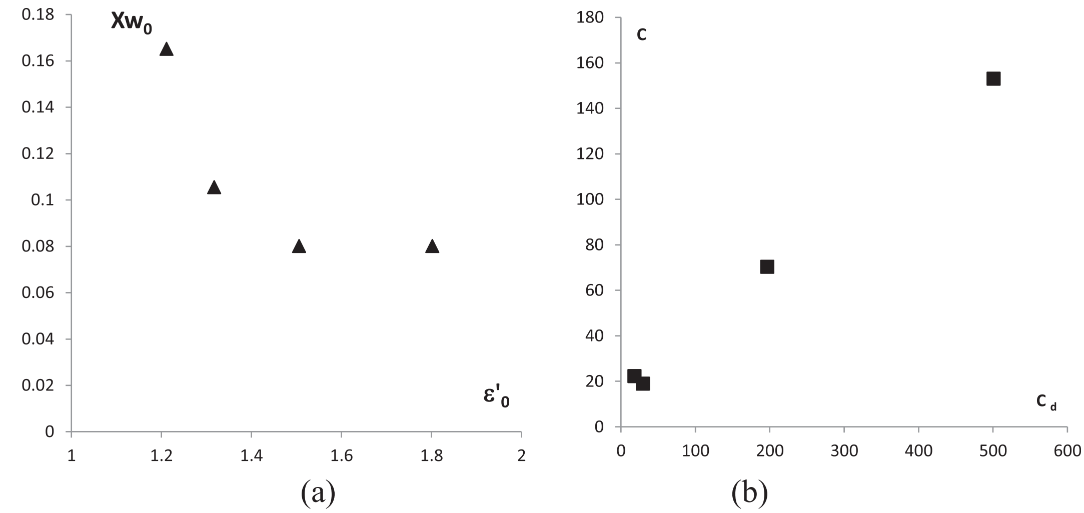

Fig. 9. Comparison of the GAB parameters and dielectric isotherm parameters. (a) Relation between X_{W0} and ε'_0 and (b) Relation between C and C_d parameters.

isotherms, the parameters of equation (4) have been compared in Fig. 8.

Fig. 8 relates in primary axis the minimum dielectric constant with the treatments, where it is possible to observe a homogeneous decrease with the MW power. Minimum value of dielectric constant at 20 GHz (γ-dispersion) represents the induction effect of the minimum quantity of adsorbed water or the monomolecular moisture layer. Therefore, the application of MW power in the dehydration process produces a decrease in the induction of the adsorbed water molecules (ε'_0). This decrease could be caused by the diminution of the X_{w0} (reduction of molecules adsorbed) or the increase of the isosteric heat (increase in the adsorption energy). Fig. 8 shows, in secondary axis, the parameter C_d with the treatments. The parameter C_d is a modification of the C parameter of the GAB model, this parameter explains the isosteric heat or the adsorption energy of the monomolecular moisture layer. In this figure, parameter C_d increases with MW power.

Fig. 9a and b relate the GAB parameters with the dielectric isotherm parameters. Fig. 9a shows the relation between the X_{w0} and the ε'_0, where it is possible to observe that the minimum value of X_{w0} corresponds with the maximum value of ε'_0, in opposite sense the Fig. 9b shows the linear relation between C and C_d, increasing together with the MW power. Therefore, during a drying process, the application of MW power produces an increase of the isosteric heat or adsorption energy of the monomolecular layer, improving the surface tension of samples and thus the hygroscopicity, explaining the reduction of the ε'_0 independently of the quantity of the water molecules adsorbed.

4. Conclusions

It was possible to develop a dielectric isotherm technique by adapting the GAB model to predict the water activity in dried orange peel by using ε' (20 GHz). The physical meaning of the dielectric isotherm parameters (ε'_0 and C_d) was studied. The value of ε'_0 at 20 GHz (γ-dispersion) represents the induction effect of the minimum quantity of adsorbed water or the monomolecular moisture layer. The parameter C_d is related with isosteric heat or the adsorption energy of the monomolecular moisture layer, as well as the C parameter of the GAB model. The application of MW power produced an increase of the isosteric heat or adsorption energy of the monomolecular layer, improving the surface tension of samples and thus the hygroscopicity, explaining the reduction of the ε'_0 independently of the quantity of the water molecules adsorbed.

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