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Dielectric Properties of Low Moisture Foods Measured by Open-ended Coaxial Probe and Cavity Perturbation Technique

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Abstract

The measurement of dielectric properties of foods is essential in the design and control of microwave drying systems as they describe the capability of a material to absorb, transmit and reflect electromagnetic energy. The relative complex permittivity, ϵ_r^* , explains the interactions that may take place between a product and microwave. ϵ_r^* is influenced by its composition (including the moisture content) and temperature. The real component of this complex permittivity is related to energy storage and is called dielectric constant, ϵ_r' . The imaginary component, the loss factor or ϵ_r'' , is associated to energy dissipation. The dielectric properties of selected low-moisture products (corn starch, curry, paprika, dried chives, rice grain and wheat grain) were evaluated by open-ended coaxial probe (OECF) and cavity perturbation techniques. Semi-skimmed milk powder was heated at 50 and 60°C, and the change in dielectric properties with increasing temperature was measured. The increase in moisture content (from 7.19 to 13.08%, wet basis, w.b.) and its influence on the relative complex permittivity was verified for semi-skimmed milk powder. The results showed that the dielectric constant tends to increase with the increase in temperature and moisture content, and with the decrease in frequency from 2450 to 915 MHz. Values ranged from 1 for corn starch (OECF at 2450 MHz) to 4.36 for rice grain (cavity perturbation at 915 MHz). The loss factor ranged from 0.02 for curry to 0.48 for rice grain, both at 2450 MHz. Open-ended coaxial probe requires close contact between the sample and the probe; therefore, this system was not fit for measuring the dielectric properties of the tested powders (air gaps between the probe and the samples). The cavity perturbation technique has been proven to be reliable for many foods, especially at low moisture contents, but requires a specific microwave applicator to be designed for each evaluated frequency.

1. Introduction

Low moisture foods (less than 20% moisture) with low water activity (a_w lower than 0.7) are commonly used for various applications in food industry (Ozturk et al., 2016). Even though they do not favor microbial growth, the contamination of these products has reached a major safety concern. *Salmonella*, for example, was reported in several low moisture powders over the last years (Alfaifi et al. 2014; B. Lin and Wang 2020; Sekhon et al. 2021; Zhang et al. 2020). This microorganism has a high heat resistance even in low-moisture foods, therefore conventional pasteurization treatments, as steam and hot air, may not be completely effective. Moreover, these techniques require long processing times, which can cause a non-negligible loss of quality. Also, particulate products are often infested by insects.

In view of this context, microwave (MW) heating is a valuable means to process low-moisture products, as it heats the materials volumetrically leading to the accumulation of internal vapor pressure that moves the moisture out of the material (Feng et al. 2012). The use of microwaves can promote shorter time to reach target temperatures, thus limiting surface overheating and resulting in food with better quality and treatment at a reduced cost. Besides pasteurization, the application of microwaves, and consequently knowledge of dielectric properties, is ideal for disinfection, sterilization, drying and extraction of biochemicals, for example (Routray and Orsat 2018). Despite the wide possibilities of industrial applications, the microwave technology is still poorly understood, mainly when it comes to wave-matter interactions (Curet et al. 2014).

To develop an effective treatment, it is important to have knowledge of dielectric properties, as they demonstrate the interaction between the electromagnetic energy and the food (Ozturk et al. 2016). The determination of these properties is essential to develop optimized microwave applicators, as it helps to understand the relationship between the electromagnetic field and the material being processed. Also, it allows to quantify permittivity changes by differences in composition (such as added sugar and salt), provides a means of calculating the power penetration depth, enables the evaluation and suitability of packaging materials, along with others (P. Risman 2009; Sharma and Prasad 2002).

The dielectric properties of a material characterize the interaction of the product with electromagnetic fields. It consists of the real and imaginary parts of the complex permittivity ϵ_r^* ($\epsilon_r^* = \epsilon_r' - j \bullet \epsilon_r''$; $j^2 = -1$), which are named dielectric constant and loss factor, respectively, and are frequency and temperature dependent. The relative dielectric constant ϵ_r' represents the ability of a material to store electrical energy. The relative dielectric loss factor ϵ_r'' expresses the conversion of electromagnetic energy as heat. These properties, once obtained, can be used, for example, as inputs for modeling microwave heating (Lau et al. 2020; S.O. Nelson 2006). Many factors influence the dielectric properties of foods, as frequency, temperature, moisture content (MC), bulk

density and composition (especially salt and fat contents) (Nelson, 1984; Tang, 2005). Dielectric properties are essential to estimate the energy conversion efficiency, the absorbed power, and to calculate the penetration depth when applying MW to a material, being fundamental parameters for the design of processes and equipment (Wang et al. 2013).

Several studies have been conducted to investigate the dielectric properties at microwave frequencies for low moisture foods such as vegetable powders (Ozturk et al. 2016; Qi et al. 2021), flours (Bansal et al. 2015; W. Guo et al. 2008; Wenchuan Guo et al. 2010; B. Lin and Wang 2020; Zhu et al. 2012), starch (Tao et al. 2020), spices (Wenchuan Guo and Zhu 2014; Ozturk et al. 2018), chili powder (Li et al. 2018), among others. However, foods are complex materials with a wide range of compositions and dielectric properties, so it is often mandatory to measure the permittivity under specific conditions in order to obtain reliable data. Furthermore, most the known techniques have reduced accuracy for low-moisture and frozen foods (S. O Nelson 1994; Stuart O. Nelson and Bartley 2002).

The transmission and resonance techniques are used for measuring dielectric properties. The transmission techniques, as the open-ended coaxial probe (OECF), are able to sweep microwave frequencies for a determined frequency range but lack accuracy to measure low loss factors. The resonance techniques, on the other hand, are only used for the characterization of dielectric properties at one or certain frequencies at a time. Nevertheless, they have higher accuracy than measurements by the transmission techniques, especially to determine the dielectric loss (Sheen 2005, 2007).

The open-ended coaxial probe is a cut off section of the transmission line technique. The material is measured by immersing the probe into a liquid or touching it to the flat face of a solid (or powder) material. The reflected signal (S11) is measured and related to the complex permittivity. A typical measurement system using a coaxial probe method consists of a vector network analyzer, a software to calculate permittivity, and a coaxial probe. Even though it can be applied to granular materials, this technique is best suited for measuring the dielectric properties of homogeneous liquids and soft semi-solids, as it requires close contact between the flat tip of the probe and the product (Keysight Technologies 2017). The OECF is one of the most applied methods, and it has been used for granular materials as well. However, the accuracy of measurements for food products at temperatures other than 25 °C may be reduced (Routray and Orsat 2018).

Resonance techniques are generally divided in two types, that is, one in which the resonance is achieved by the dielectric sample and the other where the resonance is promoted by the metallic walls of the cavity. In the latter case, the presence of a dielectric sample within the cavity induces only a “perturbation” on the electromagnetic field distributions in the cavity. The cavity perturbation method is based on the shift of the resonant frequency and change in quality factor due to the presence of a small sample inside the cavity. This technique was designed for fully inserted samples that occupy a narrow volume inside the cavity (Bethe et al. 1943; Rubinger and Costa 2007; Slater 1946), and has proven to be reliable for dielectric measurements of many foods over several frequencies, temperatures, and moisture contents. Some of the advantages are that the samples are easy to prepare, the measurement and calculation of dielectric properties are fast, and the material needed is simple and reasonably inexpensive. The disadvantages may include the small size of the sample in relation to the size of the cavity, which can interfere with the test's reproducibility, the shape of the sample to be measured (i.e., a cylinder with a small diameter occupying the full height of the cavity), and the need of a different cavity for each evaluated frequency (Ohlsson et al. 1974).

The objective of this study was to measure the dielectric constant and loss factor of low moisture foods, comparing the results obtained by open-ended coaxial probe and cavity perturbation technique. For one preselected product, semi-skimmed milk powder, the dielectric properties were measured at different levels of water activity and temperatures using the open-ended coaxial probe technique.

2. Materials And Methods

2.1. Low moisture content foods

Corn starch, paprika, curry, dehydrated chives, wheat grains and rice were purchased from commercial establishments. Semi-skimmed milk powder was produced at ONIRIS University (Nantes, France).

The products were kept in their original packaging and stored in a dry place, protected from excessive light.

The samples of semi-skimmed milk powder, taken from the same batch, were also stored in desiccators with saturated salt solutions at room temperatures, to equilibrate with relative humidity (RH) of 44% (K_2CO_3), 58% (NaBr), and 85% (KCl). The dielectric properties of the product with changes in water activity were measured by open ended coaxial probe.

The semi-skimmed milk powder was chosen to be heated up to 50 and 60°C for the measurements by OECP and cavity perturbation, to assess the impact of increase in temperature on the dielectric properties of this product.

2.2. Moisture content and water activity

The moisture content was obtained by drying the samples for 24 h in a conventional oven at 105°C (MEMMERT GmbH, Germany), according to AOAC (2012).

Water activity (a_w) was measured with a digital hygrometer (Aqualab, Decagon Devices, USA). All the measurements were performed in triplicate.

2.3. Tapped density

The tapped density of powders was determined as the methodology previously described by Bansal et al. (2015). As stated in the literature, the dielectric properties of granular or particulate materials depend on sample density (Stuart O. Nelson 1984; Venkatesh and Raghavan 2004). The tapped density allows to accurately determine the mass of product that should be used for the measurements with the coaxial probe. Samples were put in a graduated glass cylinder (capacity of 25 mL) and then tapped 80 times on a table, from a height of around 5 cm. The final volume, once the particles settled, was used to calculate the tapped density for each product.

2.4. Determination of dielectric properties by coaxial probe

The dielectric properties were determined with an open-ended coaxial probe (Dielectric probe kit 85070^E, Agilent Technologies, Malaysia) coupled to a Vector Network Analyzer (ENA 5062A, Agilent Technologies, Malaysia), which was switched on for at least 30 min before the calibration. The calibration procedure consisted of measuring the reflected signal S11 at the same frequency of the tests and at open circuit (air), short circuit (metal) and with a load (distilled water at 20°C).

A custom-designed device, shown in Fig. 1.a, allowed the proper contact between the probe and the surface of the product. The conception of this device was based on published works found in the literature (Bansal et al. 2015; Boreddy and Subbiah 2016; W. Guo et al. 2008; Li et al. 2018). Once the device was filled with predetermined volume of sample (using the tapped density to determine the mass to be introduced), the screw was turned to move the product towards the probe, adjusting the volume to the respective mass and providing an optimal contact at the probe-product interface. The reflected signal S11 was obtained for 101 frequencies in the 915–2450 MHz range with a minimum of 5 measurements for each product (a new sample was used for each measurement). The dielectric constant and loss factor were calculated from the magnitude and phase of the reflected wave by the Agilent software, which is coupled to the Vector Network Analyzer (VNA).

For the measurements of semi-skimmed milk powder at temperatures of 50 and 60°C (controlled by an optical fiber with datalogger H201, Rugged Monitoring, Canada), a water bath (Julabo model F32, Germany) promoted the heating and circulation of water around the product. The system was calibrated using the open circuit (air), short circuit and distilled water at 20°C. Due to the limitation of this experiment and the difficulty in obtaining good contact between the probe and the product, the samples were not replaced once a proper contact was achieved. The powder was placed in an adapted flask and the measurements were performed using the apparatus displayed in Fig. 1.b. At least five measurements were registered for the same sample.

2.5. Determination of dielectric properties by cavity perturbation technique

The devices used for the determination of dielectric properties by cavity perturbation were straight WR340 and WR975 waveguides made of brass and operating at 2400–2500 MHz (Fig. 2.a) or 900–930 MHz (Fig. 2.b), respectively. Both cavities

transmitted the fundamental mode TE₁₀. The minimum reflected power from the load is achieved by placing the empty sample container (quartz tube without product) inside the cavity, and then reaching the adaptation through an iris made of copper, which is fixed between the waveguide transition and the applicator, and a manual sliding short-circuit. The method consists in placing a sample in the location of maximum electric field and measuring the shift in the resonant frequency of the cavity (Δf) caused by the insertion of a small dielectric sample inside the cavity. The change in frequency is related to the real part of the complex permittivity, ϵ_r' , and the decrease in the quality factor of the cavity (Q) is referenced to the imaginary part, ϵ_r'' .

For a rectangular cavity working in transverse electric (TE) mode the complex permittivity of the sample can be calculated using Equations 1 to 4 (Dube et al. 1988; Sheen 2007):

$$\epsilon_r' = \frac{V_C \cdot (f_C - f_S)}{2 \cdot V_S \cdot f_S} + 1$$

1

$$\epsilon_r'' = \frac{V_C}{4 \cdot V_S} \left(\frac{1}{Q_S} - \frac{1}{Q_C'} \right)$$

2

$$Q_S = \frac{f_S}{(\Delta f)}$$

3

$$Q_C' = Q_C \left[1 + (\epsilon_r' - 1) \frac{V_S}{V_C} \right]$$

4

where V_C and V_S are the volumes of the empty cavity and the sample, f_C and f_S are the resonant frequencies, Q_C and Q_S are the quality factors of the cavity without and with the sample inside the cavity, respectively.

Once the resonance frequency f_S is found, the value of Q_S is obtained by computing the ratio of f_S over the frequency band of 50% reflection of incident power below and above f_S , as demonstrated in Fig. 3 (Venkatesh 1996).

Following the impedance matching through a manual sliding short-circuit and fixed iris, the dielectric properties measurements simply consisted of filling the samples into a quartz tube of specified diameter (16 mm for the WR975 and 4 mm for the WR340 waveguide) and placing it inside the cavity; the changes in resonant frequency and transmission attenuation, in comparison with the empty cavity containing only the sample holder, were used to calculate the dielectric properties and quality factor according to Equations 1–4. At least three measurements were made for each product.

For the dielectric properties of semi-skimmed milk powder at temperatures of 50 and 60°C, the samples were put inside the respective tube (the same used for the measurements) and placed into a beaker with constantly heated water. The temperature was controlled by a thermometer (TESTO 104-IR, Testo®) and once the sample reached the desired temperature, the tube was immediately placed inside the cavity to proceed with the experiment.

3. Results And Discussion

3.1. Determination of dielectric properties by open-ended coaxial probe (OECF) and cavity perturbation technique

Table 1 shows the moisture content (% wet basis, w.b.) and water activity (a_w) of the original products, as well as the dielectric properties measured by OECP and cavity perturbation technique.

Table 1
Physical and dielectrical properties of low moisture foods at 2450 and 915 MHz measured by open-ended coaxial probe and cavity perturbation.

PRODUCT	Tapped density (g/cm ³)	Moisture content (%)	a_w	Open-ended coaxial probe		Cavity perturbation		Open-ended coaxial probe		Cavity perturbation	
				2450 MHz		2450 MHz		915 MHz		915 MHz	
				/	''	/	''	/	''	/	''
				ϵ_r	ϵ_r	ϵ_r	ϵ_r	ϵ_r	ϵ_r	ϵ_r	ϵ_r
Corn starch	0.464 ± 0.006	5.33 ± 0.000	0.124 ± 0.000	1.00 ± 0.02	0.06 ± 0.01	1.72 ± 0.00	0.36 ± 0.00	1.14 ± 0.02	0.12 ± 0.00	1.56 ± 0.13	0.12 ± 0.12
Curry	0.659 ± 0.006	6.85 ± 0.001	0.435 ± 0.002	1.23 ± 0.04	0.02 ± 0.01	1.72 ± 0.16	0.28 ± 0.05	1.37 ± 0.04	0.07 ± 0.00	1.96 ± 0.00	0.02 ± 0.00
Paprika	0.619 ± 0.001	7.19 ± 0.001	0.479 ± 0.001	1.06 ± 0.09	0.10 ± 0.04	1.72 ± 0.00	0.24 ± 0.00	1.53 ± 0.09	0.20 ± 0.04	1.96 ± 0.00	0.24 ± 0.00
Dried chives	/	9.50 ± 0.002	0.45 ± 0.002	0.70 ± 0.01	0.12 ± 0.01	1.24 ± 0.00	0.04 ± 0.05	1.34 ± 0.02	0.41 ± 0.01	1.24 ± 0.24	0.20 ± 0.07
Rice grain	/	11.09 ± 0.001	0.491 ± 0.001	2.69 ± 0.02	0.22 ± 0.00	3.17 ± 0.16	0.84 ± 0.08	2.75 ± 0.02	0.11 ± 0.00	4.36 ± 0.00	0.64 ± 0.21
Wheat grain	/	10.74 ± 0.001	0.538 ± 0.002	2.91 ± 0.09	0.50 ± 0.02	2.52 ± 0.11	0.56 ± 0.05	2.38 ± 0.05	0.53 ± 0.01	2.92 ± 0.00	0.48 ± 0.00

The dielectric properties found were close to those reported on the literature, although there are few published data for low moisture foods, particularly regarding the products that were measured in this study. In can be seen in Table 1 that products with higher moisture contents and water activity did not necessarily present higher ϵ_r' , which implies that the nature of the food (composition and physical state) is an important factor that influences the given values.

Additionally, temperature and moisture content are the most investigated factors whilst measuring the dielectric properties of food products. These properties usually drop quickly with the decrease in water content up to a critical moisture level (for example, about 12% w.b. for diced apples at 22°C). Below this level, there is little reduction in the loss factor, due to the bound water. The reduced loss factor with decreasing moisture content makes dehydrated foods less able to convert electromagnetic energy into thermal energy (Routray and Orsat 2018; Tang 2005).

Depending on the evaluated frequency (2450 or 915 MHz), the dielectric properties changed slightly (for the ϵ_r' of all products at 2450 and 915 MHz, there was a standard deviation of 0.65 for OECP and 0.92 for the cavity perturbation technique). At higher frequency, 2450 MHz, ϵ_r' and ϵ_r'' tended to decrease in comparison to 915 MHz, for most tested samples and regardless of the measurement technique. Indeed, the lower the frequency, the more charges can be set in motion and therefore polarize the medium, which is directly related to the dielectric constant.

Table 2 displays the permittivity of semi-skimmed milk powder stored at several relative humidity (resulting in different water activity). Evaluating the results of dielectric properties measured by OECP, it can be seen that the ϵ_r' of the product with 13.08% of moisture content, for example, decreased from 2.10 to 1.95 when frequency was increased from 915 to 2450 MHz. However, the values overlap considering the standard deviations, so only a behavior trend can be implied. The loss factor, likewise,

decreased from 0.26 at 915 MHz to 0.16 at 2450 MHz. Nevertheless, it seems that the dependence of the loss factor on frequency may be less predictable than that of dielectric constant (Berbert et al. 2002). Guo et al. (2008) reported that the dielectric constant and loss factor of chickpea flour, measured by OECP, were influenced by the frequency, especially at high moisture contents (15.8 and 20.9% w.b.).

Table 2

Dielectric properties of semi-skimmed milk powder at different moisture contents and water activity measured by open-ended coaxial probe at room temperature.

PRODUCT	Tapped density (g/cm ³)	Moisture content (% w.b.)	a _w	Open-ended coaxial probe 2450 MHz		Open-ended coaxial probe 915 MHz	
				'	''	'	''
				ε _r	ε _r	ε _r	ε _r
Semi-skimmed milk powder	0.612 ± 0.005	8.54 ± 0.001	0.458 ± 0.001	1.01 ± 0.04	0.07 ± 0.00	1.10 ± 0.05	0.15 ± 0.00
	0.585 ± 0.005	9.97 ± 0.003	0.654 ± 0.002	1.22 ± 0.07	0.06 ± 0.01	1.73 ± 0.08	0.11 ± 0.00
	0.570 ± 0.006	13.08 ± 0.003	0.786 ± 0.003	1.95 ± 0.08	0.16 ± 0.00	2.10 ± 0.09	0.26 ± 0.01

Ozturk et al. (2016), using a precision LCR meter and liquid test fixture to measure the dielectric properties of vegetable powders, noted smaller values of dielectric constant and loss factor with the increase in the radio frequency range (from 1 to 30 MHz), generally agreeing with the moisture content of each product. However, two of the vegetable powders, chili powder and potato starch, had higher moisture content but lower ε_r' and ε_r''. The authors pointed out that the dielectric properties are not only affected by moisture content, that is, the bulk density and particle size of the product may also impact on the permittivity. Working with red pepper powder at 10.4% w.b. moisture content and at room temperature, Guo & Zhu (2014) verified that when the frequency increased from 27.12 to 4500 MHz the dielectric constant decreased on a smaller scale, from 3.75 to 2.76. At 30.8% w.b. moisture content, the decrease in ε_r' was much more pronounced, from 35.07 to 12.53. Although this study is based on much larger frequency variations, the fact that the samples used in the present work had no more than 13% w.b. moisture content may explain the small differences in dielectric properties with the increase in frequency.

Table 3 shows the measurements of semi-skimmed milk powder at room temperature and heated up 50 and 60°C. The powder at room temperature presented a ε_r' between 1.28 to 1.72 and ε_r'' of 0.02 up to 0.16, depending on the frequency and measurement technique. At higher temperatures, both ε_r' and ε_r'' increased, but the loss factor remained low, especially when OECP was used. For skimmed milk powder at frequencies above 200 MHz up to 3 GHz and using the probe, Lau et al. (2020) reported values of ε_r' from 2.5 and 3.5 and ε_r'' between 0 and 0.5, for temperatures from 20 to 60°C, respectively. An increase in the dielectric constant with the increase in temperature may be attributed to the operating frequency, the ratio between bound-water and free water contents, and the raise of ionic conduction and mobility of ions (Calay et al. 1995; Stuart O. Nelson and Bartley 2002). The changes in permittivity of semi-skimmed milk powder were relatively small, as well as noticed by Guo et al. (2008) for chickpea flour at temperatures below 40°C. Nevertheless, the effect of increasing frequency on the decrease of permittivity was much more pronounced for the powder at higher temperatures than at room temperature. Dependence of dielectric properties on frequency, temperature and moisture content is very common in the literature and has also been reported by other authors (Boreddy and Subbiah 2016; Li et al. 2018; Y. Lin et al. 2016; Ozturk et al. 2016, 2018; Qi et al. 2021; Zhu et al. 2012).

Table 3

Dielectric properties of semi-skimmed milk powder at different temperatures and measured by open-ended coaxial probe and cavity perturbation.

PRODUCT	Tapped density (g/cm ³)	Moisture content (%, w.b.)	a _w	T (°C)	Open-ended coaxial probe		Cavity perturbation		Open-ended coaxial probe		Cavity perturbation	
					2450 MHz		2450 MHz		915 MHz		915 MHz	
					'	''	'	''	'	''	'	''
					ϵ_r'	ϵ_r''	ϵ_r'	ϵ_r''	ϵ_r'	ϵ_r''	ϵ_r'	ϵ_r''
Semi-skimmed milk powder	0.582 ± 0.001	7.19 ± 0.001	0.339 ± 0.002	Room temperature	1.28 ± 0.06	0.02 ± 0.01	1.72 ± 0.00	0.16 ± 0.05	1.45 ± 0.10	0.02 ± 0.01	1.49 ± 0.00	0.16 ± 0.00
				50.2 ± 0.13	2.05 ± 0.01	0.08 ± 0.00	1.73 ± 0.00	0.32 ± 0.05	2.04 ± 0.01	0.03 ± 0.00	2.12 ± 0.21	0.32 ± 0.11
				60.03 ± 0.04	2.27 ± 0.01	0.08 ± 0.00	1.89 ± 0.11	0.37 ± 0.00	2.30 ± 0.01	0.08 ± 0.00	2.20 ± 0.24	0.24 ± 0.00

Comparing the same frequency and both tested measurement techniques, it is noticeable that the use of the cavity perturbation technique resulted in higher dielectric constant and loss factor for almost all products. The loss factor values obtained with OECP, in particular, were closer to zero for most products. During the tests, some of the repetitions gave negative results for the ϵ_r'' and therefore were disregarded. This may be due to inaccuracies related to a bad contact between the probe and the surface of the samples and the disturbances when the device filled with sample was connected to the measurement system. Also, all the products showed oscillations in the measured values with the shift in frequency (as represented by Fig. 4 for the semi-skimmed powder at different moisture contents). This behavior was noticed by published studies that worked with low moisture foods, such as egg white powder (Boreddy and Subbiah 2016), whey protein gel and mashed potato (Chen et al. 2013), chestnut flour (Zhu et al. 2012), soy flour (Terigar et al. 2010), red pepper powder (Wenchuan Guo and Zhu 2014), legume flour (Wenchuan Guo et al. 2010). Because of the extreme low values, the signal-to-noise ratio was low. Similar noise in the dielectric properties data were reported in the literature for various other products. There are possible sources of error that could happen even after calibration and that could cause these oscillations and affect the accuracy of the measurement, such as cable stability, air gaps, and sample thickness (Chen et al. 2013). Additionally, the value of ϵ_r' for dried chives at 2450 MHz, for example, was aberrant, as it was smaller than the value for air (equal to 1). This may be proof of the limitation of OECP measurement method, that is, it highlights the range of values that this technique is capable of measuring accurately.

During measurements by cavity perturbation, the standard deviations in the dielectric properties of some products were zero. This means that when the same sample from each replicate was inserted into the cavity, the change in resonance frequency with respect to the resonance frequency of the empty cavity frequency was the same, leading to the same ϵ_r' . This observation could mean that more accurate and repeatable results can be obtained for the dielectric constant when the cavity perturbation technique is employed, but it could also imply that the execution of the cavity perturbation technique was not accurate enough to this type of product (which would result in the same erroneous value for the dielectric constant). Nevertheless, the results obtained by cavity perturbation gave coherent values, even if the comparison with the literature is not possible for the exact same foods. So, the hypothesis that the cavity gives accurate results seem more plausible.

Concerning the use of OECP or cavity perturbation, the most suitable measurement technique for a certain product depends on the frequency of interest, the nature (physical and electrical) of the dielectric material to be measured, and the degree of accuracy required (S.O. Nelson 2006). Lau et al. (2020) affirmed that the open-ended coaxial probe may have reduced accuracy for measurements below 200 MHz and for materials with low values for dielectric constant and loss factor. Table 4 shows the results found in the literature for the dielectric properties of corn starch, curry, paprika, semi-skimmed milk powder, dried chives, rice grain, and wheat grain.

Table 4

Scientific articles found in the literature reporting the dielectric properties of the same products that were used in this work.

PRODUCT	LITERATURE
Corn starch^a	Technique: OECF ¹ Frequency: 2450 MHz Sample conditions: 30°C and 1% MC ² / $\epsilon_r = 2.74$ ” $\epsilon_r = 0.14$
Curry^b	Technique: LCR meter and liquid test fixture Frequency: 1–30 MHz (radio frequency) Sample conditions: 23°C and 8.3% MC / ϵ_r between 2–3 ” ϵ_r between 0-0.3
Paprika^b	Technique: LCR meter and liquid test fixture Frequency: 1–30 MHz (radio frequency) Sample conditions: 23°C and 12.3% MC / ϵ_r between 4 to 5 ” ϵ_r between 0 to 0.3
Semi-skimmed milk powder^c	Technique: OECF ¹ Frequency: 1 MHz to 3 GHz Sample conditions: 20°C and 3.65% MC / ϵ_r around 2.2 ” ϵ_r close to 0
Dried chives	Not found

PRODUCT	LITERATURE	
Rice grain ^{de}	Technique: X-band and K-band MW measurement system Frequency: 11 and 22 GHz Sample conditions: 24°C, 11.5% MC ² Density = 0.443 g/cm ³ / $\epsilon_r = 1.76$ ” $\epsilon_r = 0.10$	Technique: coaxial air-line system Frequency: 2450 MHz Sample conditions: 24°C, 11% MC ² / ϵ_r around 2.2 ” ϵ_r around 0.25
Wheat grain ^f	Technique: power signal generator Frequency: 2450 MHz Sample conditions: bulk density of 0.6 to 0.9 g/cm ³ , 10.4% MC ² / $\epsilon_r = 2.55-2.90$ (depending on the bulk density) ” $\epsilon_r = 0.26-0.30$ (depending on the bulk density)	Technique: power signal generator Frequency: 2450 MHz Sample conditions: bulk density of 0.6 to 0.9 g/cm ³ , 10.4% MC ² / $\epsilon_r = 2.55-2.90$ (depending on the bulk density) ” $\epsilon_r = 0.26-0.30$ (depending on the bulk density)
^a (Ndife et al. 1998)		
^b (Ozturk et al. 2018)		
^c (Lau et al. 2020)		
^d (You and Nelson 1988)		
^e (Noh and Nelson 1989)		
^f (Sokhansanj and Nelson 1988)		
¹ OECP = open-ended coaxial probe		
² MC = moisture content		

Errors in the measurement of the dielectric constant through cavity perturbation technique are mostly associated with inaccuracies in determining of the frequency shift and volume ratio. If these values are properly identified, the measurement error of the dielectric constant can be kept close to 2%. For the measurements of this study, occasionally there were no frequencies corresponding to a S11 value of 50% reflection of incident power (or -3 db), and in this case the corresponding frequency values closest to the 50% reflection were chosen. This decision caused a difference in the calculated loss factor, estimated in around 0.18 for paprika powder (above or below the reported loss value). The precision of loss factor values is constrained by the frequency shift error and especially the quality factor change error, being very difficult to control. This quality factor change error deteriorates the measurement accuracy of dielectric loss because of the combination of various factors, such as homogeneity of the specimen, coupling condition, specimen holes, and uncertainty. This inaccuracy is extremely hard to estimate. The variation

of data reported by the literature is very wide (Risman & Bengtsson, 1971; Dube et al., 1988; Sheen, 2005). The dielectric loss factors for vegetable powders, for instance, were very low due to the small amount of water in these products. For broccoli powder and onion powder, the ϵ_1'' values were not shown by the authors because they were too low, and the instrument could not give stable values (Ozturk et al. 2016).

According to Sheen (2009), the cavity perturbation technique is good for measurement of the real part of permittivity but is not adequate for extremely low loss factors. Moreover, P. Risman, (2009) reported that a maximum of 2 mm in diameter is necessary for lossy materials at 2450 MHz and using a TM_{010} circular cavity, otherwise the quality factor will be too low or the attenuation in transmission will be too high, reducing the resolution and allowing weak non-modal field signs to interfere in the main mode. The experimental approach of the present work was to abstract these considerations, and the appropriate diameter of the tube was chosen based on the consideration of minimum perturbation inside the cavity.

4. Conclusions

It is well known that one of the major limitations to the application of microwave and radiofrequency technologies on an industrial scale is the non-uniform heating. The determination of dielectric properties of food materials is the first step to evaluate the heating behavior according to each type of product, in order to improve homogeneity and quality of food, develop new products and processes, enhance microbial decontamination (and prevent bacterial growth), disinfestation, and in-pack pasteurization, in addition to the optimization and increase of process lines.

Dielectric properties data on foods have been published by many authors over the years. However, foods are complex materials in terms of physical state, composition and consequently in their dielectric behavior. Thus, it is usually necessary to measure the permittivity under the conditions of interest to obtain reliable data.

Several methods allow the measurement of dielectric properties of low loss materials, however, not all of them take into account the effects introduced by a real measurement system. These effects include noise, crosstalk, coupling losses, transmission line delay and impedance mismatch, and not considering them may lead to significant uncertainty in the measured Q-factor.

By observing the results reported by this study, it is evident that the dielectric properties vary with changing frequency and increase along with moisture content and temperature. The dielectric properties of the powders and grains were determined by the OECP, which is more appropriate for homogeneous liquids and soft semi-solids, and by the cavity perturbation technique using a waveguide not adapted for these products. This may explain possible inaccuracies in the measurements. However, it is not possible to recognize errors neither confirm the accuracy of the results since there are not enough data in the literature to allow comparison with the collected data.

Based on the performances of both methods on the tested food samples, the cavity perturbation technique is advantageous at measuring the dielectric properties of products when the magnitudes of the losses are expected to be low. In addition, the deviation of the OECP method at higher temperatures may demand additional procedures, such as the multi-temperature calibration suggested by Chen et al. (2013). The most suitable measurement technique for a food depends also on the frequency of interest, the physical and electrical characteristics of the dielectric material and the degree of accuracy required.

Declarations

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Conflict of Interest Statement

The authors certify that they have no conflict of interest in the subject matter or materials neither personal relationship that could have appeared to influence the work reported in this paper.

Data Availability Statement

The authors confirm that the data supporting the findings of this study are available within the article or its supplementary materials.

Authors Contributions Statement

Sébastien Curet, Olivier Rouaud, Lionel Boillereaux, and Alexandre Thillier conceived the presented idea and wrote the research project.

Sébastien Curet, Olivier Rouaud and Lionel Boillereaux participated in the interpretation of the results obtained with the open-ended coaxial probe at Oniris (Nantes, France).

Alexandre Thillier bought the food products used for the tests at SAIREM (Décines-Charpieu, France).

Ana Caroline Cichella Frabetti assembled the instruments for the cavity perturbation technique and carried out the experiments with support from Hugo Curto, Tristan Garnault and Sébastien Curet.

Hugo Curto and Tristan Garnault helped in carrying out the calculations of dielectric properties with the cavity perturbation technique.

Ana Caroline Cichella Frabetti wrote the main manuscript text.

All authors supervised the findings of this work, discussed the results and contributed to the final manuscript.

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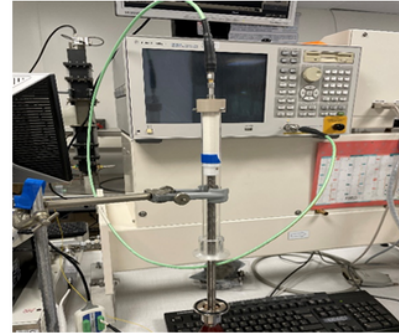
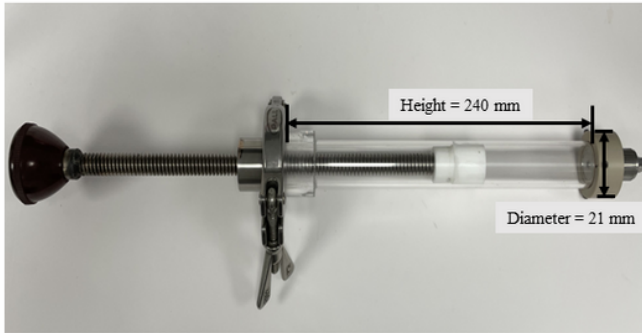
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Figures

(a)



(b)

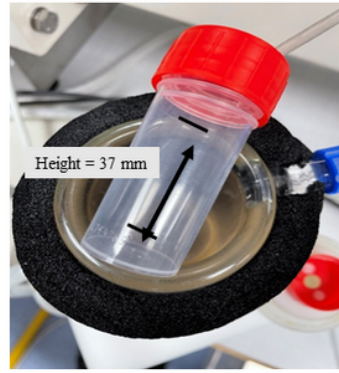
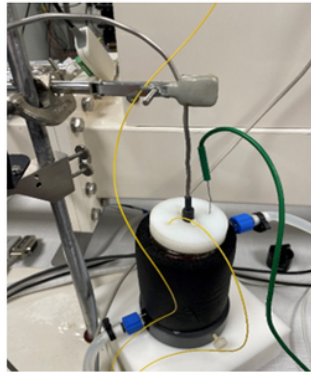


Figure 1

(a) Custom-designed device that allowed greater contact between the food powders and the open-ended coaxial probe (OECF);
(b) Experimental device used for OECF measurements of semi-skimmed milk powder at 50 and 60 °C

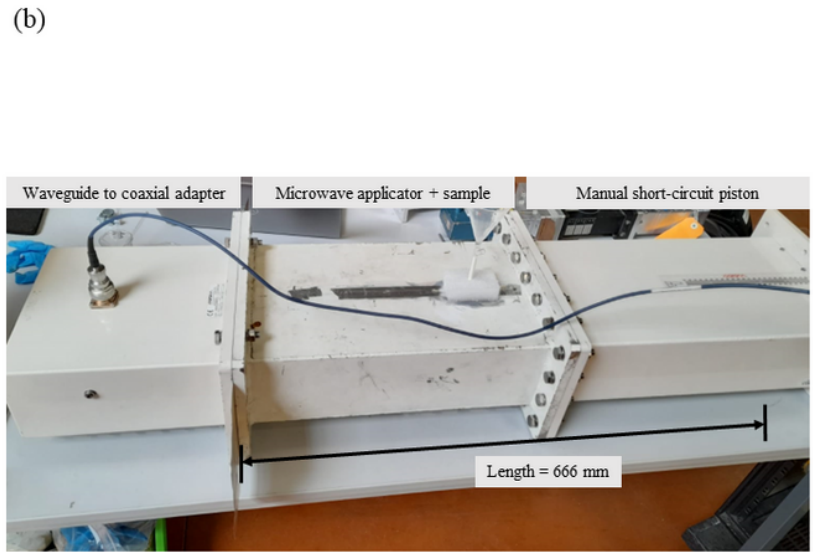
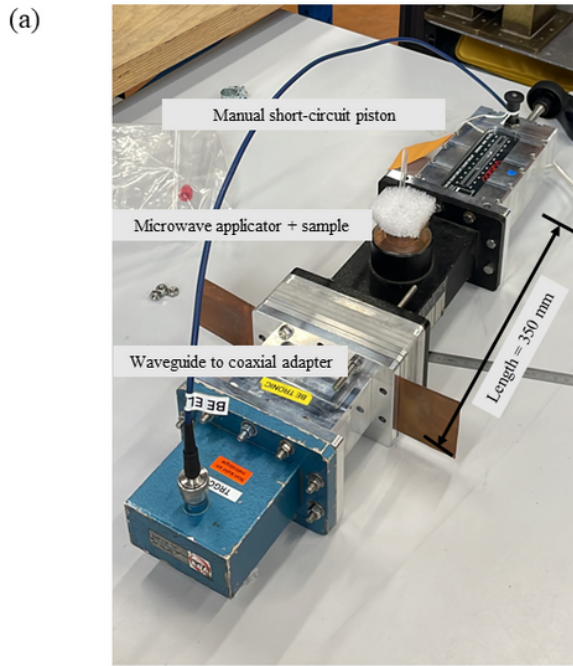


Figure 2

(a) WR340 operating at 2400-2500 MHz and (b) WR975 operating at 900-930 MHz waveguides used to perform the cavity perturbation technique

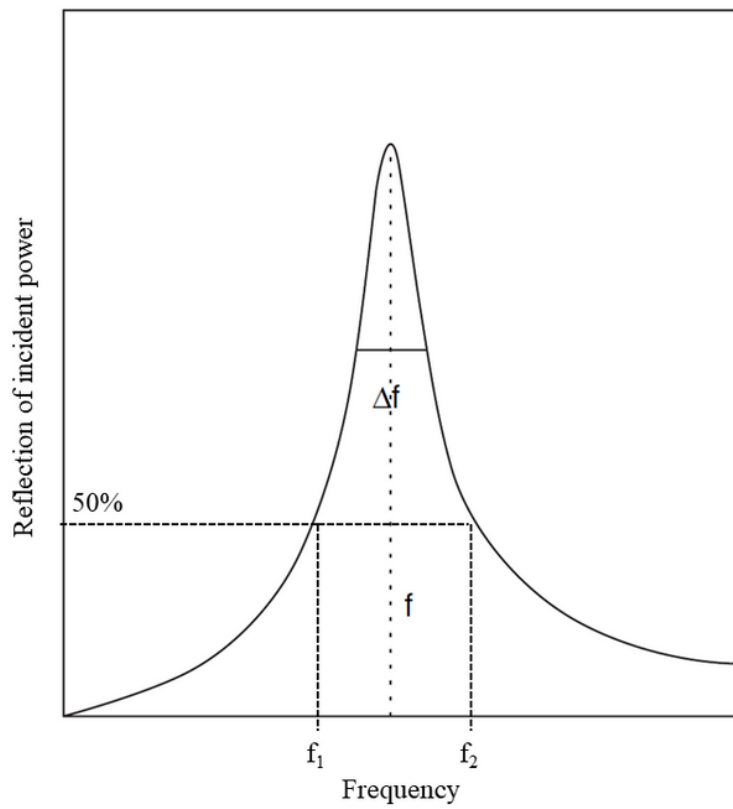


Figure 3

Resonant peak obtained by cavity perturbation method and related parameters to calculate the dielectric properties of a sample

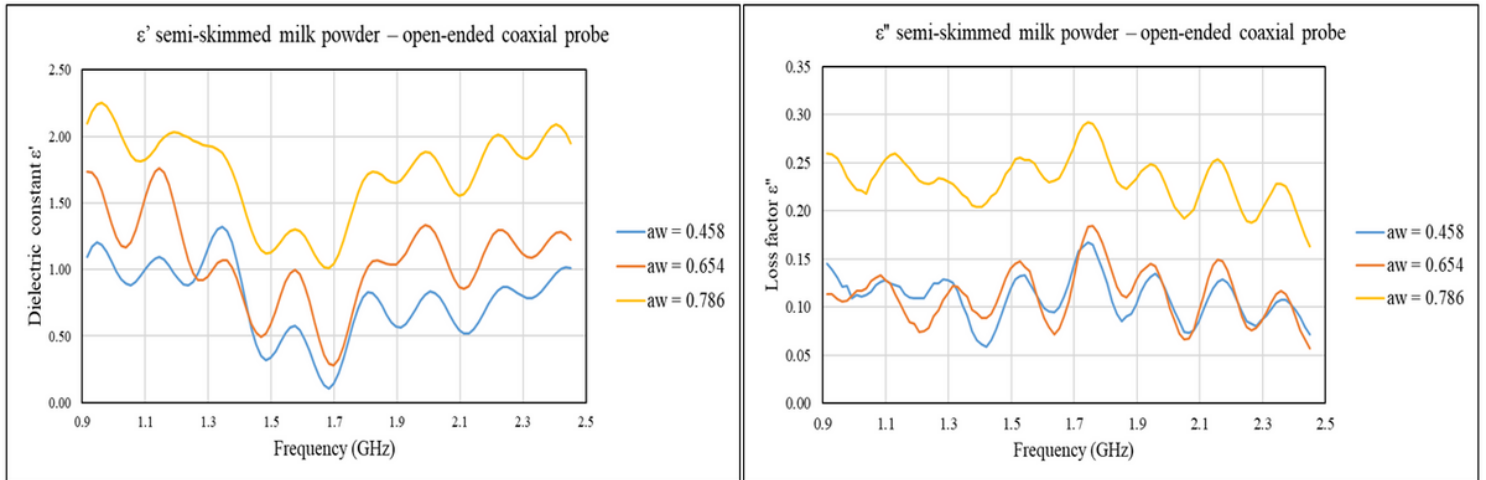


Figure 4

Oscillations observed during the measurements of semi-skimmed milk powder with open-ended coaxial probe