

ULTRA-BROADBAND CHARACTERISATION SYSTEM FOR THE DIELECTRIC PROPERTIES OF FOOD MATERIALS

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Abstract: The understanding of the dielectric properties of food material is important for electromagnetic-based treatments from reheating in a domestic microwave oven to sterilization processes. This is due to aspects such as the uniformity and rate of heating of the food being highly dependent upon the dielectric constant and dielectric loss of the food material, with both parameters being intrinsically temperature-dependent. In this work, a method for ultra-broadband (1 Hz to 1.6 GHz) characterisation of food materials is described. The ease of use of this system allows for rapid testing of a food standard – in this case, instant mashed potato – with various combinations of moisture and salt content. In addition to the ultra-broadband measurements, the system also allows for automated temperature sweeps (-100°C to 300°C). By obtaining these ultra-broadband datasets, additional understanding can be gained of the mechanisms behind the variation of the dielectric properties. One measurement system provides dielectric data covering very different heating applications: – e.g. microwave (0.4 – 5.8 GHz), radio frequency (1 – 100 MHz), ohmic (50 Hz) – as well as measurement and imaging applications.

The development and test of a dielectric measurement system is preferably conducted via the use of a standard of known properties as a calibration. Given the interest here is food, then instant mashed potato is an ideal material as key parameters can be readily manipulated (moisture, salt) which along with temperature allow measurement of repeatability and consistency; and relative ease of measurement. The heating of a food material via electromagnetic waves is controlled by its dielectric properties. The permittivity of a material is expressed by

$$\epsilon^* = \epsilon' - \epsilon'' \quad (1)$$

where ϵ' is the dielectric constant and ϵ'' is the dielectric loss. The dielectric constant reflects the material's ability to store electromagnetic energy, which relates to the penetration depth of the material,

$$D_p \approx \frac{4.9\sqrt{\epsilon'}}{f\epsilon''} \quad (2)$$

and the material wavelength,

$$\lambda_m = \frac{c}{f\sqrt{\epsilon'}} \quad (3)$$

where f is the frequency and c is the speed of light (in a vacuum). The penetration depth controls the uniformity of heating within the material, whilst the material wavelength partially dictates the distance between hot and cold spots. The presence of hot and cold spots can also be affected by the shape and size of the food material. The dielectric loss measures

a material's ability to dissipate electric energy as heat, therefore controlling the heating rate of the material.

Due to the importance of understanding these properties for electromagnetic-based food preparation and sterilization processes, there has been a vast amount of published research concerning measurement of the dielectric properties of various food materials [1-6]. A food standard (or model food) is desirable as it would result in a relatively flexible control over the moisture, salt and sugar content. Mashed potato is commonly used as a food standard due to it being relatively homogenous once mixed, with its properties being easily modified by varying its moisture and salt content to encompass the dielectric properties of many food materials [7]. By using instant potato powder, the parameters – moisture and salt content – of the mixture can easily be controlled. It is worth noting that Smash® has a base salt content of 0.17% by weight (Smash®, Premier Foods). Using conventional methods (for example, an open-ended coaxial dielectric probe) for determining the dielectric properties of foodstuff as a function of temperature is a relatively laborious task, typically restricted to a relatively small bandwidth [8, 9].

In this work, the dielectric properties of mashed potato were investigated for a range of temperatures over an ultra-wide frequency band. The measurements were conducted using a bespoke broadband dielectric spectrometer. This all-in-one system allows automated temperature measurements over two broad frequency ranges, the first being 1 Hz to 0.1 MHz and the second ranging from 1 MHz to 1.6 GHz. The foodstuff samples were confined within a cryostat for high-precision temperature control, ranging from 0°C to 80°C. Previous work in the field has focused on the dielectric properties of instant mashed potato (prepared to packet instructions – moisture content near 80%) [8]. It was shown that the dielectric properties of the mashed potato strongly depend on the temperature and the measurement frequency, especially at radio frequency, industrial, scientific and medical (RF ISM) bands (< 100 MHz). Interestingly, the authors of [8] concluded that there were no significant effects on the dielectric constant due to temperature, salt, and moisture at microwave frequencies (> 0.4 GHz). However, the results indicate a notable change in properties, for example, at 1.8 GHz the dielectric constant varied: 13.7% with a temperature sweep from 20°C and 120°C; 11.0% with an increasing moisture content (81.6% to 87.8%); and 9.7% with an increasing moisture content (0.8% to 2.8%) [8].

The conventional method used for characterising dielectric properties of liquid and liquid-like materials is the open-ended coaxial probe. In this method the probe is completely inserted into the material and the surface dielectric response is measured over a typical range of 0.2 GHz to 20 GHz at a high precision. However, this method is not without its limitations: measurement variations are possible due to the pressure forcing water from the sample, and the possibility of air bubbles forming on the end of the probe. Furthermore, for temperature measurements the process is time-consuming as the system can only measure at a single temperature in any given run.

The work presented in this paper focusses on the Novocontrol dielectric broadband spectrometer (Fig. 1a). This device measures the impedance spectrum $Z^*(f)$ of a sample material placed between two electrodes. The bulk intrinsic electric material's properties such as the complex permittivity,

$$\epsilon^*(f) = \frac{C^*(f)}{C_0}, \quad (4)$$

are evaluated from the complex sample capacity,

$$C^*(f) = \frac{1}{i2\pi f Z^*(f)}, \quad (5)$$

$$C_0 = \frac{\epsilon_0 A}{d}, \quad (6)$$

where A is the electrode area and d is the sample thickness. These measurements can be conducted automatically over a ultra-broadband frequency range: 1 Hz up to 1 GHz (9 decades).

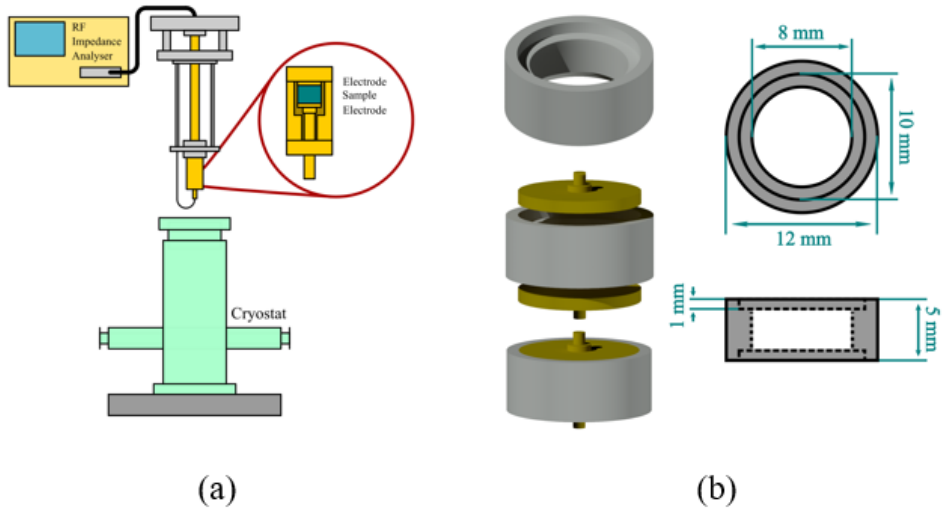


Fig. 1. (a) Diagram of the Novocontrol dielectric broadband spectrometer. (b). CAD diagram of the liquid sample holder (with brass electrodes).

To accommodate the liquid-like structure of the instant mashed potato, a sample holder was designed and fabricated from PTFE. The sample holder was built to allow for a sample volume of approximately 150 mm³. The holder's presence was calibrated out from the measurements by measuring the empty pod and subtracting the capacity of air (0.146 pF), so as to avoid affecting the measured food material's response. Once filled with the instant mashed potato, the food material moulds to the electrodes, minimising air bubbles at the surfaces.

In this work, various mixtures were investigated to explore the effects of salt and moisture content. The samples were produced by mixing different proportions of the components (by weight). The instant mashed potato powder was added to deionised water and mixed for a minimum of 30 seconds or until homogeneous. If the sample had added salt, this was added to the water and dissolved before mixing with the instant mashed potato powder. To ensure consistency over the measurements, the samples were weighed once they were placed inside the sample holder, to ensure a sample mass of 0.160 g. Table 1 shows the investigated mixture's water activity, three versions of each mixture were created: (1) 0% added salt, (2) 1% added salt, and (3) 2% added salt (by weight). The water activity was measured using a commercial water activity meter. The water activity is the amount of unbound water in a food sample.

Table 1. Table of the investigated Water:Potato mixtures. All data shown is at laboratory room temperature (21.9°C).

Water:Potato	Water Activity (± 0.01)
50:50	0.99
60:40	0.88
70:30	0.86
80:20	0.87

The initial investigation focused on the 1 MHz to 1 GHz band at room temperature, each data point was averaged over 20 measurements in a single automated sweep. Figure 2 shows the measured dielectric constant for all produced samples. When the 0% added salt samples are compared between each mixture – black curves – the moisture content has no significant effect on the dielectric constant over this frequency range. At high frequencies (>10 MHz), the salt content similarly has little effect on the dielectric properties; however, across all mixtures the salt content has a large impact on the dielectric constant at lower frequencies (<10 MHz). A comparison of the response at 1 MHz shows a drastic change with salt content: for example, a 50:50 mixture with 0% salt (black curve) has a dielectric constant of ~ 100 compared to a value of ~ 180 for 2% salt (red curve).

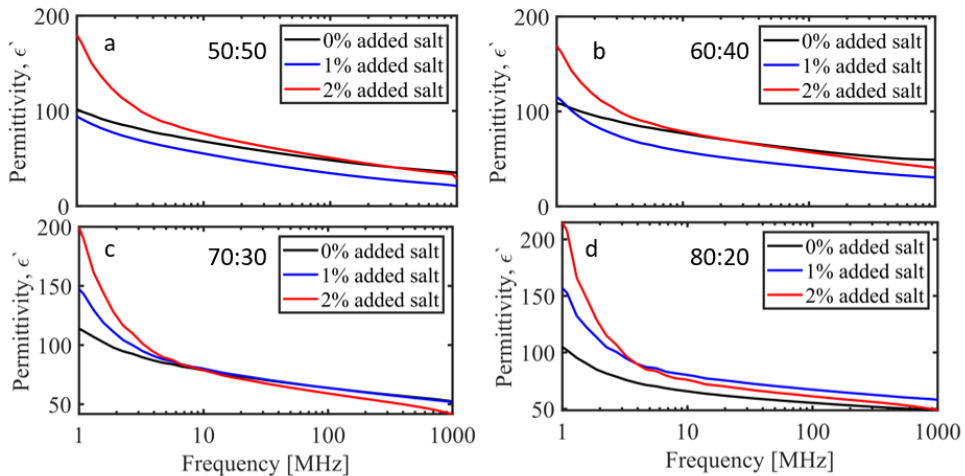


Fig. 2. Measured dielectric constant between 1 MHz and 1 GHz for various mashed potato moisture mixtures: (a) 50:50, (b) 60:40, (c) 70:30 and (d) 80:20. The salt content values investigated were 0% added salt (black curve), 1% added salt (blue curve), and 2% added salt (red curve).

For some of the curves the dielectric constant is lower for increased salt content (frequencies >10 MHz). This could be due to shielding effects from the salt ions. The sodium and chloride ions dissociate in the solution, resulting in an electric field between them which the polar water molecules tend to align with. This creates an effective shielding and lowering the water molecule's response to the external field. This results in a reduction in the dielectric constant [10]. This is also known as dielectric decrement. This behavior can be seen when investigating the dielectric properties of water and saline solutions by using an open-ended dielectric probe (Figure 3).

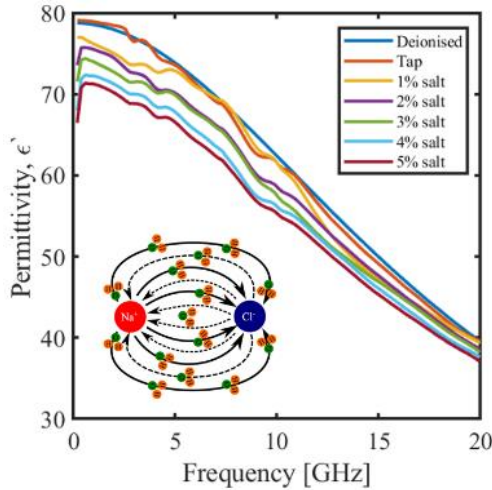


Fig. 3. The dielectric constant of deionised water, tap water and saline solutions from 1% salt to 5% salt. Inset: A schematic to show the orientation and dissociation of ions and molecules of the solutions under the influence of an external field.

Figure 4 shows the dielectric loss for the mixtures previously shown in Table 1 and Figure 2, at room temperature. Once again, the salt content has a drastic impact on the dielectric loss at RF and lower frequencies. Such a response is expected as salts – or, more specifically, dissolved ions – will increase the overall conductivity of a mixture, resulting in an increase in the dielectric loss.

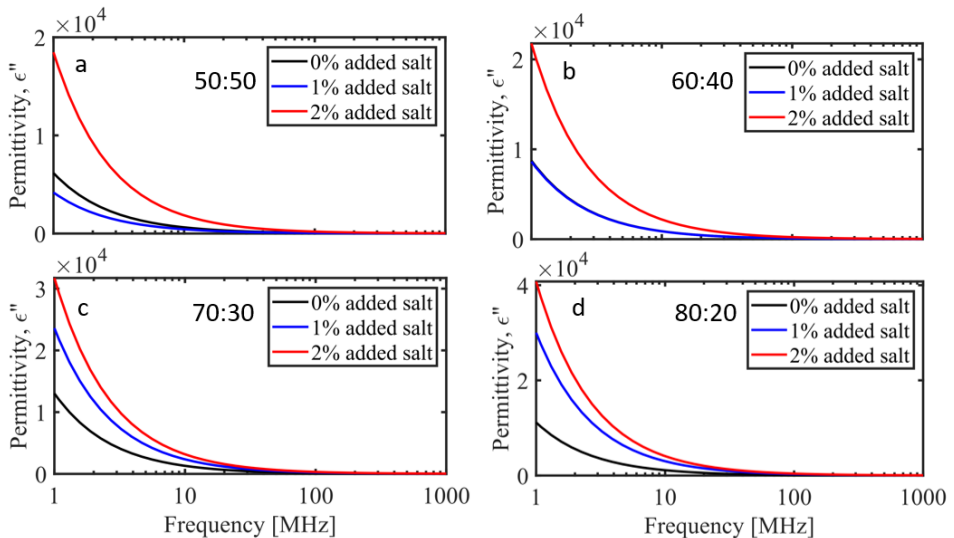


Fig. 4. Measured dielectric loss between 1 MHz to 1 GHz for various mashed potato moisture mixtures (a) 50:50, (b) 60:40, (c) 70:30, and (d) 80:20. The salt contents investigated were 0% added salt (black curve), 1% added salt (blue curve) and 2% added salt (red curve).

One of the key advances of our method is the automated temperature sweeps. One of the main parameters to understand when it comes to the dielectric properties of food materials is the temperature dependence. An understanding of the effect of temperature on the dielectric constant and loss is not only important for dielectric heating applications, but also food sterilization (130°C) and frozen food processes (temperatures down to -40°C). To regulate the temperature during the sweep, a temperature probe is inserted into the sample cell. The whole RF extension line is inserted into the cryostat (Fig. 1a) where the temperature is externally controlled by software. The sample will automatically be brought to the desired temperature before the frequency sweep is measured – with the sample held at that temperature.

Figure 5 shows the dielectric constant and dielectric loss for two mixtures: 50:50 and 70:30 for a temperature range from 0°C to 80°C. The dielectric constant is relatively constant with temperature at microwave frequencies; however, there is a sharp increase at radio frequencies. The dielectric loss gradually increases with increasing temperature.

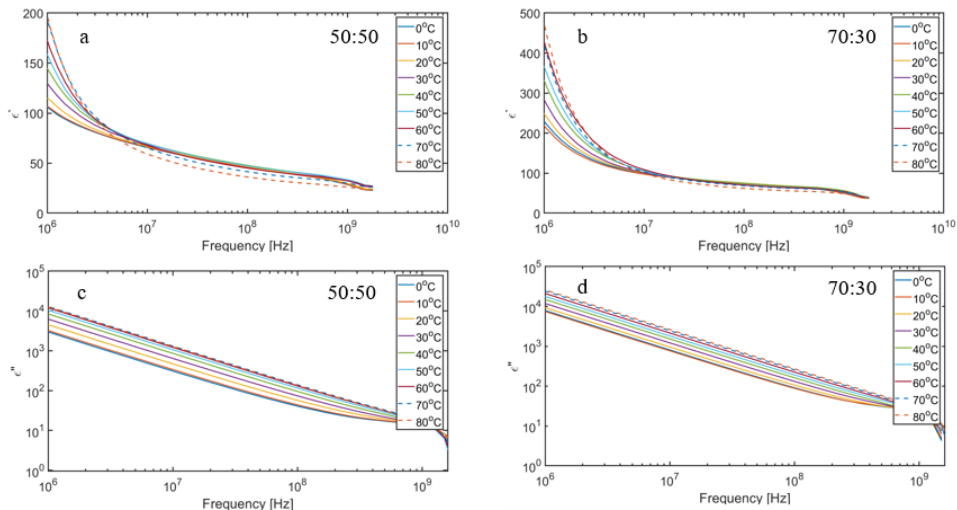


Fig 5. Measured dielectric constant between 1 MHz and 1 GHz for (a) 50:50, and (b) 70:30 potato mixtures. Measured dielectric loss between 1 MHz and 1 GHz for (c) 50:50, and (d) 70:30 potato mixtures.

Using this method, the dielectric properties can be measured at the relatively unexplored low frequencies (1 Hz to 1 MHz). Figure 6 shows both the previous data (1 MHz to 1 GHz), as well as the low frequency measurements for mixtures of 50:50 and 80:20 for 0%, 1%, and 2% added salt. The enormous values (10^9) seen at the lowest frequencies (< kHz) that disagree with Debye-type processes could be the result of a few different effects. It has been suggested that the water molecules can auto-dissociate to form ion pairs at the low frequencies where the Maxwell-Wagner-Sillars (MWS) phenomenon is dominant [11-13]. For higher frequencies, only the water dipoles can respond to the polarity changes, so the dielectric constant follows Maxwell-Boltzmann statistics. It is thought that the MWS effect is driven by charge carriers being blocked at inner dielectric boundaries on a mesoscopic scale. This can result in a separation of charges which gives rise to an additional polarisation contribution. For dielectric spectrometer based measurements that use an upper and lower electrode, low frequency measurements can often lead to electrode polarisation effects (measurement artefacts), associated with charge transport between the two plates on timescales similar to the lower frequencies of measurement. However, for instant mashed

potato it is also feasible that there may be MWS polarisation processes associated with the charge transport around them.

The phenomenon known as electrode polarisation can present a major effect on low frequency measurements. Due to this, various approaches have been proposed to overcome the effective masking of the material of interest. Proposed correction techniques include: electrode distance variation technique, four-electrode technique, as well as, comparison and substitution methods [14-15]. To further test the effects at play – and to gain the true values of the dielectric properties – various investigations into electrode separation and sample sizes would need to be conducted, in the hope of isolating any effects due to electrode polarisation effects (that would be creating measurements artefacts). Qualitatively comparing the values for the mixtures (and deionised water), there is an increase in the dielectric constant between deionised water and instant mashed potato samples. There is also an increase with salt content; however, it is shown again that the dielectric constant is relatively constant with moisture content.

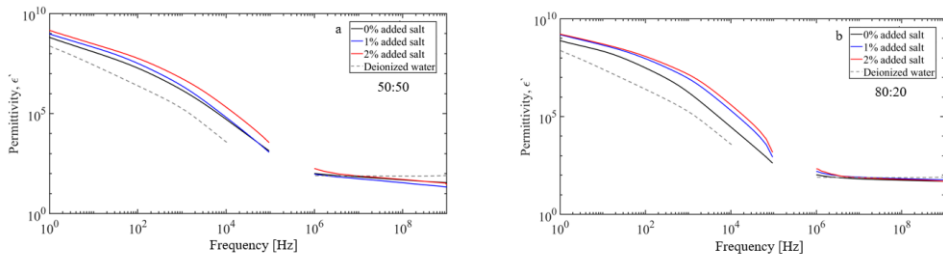


Fig. 6. Measured dielectric constant between 1 Hz and 1 GHz for various mashed potato moisture mixtures: (a) 50:50, and (b) 80:20. The salt contents investigated were 0% added salt (black curve), 1% added salt (blue curve), and 2% added salt (red curve). Deionised water is shown as the grey dashed line.

Figure 7 shows both the previous data (1 MHz to 1 GHz), as well as the low frequency measurements for mixtures of 50:50 and 80:20 for 0%, 1%, and 2% added salt. Once again, there is a gradual increase in the dielectric loss with salt content (matching with the high frequency data). This data also further proves that the dielectric properties are relatively constant with moisture content.

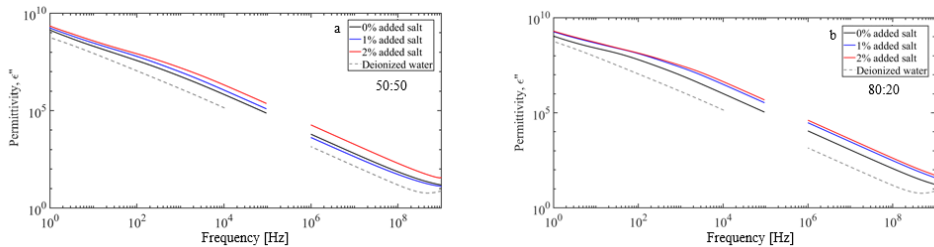


Fig. 7. Measured dielectric constant between 1 Hz and 1 GHz for various mashed potato moisture mixtures: (a) 50:50, and (b) 80:20. The salt contents investigated were 0% added salt (black curve), 1% added salt (blue curve), and 2% added salt (red curve). Deionised water is shown as the grey dashed line.

In the presented work, a method for ultra-broadband (1 Hz to 1.6 GHz) characterisation of food materials is described. The ease of use of this system allows for rapid temperature-dependent testing of a food standard – instant mashed potato – with

various combinations of moisture and salt content. It was shown that the dielectric constant is relatively constant at microwave frequencies, but sharply increases into radio and very low frequencies. The extremely large values seen are believed to be due to a combination of the MWS effect and electrode polarisation effects. The presence of these effects result in an increase in the dielectric properties, which are orders of magnitude larger than expected from the Debye model. As the frequency is increased the measured dielectric response becomes comparable to the Debye model predictions (dominated by molecular fluctuations).

Interestingly, it was shown that the moisture content seemed to have little influence on the dielectric response over the investigated range. The salt content had a small effect on the dielectric constant, with the exception of a sharp increase at low frequency (< 10 MHz). However, the dielectric loss was shown to have a drastic dependence on the salt content, due to the overall increase in the conductivity of the sample. Finally, it was shown that the temperature dependence of the dielectric constant was relatively constant at microwave frequencies, but sharply increases into radio frequencies. The dielectric loss gradually increases with temperature.

The next stages for this work will be to improve the measurements at lower temperatures, allowing samples to be taken down to -100°C, enabling an investigation into the dielectric properties of frozen food materials over the ultra-wide band of 1 Hz to 1 GHz. This would provide interesting and useful information as, at commercial freezing temperatures (down at -40°C), the system is not static. Another option would be to extend the upper temperature investigated to 130°C as this temperature is important for the food sterilisation process and there may be useful information that arises across the ultra-wide band measurement. The final extension would be to explore the effects that occur at low frequency, finding evidence for ion pair resonances and Maxwell-Wagner-Sillars effects. It would also be important to isolate any effects that could be occurring due to electrode polarisation effects, by varying the volumes of the samples.

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