Techniques for Temperature-Dependent Dielectric Measurements: a Review

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Abstract—**In this paper, a preliminary review of the dielectric measurement methods as a function of both frequency and temperature is presented. In particular, the two most popular approaches for heating the samples up to ablative temperatures (i.e., water bath and antenna applicator) and two dielectric measurement setups (i.e. open-ended coaxial probe and resonant cavity) are analysed and compared, highlighting the pros and cons of each approach.**

Keywords—Frequency- and temperature-dependent dielectric properties, heating methods, experimental setup, dielectric measurements

I. INTRODUCTION

Exact knowledge of the dielectric properties of tissues is of fundamental importance for the design of all devices involving electromagnetic (EM) fields. Dielectric properties describe the interaction between the EM fields and the investigated tissue, and are strongly dependent on frequency and temperature. In addition to the standard microwave-based technologies, dielectric spectroscopy is also used to study the dynamics and composition of complex materials in the food industry [1, 2], as well as in material engineering [3]. In these areas, various measurement setups have been proposed and used, not only to characterise samples (solid, semi-solid, liquid and powder) in frequencies from a few kHz to tens of GHz, but also in a very broad range of temperatures (from cryogenic to ablative temperatures).

An important application of electromagnetics, in particular microwaves, that is of increasing interest in the scientific community is that of applying EM fields for biomedical applications, for both diagnostic and therapeutic purposes [4, 5], especially in oncology. This requires an accurate and unambiguous knowledge of the dielectric properties of biological tissues of interest as a function of frequency and temperature. Several campaigns for the dielectric characterisation of biological tissues have been proposed in recent years up to 50 GHz [6-8]; however, the results presented by the various research groups have not always been in agreement, highlighting the need to standardise measurement protocols of tissue dielectric characteristics. In this regard, the Electromagnetic Research Group (EMRG) at the University of Malta, of which some of the authors of this contribution are members, is working on best-practices guidelines for dielectric measurements as a consensus among the international dielectric community, leading the COST Action MyWave (CA17115). This led to a deep investigation of the impact of confounders as hydration on the dielectric measurement data [9, 10], also providing well-defined guidelines for dielectric measurements at room temperatures. However, the procedures for a rigorous dielectric characterisation of tissues as a function of temperature, as well as on the quantitative analysis of the impact of confounders such as blood perfusion and heat deposition rate in the tissue on the measured dielectric properties are still unclear. In this paper, an overview of the techniques for temperature-dependent dielectric characterisation of materials is presented. The methods used to heat materials as well as those used to measure the dielectric properties in these studies are reviewed, discussing the pros and cons of each of the techniques presented. In particular, this article is divided into as follows. In section II, measurement methods for high temperature, together with methods to heat samples, as well as a brief introduction to data representation is done. In Section III, some discussion about the pros and cons of each approach is presented; then, some conclusions are derived.

II. MATERIAL AND METHODS

A. Heating methods

The need to investigate temperature-dependent dielectric properties has led to the use of various heating methods, depending on the desired temperature, the type of application and the type of Material Under Test (MUT). These methods include the application of a water jacket surrounding the material, the use of copper templates, the application of industrial furnaces or convectional ovens and microwave ovens. In this paper, the most two commonly used methods are reviewed and these include the application of a heating element and other methods that directly heat the MUT.

A rudimentary way of heating materials in liquid form is by placing a filled glass beaker or any other form of glass or metallic container on a hot surface, as is shown in Fig. 1A. Such a method was employed to measure the dielectric properties of blood in [11]. In this study, the temperature range was varied between 30°C and 50°C, to investigate the dielectric properties at hyperthermic temperatures for medical applications. Uniform heating was ensured by continuous stirring during heating using a magnetic stirrer hotplate. Another application was to measure the dielectric properties of different beverages for microwave-assisted pasteurisation applications [12]. In this case, the temperature range was varied between 10°C and 70°C. The limitation of having a container above a heat source to warm material is the non-uniform heating of the MUT, especially for semi-solid or solid materials, as one is only reliant on the heat conduction property of the material and more prone to temperature fluctuations. An alternative heating method to this is to submerge a glass or metallic beaker, flask or any other non-permeable container filled with the MUT inside a temperature-controlled water bath, as shown in Fig. 1B. This method, is commonly used to investigate dielectric properties of biological tissues at hyperthermic temperatures ranging from 30°C to 50°C for liver, muscle and fat [11, 13]. Unlike the previous method, more homogenous heating is provided because of a larger heated surface area is in contact with the container. Hence this method is better appropriate to heat solids and semi-solids. Higher temperatures were achieved using this method to investigate dielectric properties for magnetic resonance imaging-guided focused ultrasound surgery. In this case, ablation temperatures between 36°C and 60°C were achieved to characterise porcine uterus, liver, kidney, urinary bladder, skeletal muscle, and fat tissues [14]. In [15], such a method was used to investigate the dielectric properties of bovine liver tissues at ablation temperatures, up to 79.2°C. The limitation of using a water bath is that the highest temperature cannot exceed 100°C. Higher temperatures were achieved in another study by substituting water with oil by having a temperature-controlled oil bath as described in [16]. This method was used to measure the dielectric properties of sweet potatoes purees required in applications such as microwave processing of food.

Radio-frequency radiation can also be utilised to heat biological tissue. The method described in [17] consists of a signal generator connected to two stainless steel electrodes. The sample, in this case, porcine liver tissue, is sandwiched between the electrodes, as is shown in Fig. 2A. Heat is generated by the Joule effect [17]. Temperatures from 37°C to 100°C were reached using this method when investigating the dielectric properties of biological tissues. A more invasive method to heat up the MUT is to insert an energised radiating microwave antenna in the material. Biological tissue is typically used for studies related to thermal ablation, where the material surrounding the antenna is heated to the desired temperature. In [18], ovine kidneys were heated to around 60°C, and after the procedure, the ovine kidneys were bisected, and their dielectric properties were measured. Another study also related to medical applications described in [19], report on the dielectric properties of porcine liver during the heating process. The latter utilises an open-ended probe positioned perpendicular to the microwave antenna to minimise coupling. The temperatures achieved in this study was from 25°C to 93°C and a schematic of the setup is illustrated in Fig. 2B.

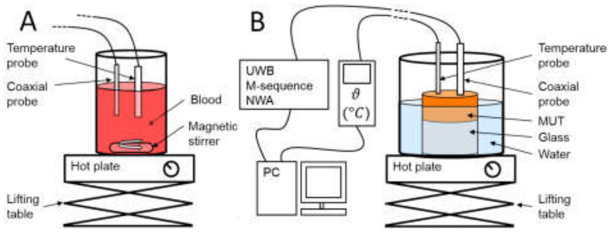

Fig. 1: shows different heating methods using a hotplate [11]; A: liquid sample placed directly on top of the heat. source B: water bath method.

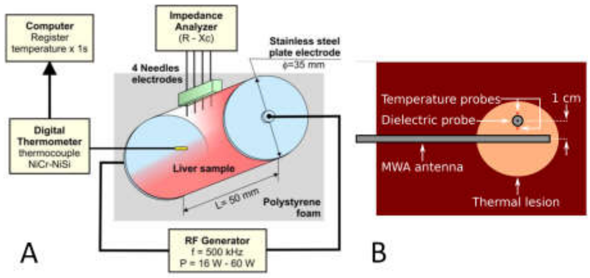

Fig. 2: shows two different heating methods using non-ionising radiation; A: heating induced by radio-frequency [17], B: heating induced by microwave antenna [19].

B. *Dielectric measurement Techniques*

There exist various methods that are used for measuring the dielectric properties of materials and in this section, two of the most commonly used methods are reviewed: the open-ended coaxial reflection and the resonant cavity techniques.

The open-ended coaxial cable measurement technique is performed by means of a coaxial cable of a known length with one side typically having an SMA connector attached, connected to a measuring device (vector network analyser or impedance analyser), whilst the other end is left flush and open-ended. The latter is immersed or placed in contact with the MUT and the reflection coefficient is measured and then used to derive the dielectric properties of the MUT. This technique is used for various applications in the food industry and also medical applications. In the reviewed papers, the dielectric properties of various food products are reported for food processing and preparation. This technique is also used to measure the dielectric properties of semi-solid, liquids and powdered food, including ground wheat flour, apple juice and cheese. An example of this, is presented in [20], where the frequency range was between 27 MHz and 1.8 GHz, and the sample was heated from 5°C to 100°C, with the calibration being done at 25°C. Dielectric measurements at higher frequencies, 915 MHz and, 2.45 GHz, were used in [21]. This study investigated semi-solid samples, where the temperature was varied between 7°C to 90°C, following a calibration at 25°C. In both studies [20, 21], the dielectric properties were measured whilst heating the MUT.

The open-ended technique, shown in Fig. 3A, is also widely used in studies focusing on the dielectric properties of biological tissues. This data is of critical importance when it comes to numerical modelling of medical devices during technological development and their optimisation. In [22], the dielectric properties of porcine glandular and gonad tissues were measured *in vivo* while the animal was under anaesthesia. The frequency range was from 50 MHz to 20 GHz, and the calibration was performed periodically before insertion. Other existing studies instead opt to measure the dielectric properties of an *ex vivo* tissue in elevated temperature as described in [23], to emulate the ablation process. In this case, the open-ended coaxial probe is immersed in excised bovine livers together with a radio frequency antenna as a heating device. The temperature range investigated in this study was between 5 °C and 100 °C with a measurement band of 500 MHz to 5 GHz. In this case, the calibration was also performed before the measurement process and at a fixed temperature.

Whilst there exist commercially available open-ended coaxial probes, such as Agilent 85070E, there are laboratories that manufacture open-ended coaxial probes in-house and these are designed to fit specific experimental requirements. An example of this is an open-ended coaxial probe manufactured for extremely high temperatures used to conduct dielectric measurements of thin alumina and sapphire samples at 600°C to 800°C [24]. Such probes are made of a metallised-ceramic probe and are mostly used for applications in microwave material processing. The frequency band investigated in this study was from 0.5 GHz to 3.0 GHz, and the measured results were compared with those measured using cavity perturbation techniques, obtaining good agreement.

The latter method is widely used for the characterisation of the dielectric properties of various materials, in many fields ranging from physics and material science to medicine and biology. The system usually consists of a resonant cavity, such as standard resonant cavities (eg. Rectangular or circular waveguides, coaxial cable resonators, etc) or custom made cavities designed to resonate at a particular frequency or for specific application. The MUT needs to be machined with adequate dimensions to be inserted in the cavity and this causes a perturbation in the field due to the distinct material properties inserted in the cavity as compared to an empty (unperturbed) cavity. Analytical expressions for the resulting shift in the resonant frequency and Q factor can be derived by referring to the unperturbed (empty) cavity, from which the dielectric properties of the MUT can be obtained. A typical setup of such a dielectric measurement technique is presented in Fig. 3B.

In [26], a cylindrical resonant cavity was used to measure the dielectric properties of vegetable oils during a heating cycle, from 28°C to 200°C. Measurements were conducted at 2.5 GHz and calibration was done using a standard Open-Short-Line to correct for the length of the cables and their transitions. The dielectric measurements were then correlated to lipid oxidation which is important for optimising heating processes in different applications, such as cooling fluids and frying of foods.

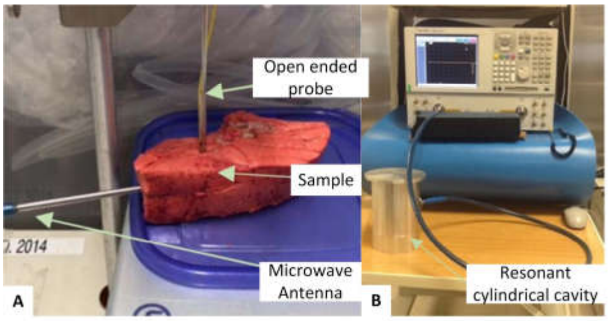

Fig. 3: Two dielectric measurement techniques; A: open-ended coaxial probe [25], B: Resonant cavity perturbation [26].

The dielectric properties of semi-solids can also be measured using a resonant cavity technique. An example of this, is presented in [27], where the latter technique was used to characterise meat samples at 2.43 GHz. However, in this study, meat was homogenised to fit the cavity and dielectric properties were obtained after heating the samples using a microwave oven.

III. DISCUSSION AND CONCLUSION

In this paper, a preliminary review of the measurement methods and heating techniques for temperature-dependent dielectric properties of materials is presented. Materials other than biological tissues are considered in this review as this could provide a better insight to address challenges with current methods and techniques. It is important to note that prior to selecting a heating method, one must consider the material's thermal properties, its state, heating rate and the implemented measurement technique and whether these are also dependent on the temperature. An example of this is when heating of the MUT (eg. biological tissues) will cause changes in the hydration levels of the samples, particularly when conducting dielectric measurements, characterising the γ-dispersion. Additionally, in most of the reviewed studies, the calibration methods used are not temperature-dependent and thus do not consider any systematic errors in the setup due to temperature changes. Moreover, some studies conduct discrete measurements whilst others opt for continuous measurement whilst heating the MUT. This may result in additional errors since the measurement setup and MUT are not in thermal equilibrium.

The choice of method for dielectric measurements highly depends on the MUT and the frequency of interest. In the case of liquid and semi-solid materials, open-ended coaxial probe technique is very easy to use. Resonant cavities can also be used in the case of liquids, and however, in some cases, this can present additional challenges related to confining the MUT inside the cavity. There exist studies that propose the use of Kapton foil to contain the MUT [28]. However, this needs to be carefully considered when calibrating the system. In the case of a solid MUT, both measurement techniques can present some challenges, as for the resonant cavity is concerned, the sample needs to be machined to exactly fit inside the cavity, while for the open-ended coaxial probe, the fringing fields emanating from the open-tip needs to be absorbed by the MUT. Additionally, perfect contact (no air gaps) between MUT and open-tip is required for accurate determination of the dielectric properties, and this can be a significant challenge. In the case of biological tissues, which for most of the tissues can be considered as semi-solid, careful consideration needs to be taken. Particularly, when using a resonant cavity, homogenisation can be considered but this could only be accurate for high-frequency dielectric measurements, characterising the γ-dispersion. Homogenisation of samples will destroy all cellular membranes, and thus such method cannot be considered for low frequency such as in the β-dispersion.

From the whole population of studies, some of which are not presented in this paper, one can also add that the method in which the results were presented is also of great importance. Result standardisation will allow for direct comparisons between one study and another. Furthermore, these results often serve as a foundation for newer ones. The most common methods of describing the temperature dependence of the dielectric properties are: actual dielectric measurements at particular temperatures, dielectric plots of variations as a function of both frequency and temperature, data at a single frequency as a function of temperature, providing temperature coefficients and Cole-Cole parameters and their variation with temperature. While all mentioned are valid methods for data representation, direct comparison between studies of non-similar methods is challenging.

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