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Microwave Characterization and Modeling of the Carasau Bread Doughs During Leavening

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ABSTRACT This work deals with the electromagnetic characterization of Carasau bread doughs, in the microwave range region. The microstructure, texture, product quality, and dielectric properties were investigated, jointly with a thorough thermogravimetric analysis. The goal is to link the physical properties with the product quality and to process variables. The variation of the dielectric properties during the dough leavening is studied. The dielectric properties of raw materials (water, yeast, salt, and semolina) were measured from 500 MHz to 8.5 GHz. Given the heterogeneous character of bread microstructure, the possibility of predicting by using mixing formula the final characteristic of a standard Carasau bread dough, manufactured with three different semolina wheat batches, is investigated. A power-law model can predict the average dough properties for frequencies higher than 2.45 GHz. This work proposes a dielectric spectroscopy model for this food material. A third-order Cole-Cole model can describe the dielectric spectra of the Carasau doughs over the entire frequency range. The decrease of the complex permittivity of the dough during leavening can be modeled with a reduction of the static permittivity and a decrement of the electrical conductivity. This study can foster the development of a microwave inline system for leavening monitoring.

INDEX TERMS Carasau bread dough, dielectric materials, dielectric measurements, dielectric spectroscopy, food manufacturing, microwave measurements, open-ended coaxial probe, thermogravimetric analysis.

I. INTRODUCTION

Industry is facing a transition towards a new paradigm made of digitalization, automation, precision, reduced waste, and high quality [1]–[3]. Among the several industrial branches, food industry sector is involved in the challenge of keeping pace with this technological revolution [3]–[5]. In this framework, the development and availability of cost-effective engineering solutions for increasing the competitiveness of small size, traditional activities is a major concern [3], [6].

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Typical bakery industries are constrained to increase and automate their production while seeking high quality and traceable products [7]. In this work, we focus on the industrial advancement of a traditional, small-scale bakery of the Carasau bread industry in Sardinia, Italy [8].

Carasau bread is a flat, crispbread whose demand from Italy and Europe is growing [8]. To increase the level of automation of the industrial production of this process hybrid Petri nets were investigated [9]. A heterogeneous, three-tier wireless sensors network (WSN) was designed, proposed, and tested for monitoring the process and providing feedback about the bread manufacturing steps [8], [10]. As shown

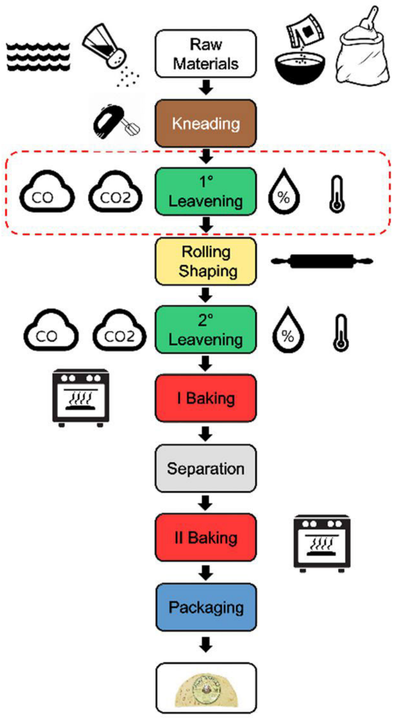

FIGURE 1. **Manufacturing process of the traditional Carasau bread.**

in Fig. 1, the Carasau manufacturing initiates by mixing and kneading the raw materials, i.e., water, salt, yeast, and semolina. Then, the dough is placed, for 30-40 min, in a metallic box and undergoes the first leavening step. The leavened good is then sheeted, cut in a circular shape (∅ = 18-36 cm), and experiences a second, longer leavening, followed by baking at 400°C. The baked bread disks are separated manually into two sheets, which are baked again (Fig. 1). Then the product is packaged and sold. In the process of Fig. 1, the more critical steps are dough preparation and the first leavening [8]–[14]. The dough composition and the leavening condition can drastically affect the number of scraping and the final product quality, thus affecting the productivity and competitiveness of the bakery.

Despite the use of a WSN for monitoring the environmental (e.g., temperature, pressure, humidity, etc.) and the processing parameters (i.e., machinery-related quantities) is promising, different studies focused on the possibility of developing physical sensors for linking the final, organoleptic product properties to ingredients and process variables. In particular, the rheological properties of Carasau bread doughs were investigated in terms of composition and quality [11], [12]. It has been found that water is the most critical component in determining dough viscosity, whilst salt reduces the elasticity [11], [12]. However, the dough properties after the leavening and their impact on the bread-making process were not investigated. Similarly, by using thermogravimetric analysis (TGA), the thermal properties of Carasau doughs were investigated to reduce the number of off-specification products [13], [14]. The TGA performed on semolina doughs revealed differences in the dough characteristics, mainly due to the different composition and availability of water [13], [14].

Although the relevant insight gained in the dough composition, rheological and TGA methods do not ensure the possibility of designing an in-line, non-destructive device for monitoring the leavening and enriching the technological level of the Carasau bread production process. In this framework, dielectric spectroscopy (DS) stands out as an appealing tool for analyzing food products and developing innovative quality control methods to favor large-scale production [15]–[18]. In particular, applied to doughs and bread, DS in the microwave (MW) range (300 MHz-300 GHz) offers a nanosecond timeframe for observing the dipolar relaxation of starch and gluten matrix at glass transition temperature [19]. Indeed, Zuercher *et al.* firstly measured the dielectric characteristics of a single one commercially available bread dough and yeast under various conditions (normal, extra water, extra flour, and raw or baked) by using an open-ended coaxial line from 600 MHz to 2.45 GHz [20]. However, the characterization was mainly oriented to microwave baking and the work suffers from a limited range of frequencies. For this reason, the authors did not accurately model the dielectric spectra, scarcely focusing on the dynamic of the leavening process and its correlation with the electromagnetic signature of the doughs. Chin *et al.* analyzed how the dielectric properties of doughs, made with commercial flour (14.3% protein, 0.6% ash, 12% moisture), variable densities, salt content, water levels affected the signal transmission (only attenuation and phase shift) in a coaxial cell, working in the microwave range from 300 MHz to 6 GHz [21]. The dielectric permittivity was not measured, but it was found that the salt could increase microwave attenuation, with scarce effect on the phase shift, whilst water amount causes a slight increase of both attenuation and phase shift [21]. Liu *et al.* analyzed the white bread (60% protein, 3% ash, 1% fat, 51 carbohydrates, 37% water, 80% porosity) dielectric properties in the frequency range between 1 and 1800 MHz, and a temperature range of 25°C-85°C for developing RF or microwave pasteurization methods [22]. The moisture content was modeled through the Lichtenecker equation, so that the logarithm of the dielectric permittivity of white bread, as an effective, homogenized medium, is equal to the sum of the logarithms of the dielectric properties of its components, scaled by the respective volume fraction [22]. The frequency dependence was assumed to be polynomial. In ref. [22] it is shown that cooked white bread dielectric properties are inversely dependent on frequency and linearly dependent on moisture content and present a second-order polynomial dependence from temperature.

Previous literature works which dealt with the MW spectroscopy of bread presented a restricted frequency range, poorly addressed the modeling, and did not focus on the leavening [20], [21]. This work aims to fill these knowledge gaps. Moreover, these researches did not elucidate which

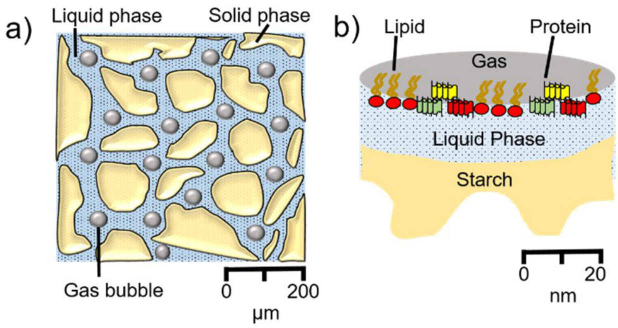

FIGURE 2. a) Sketch of the multiphase, heterogeneous dough microstructure (0-200 μm). b) Pictorial representation of the nanoscale assembly at the nanoscale (0-20 nm).

dough characteristics should be achieved to obtain a given final product texture or organoleptic features. Therefore, the feedbacks to industrial production were limited, without addressing quality control or scaling up.

In this work, for the first time, we aim to investigate the electromagnetic (EM) properties of the Carasau bread doughs, since they are essential to the development of the final product characteristics, in the MW range from 500 MHz up to 8.5 GHz with an open-ended coaxial probe. An in-depth characterization of the raw materials is performed. The electromagnetic properties of standard doughs, prepared with three different semolina batches, are analyzed during their first leavening over an extended 40 min time period. Given that, in literature, there is a lack of models for the complex permittivity of doughs, for the first time, we investigated the validity of mixing formulas for predicting the dough properties. Furthermore, in this work, the dielectric spectra are fitted with modeling equations, for the first time. Then, the complex permittivity and the model parameters are linked to the TGA data to investigate the leavening. The goal is to study how the composition affects the dough properties and then the crucial first leavening step (Fig. 1). To this aim, TGA is used as a supporting method for investigating the dough microstructure. An accurate electromagnetic characterization of the materials involved in the bread manufacturing process may foster the development of EM-based technologies for monitoring food quality indices or process scale-up, detect contaminants or food alterations [23], such as microwave imaging system for the inline inspection of cocoa cream [24], or chipless radio-frequency identification (RFID) devices for quality monitoring to be integrated with Internet of Things (IoT) systems [25].

II. MATERIAL AND METHODS

The understanding of how the structure and texture of the dough change according to its composition, during leavening is relevant for optimizing the production process and enhancing the quality attributes of the final product [26]–[28].

A. DOUGH PREPARATION

The doughs were prepared using commercial re-milled durum wheat semolina with a protein content of about 13% (carbohydrates (71%), fats (1.5%), proteins (11.7%), gluten (8.7%), gluten index (88%)), distilled water, commercial baker yeast (Saccharomyces cerevisiae) [29], and commercial iodized salt (NaCl). The proportion and amount of the four ingredients typically used during Carasau bread production are reported in Tab. 1. In this work, we focus on the standard (ST20) dough recipe. We selected the commercial semolina types mostly employed by the traditional bakery industry [9]–[14]. Semolina purchased by two different producers was considered (with labels B and V). Our investigation aims also to determine how and if the use of different semolina and different batches (called B5, B6, DV2) has a significant impact on the dielectric properties of doughs. Future works could deal with the investigation of a wider range of commercial semolina batches.

TABLE 1. Composition of standard (ST20) carasau dough.

Ingredient	Weight (g)	Fraction (%)
Semolina	300	65.36
Water	150	31.68
Salt	4.5	0.0048
Yeast	4.5	0.0048

A Sana Smart Breadmaker (SANABMS, Sana S.r.o., CZR) machine is used for dough kneading (Fig. 1) [11]–[14]. Since the mixing phase and the resulting doughs depend on the type of the stirrer and the applied stirring conditions, in this study, the kneading was carried out a room temperature (25°C) and for 20 min.

B. DOUGH MICROSTRUCTURE

From a physical point of view, the mixing of the raw materials results in a multiphase, heterogeneous system [30], as shown in Fig. 2.a. A gas, liquid, and a continuum dough phase can be distinguished. In this microstructure, the presence of air bubbles determines a foam-like arrangement [28], [30]. At the microscale, the dough phase is made of starch, gluten, and a disperse phase of yeast and starch. The dough molecular structure consists of a 3D network of saccharides (i.e., starch), lipids, and proteins, held by water adsorption, as shown in Fig. 2.b [28].

During leavening, the living yeast microorganisms work by initiating a set of metabolic reactions which causes the transformation of polysaccharides into carbon dioxide (CO_2), ethyl alcohol (C_2H_5OH), acetic acid (CH3COOH), and lactic acids (e.g., $C_3H_6O_3$). During the leavening phase (Fig. 1), the specific volume of the loaf increases due to the presence of air bubbles: CO_2 diffuses into the air nuclei created by the mechanical mixing, causing the gas bubble expansion [30]. Furthermore, the softness of the dough changes, and the pH decreases, as explained by the mathematical model from [30].

After the load reaches an empirically established volume threshold or overcomes a maximum leavening time established empirically [8], the product is sheeted, cut, and then baked (Fig. 1). As a result, the chemo-physical transformations that occur during leavening depends on dough composition and may drastically affect the production process of Carasau bread.

C. DOUGH INGREDIENTS: KNOWN EFFECTS

One of the most critical factors in bread production is the water quantity in the dough [11]–[14], [28], [30]. In fact, the polar water molecules behave as solvent and medium for the liquid-phase reactions of the other components, while regulating the interactions with hydrophobic gluten elements [11]–[14], [31]. As the water content increases, the volume of inclusions increases and viscosity decreases [12]. The TGA findings demonstrate that the temperatures of primary and secondary mass losses and the area under the associated exothermic peaks are influenced by water content in the dough [14]. From an electromagnetic point of view, the water presence turns the dough into a material with quite high relative dielectric permittivity (ε_r = 20-40) and lossy behavior [20]–[22].

The NaCl salt improves the taste of the bread and its sensorial properties [31]–[35], but it strongly affects the properties of the dough [12], [14]. Indeed, the salt addition increases the mixing resistance, while enhancing the extensibility, but decreasing the stickiness and, finally, helps in stabilizing the yeast fermentation rate, thus improving the bread texture [29]–[31]. In the liquid phase of the flour-water system (Fig. 2.b), the salt anions bind with the protein's positive charges and eliminate the repulsion among the chains, leading to an easier interaction [12], [28]. As a result, the gluten protein network microstructure modifies, i.e., glass transition temperature lowers, protein strands elongate, and their hydration lowers, strengthening the network, thus determining an increase of the dough optimal mixing time [11]–[14]. High levels of salt (2% or more) produce stiffer, sticky doughs [12]. The presence of the ionic salt, from a MW spectroscopy perspective, results in free charges which cause an additional dissipation mechanism, thus leading to an enhanced electrical conductivity.

Yeast is the main actor in leavening: yeast fermentation produces CO_2 and several metabolites that can influence the final product quality, the flavor attributes, and the physical properties of the dough [28], [31], [36]. The yeast metabolites soften the dough, due to the modification of the gluten network attributable to glycerol, ethanol, succinic acid, and glutathione [12], [14], [30]. Despite the pivotal importance of fermentation and yeast on the dough characteristics, it is difficult to perform the characterization of a living, complex, and constantly evolving system. Recently, the rheological properties of leavened doughs were investigated [11], [12]. A MW characterization of yeast was performed in [21]. Moreover, in ref. [36], yeast dielectric spectrum was investigated but the focus was on the identification of phytopathogens in plants [36]. The data from refs. [21] and [36] report a real part of relative permittivity of about several tens and losses comparable to distilled water. However, the influence and action of yeast during leavening on the dielectric permittivity has not been investigated yet.

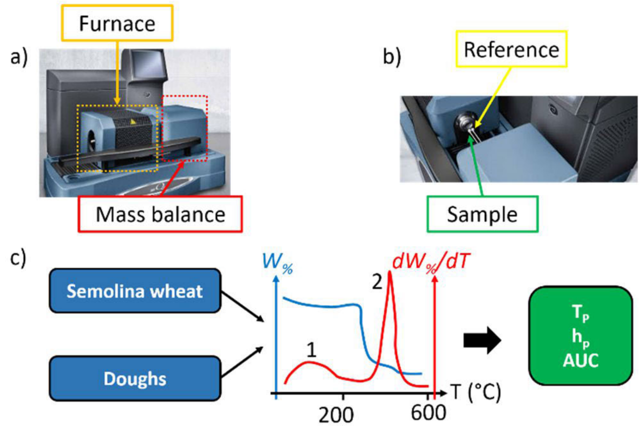

FIGURE 3. a) View of the furnace and mass balance of the Q600M TA instrument. b) View of the reference and sample pans. c) Schematic description of the experimental measurements and processing procedure. The peaks 1 and 2 are sketched.

D. THERMOGRAVIMETRIC ANALYSIS

The simultaneous Differential Scanning Calorimeter (DSC)-TGA analyzer Q600M (TA, New Castle, DE) was used as supporting method for investigating the effect of dough composition on leavened Carasau doughs [11], [12].

For each sample, a small quantity ($\sim$100 μg) of dough leavened for 40 min was put into an alumina crucible and inserted into the TGA device (Fig. 3.a-3.b). The sample was heated up to 600°C with a temperature ramp of 5°C/min (Fig. 3.c). For each run, the weight loss of the sample was registered (W), and then the percentage reduction ($W_\% = 100 \cdot (W - W_0)/W_0$) and the derivative of the latter for the temperature were calculated (DTG = $dW_\%/dT$) [13], [14]. Three replicate measurements for every sample were performed and then the average value was evaluated.

After this, two main peaks in the derivative curve (DTG) were individuated and characterized by the following parameters (Fig. 3.c): peak temperature (T_P), height (h_p, %/°C), and the area under the peak (AUP, in %) [14]. For the first peak (peak 1) the quantification of the left and right (with respect to the peak temperature) integrals was also performed, whereas for peak 2, an estimation of the temperature range, useful for the integral computation, was done. The peak temperatures and their heights were determined by means of a regression curve (second or third-grade polynomial) in a narrow temperature range around the peak. The integrals were determined as the total weight loss percentage between the considered temperature range limits (Fig. 3.c). The temperature range was conventionally fixed in 25–200°C for the first peak and determined for the second one from the intersections between the abscissa axis and the tangent passing for the inflection

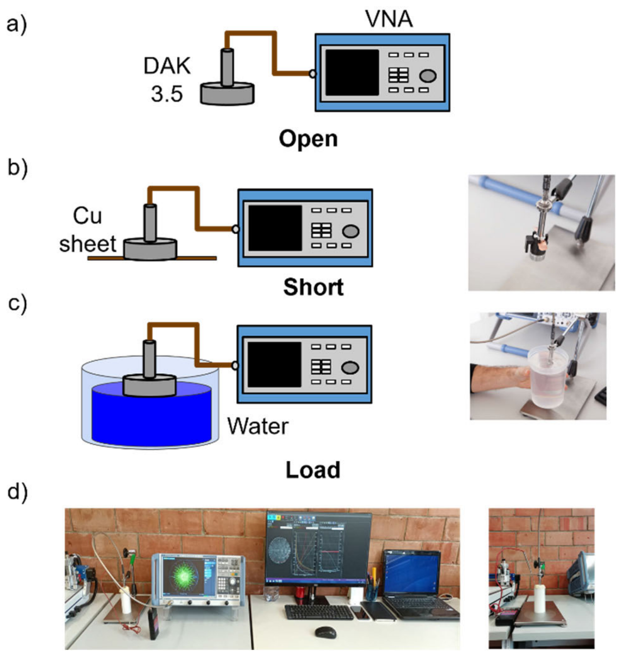

FIGURE 4. a) Open b) Short and c) Load steps for the calibration of the open-ended dielectric probe measurement setup. d) Experimental setup in the laboratory. The dough sample is placed inside a PLA cylinder and its temperature is monitored.

point of each (ascending and descending) part of the curve around the peak (Fig. 3.c).

E. OPEN-ENDED COAXIAL PROBE MEASUREMENTS

Microwave dielectric spectroscopy is a non-destructive, powerful method for characterizing food materials [15]–[24].

The response of a material to the EM field in the MW range is expressed in terms of the complex permittivity $\varepsilon = \varepsilon' - j\varepsilon''$, where ε' is the real part (in-phase component) and ε'' is the imaginary part (out-of-phase component) of the permittivity [15]–[18]. The real part of the permittivity is related to the energy stored in the material under test (MUT) and a measure of the dipole moments, whilst the imaginary part accounts for the losses and the energy dissipated inside the MUT [37], [38]. The different polarization and relaxation mechanisms (e.g., ionic, orientational, and interfacial) can be visualized by broadband MW spectroscopy [37]. As a result, the complex permittivity is the signature of the microstructure and composition of the MUT [37], [38].

In this work, the dielectric measurements were carried out by using an open-ended coaxial probe dielectric assessment kit (DAK) system (Fig. 4). The coaxial probe is connected to the vector network analyzer (VNA), which measures the complex reflection coefficient at the probe-material interface, and the measured reflection coefficient is used to extract the dielectric parameters by the DAK software. The measurement provides the dielectric spectra of the material as a function of frequency by retrieving the permittivity from the probe scattering parameters, according to a well-known capacitive model [37].

The measurement system comprises a VNA (Rhode & Schwarz ZNB 8) with frequency ranges between 9 kHz-8.5 GHz and a 3.5 DAK-probe with frequency range 200 MHz-20 GHz (SPEAG; www.speag.com). The probe is connected to the VNA using a rigid, low-loss coaxial cable and a lab jack is used to move the MUT toward the probe, as shown in Fig. 4.d.

The measurement system was calibrated following the open, short, and load (Fig. 4.a-4.c) (OSL) procedure. The shorting was performed by using a copper strip. De-ionized water (1 L) was used as load and the temperature of the solution was monitored with a digital PT100 thermometer (±0.05°C accuracy).

The semolina and dough samples were gently deployed inside a 3D printed poly-lactic acid (PLA) cylindrical sample holder, having a diameter of 5 cm and a height of 5 cm (Fig. 4.d). Such a MUT volume reduces the error associated to the fringing fields, penetration depth, and uncertainty associated to sample inhomogeneity [39]–[41]. We tried to avoid large deformation or induced mechanical stresses.

Several types of errors (e.g., VNA noise, temperature uncertainty, geometrical factors) affect the experimental procedure [15], [38]–[43]. In this framework, we designed our experiment to take into account these sources of errors. Our approach stands out as a more conservative and accurate one, with respect to previous works which dealt with the dielectric characterization of bread products [20]–[22]. In refs. [20]–[22] the number of measurements, the variability of the wheat products and the uncertainty are underreported or poorly addressed aspects. Therefore, in this work, we aim to follow the best practices and the dielectric measurements guidelines established to provide the minimum information, metadata for repeatable and reusable data, in a FAIR sense [42], [43]. Consequently, we monitored the environmental parameters of the room (i.e., temperature, relative humidity, pressure) by using the heterogeneous, multi-sensor node from [8]. Furthermore, the combined, total standard deviation, at every frequency point, was evaluated by using 0.1 M NaCl solutions as explained in ref. [44]. The combined standard deviation is obtained by summing the random uncertainty (up to 2%), systematic uncertainty (ranging from 0.1-1.4 % [41]) and the drift (0.1-0.2 % [41]) during the experiment, by using k = 2 as coverage factor for a 95% confidence interval, as found in [44]. The actual permittivity values in the following are therefore represented with error bars with an expanded uncertainty. The dielectric measurements are repeated three times for a given raw material and for any dough, given that the variability and repeatability are the largest uncertainty elements [41]. The average curves are reported and the standard deviation of the measurements is added to the combined standard deviation, as usual [42], [43].

The raw materials, i.e. de-ionized water, 1.5% NaCl+water (4.5 g of salt in 125 g of water), the three semolina batches (B5, B6, DV2), and yeast were characterized. Then, the doughs were prepared with B5, B6, DV2, according to the procedure from Sect. II.A, and were set in the PLA container.

The DAK probe was firmly placed in contact with the sample (Fig. 4.d). Then, measurements of the complex permittivity were performed for 40 min, during the first leavening phase (Fig. 1).

F. MIXING FORMULAS AND DOUGH PROPERTY PREDICTION

This work aims to retrieve the dielectric properties of Carasau doughs during leavening and provide the basis for developing an electromagnetic-based system for empowering the industrial production of Carasau bread. To this aim, an important aspect is the understanding of how the starting components (Fig. 1), their interaction, and the processing affect the semi-finished product (Fig. 3). A crucial aspect is the possibility of using effective medium theory to predict the dough properties starting from the knowledge of the dielectric behavior of dough ingredients.

The generalized Maxwell Garnett (MG) formula can be used to model the effective, complex dielectric permittivity of the dough (ε_d) [45]:

$$\varepsilon_d = \varepsilon_b + 3\varepsilon_b \frac{\sum_{k=1}^{K} f_k \frac{\varepsilon_k - \varepsilon_b}{\varepsilon_k + 2\varepsilon_b}}{1 - \sum_{k=1}^{K} f_k \frac{\varepsilon_k - \varepsilon_b}{\varepsilon_k + 2\varepsilon_b}} \tag{1}$$

where the k-th components are air (ε_0), water (ε_w) semolina (ε_s) as depicted in Fig. 3. The background permittivity (ε_b) is assumed to be that of the main dough component, i.e., semolina for the ST20 dough recipe from Tab. 1. Therefore, the number of multiphase, components is $K = 3$, in the MG case.

The Maxwell Garnett or effective medium approach is the simpler approximation as little information about the scatterers is used, but, assuming their spherical shape, only their volume fraction in the matrix is exploited [51]. In this framework, the implicit Bruggeman formula can be used to numerically find the effective permittivity (ε_d) starting from the knowledge of the properties of the single components' [45]:

$$\sum_{k=1}^{K} f_k \frac{\varepsilon_k - \varepsilon_d}{\varepsilon_k + 2\varepsilon_d} = 0 \tag{2}$$

The computation of the effective permittivity from the measured permittivity of the dough ingredients was carried out in Matlab 2021a (The MathWorks Inc., MA USA) by using the function "*fsolve*", with the Levenberg-Marquardt algorithm.

Since in ref. [22] the Lichtenecker (L) equation was used for modeling the white bread dielectric properties, in this work, we tested, with the volume fractions reported in Tab. 1, the equation [45]:

$$\log \varepsilon_d = \sum_{k=1}^{K} f_k \log \varepsilon_k \tag{3}$$

Finally, the effective permittivity was computed with the power-law (PW) by searching the best exponent value (β), according to [45]:

$$\varepsilon_d^{\beta} = \sum_{k=1}^{K} f_k \varepsilon_d^{\beta} \tag{4}$$

Eq. (1)-(4) are used to verify if, starting from the characterization of the single components, with the knowledge of their dielectric permittivity the measured dough permittivity can be framed and interpreted by using the formalism of effective medium theory.

G. DIELECTRIC SPECTROSCOPY MODELING

As suggested by best practice [42]–[44], it is desirable to model the MUT response to the microwave frequency by DS and relaxation models. The modeling will help in elucidating the molecular and microstructure interactions with the high-frequency signal while providing a reliable framework for any future device modeling. The DS modeling is mandatory if the mixing formulas would not work. In fact, some predictive models for the dielectric properties of foods have been proposed [46]. Usually, the Debye model is assumed as sufficient to model the reorientation of dipolar molecules (mainly water), considering a constant relaxation time [44], [46]. However, depending on the MUT and the frequency range, the assumption of distributed relaxation times leads to a more accurate description of dielectric spectroscopy. Therefore, multipoles Cole-Cole and Havriliak-Negami (H-N) models are often employed [44]. In this work, we did not consider any multipole H-N model because of the large number of parameters involved.

To date, no model of the dielectric spectra of bread doughs was proposed, whilst in refs. [22], [47], despite modeling the heterogeneous nature of the dough, only a polynomial model for the frequency dependence was provided. For baked bread, instead, modified Debye equations were used [48]. Therefore, there is no a priori theoretical framework with a strong physical rationale, to apply to the complex dielectric permittivity of bread doughs.

In this work, we tested different theoretical permittivity models. The modified Debye equation was considered [49]:

$$\varepsilon_{th}(\omega) = \varepsilon_{\infty} + \sum_{q=1}^{N_p} \frac{\Delta\varepsilon_q}{1 + j\omega\tau_q} + \frac{\sigma_{dc}}{j\omega\varepsilon_0} \tag{5}$$

where ω is the angular frequency $2\pi f$, ε_{∞} is the relative permittivity at optical frequencies, $\Delta\varepsilon$ is the difference between the static relative permittivity and the optical one, τ is the relaxation time (in s), whilst σ_{dc} is the static electrical conductivity (in S/m) and ε_0 is the vacuum permittivity, equal to $8.85 \cdot 10^{-12}$ Fm^{-1}. The symbol N_p is the number of poles, assumed to vary from one to three. The idea behind the proposal of the Debye-like equation is that the relaxation and dispersion of doughs could be attributed to water presence and small conductivity due to ionic salt.

Although being physically sounding, the non-linear transformation that raw materials (water, flour, salt, and yeast)

TABLE 2. Solution space of the dielectric model coefficients.

Param.	Min	Min
ε_∞	1	10
$\Delta\varepsilon$	5	80
τ (s)	10^{-12}	10^{-5}
γ	0	1
σ_{dc} (S/m)	10^{-6}	1

experience during the mixing and kneading may introduce effects that could not be described by the Debye equation. Therefore, given the peculiar dough microstructure shown in Fig. 3, we investigated whether the following Cole-Cole equation could be used [50]:

$$\varepsilon_{th}(\omega) = \varepsilon_\infty + \sum_{q=1}^{N_p} \frac{\Delta\varepsilon_q}{1 + \left(j\omega\tau_q\right)^{1-\gamma_q}} + \frac{\sigma_{dc}}{j\omega\varepsilon_0} \tag{6}$$

where γ is the broadening parameter (also called the relaxation time distribution parameter or shape coefficient). The maximum number of poles is four. Eq. (6) can cope with the macroscopic behavior of complex systems, which present non-exponential relaxation laws, such as biological tissues and polymers [50].

To retrieve the parameters ($2+2N_p$ for the Debye models, $2+3N_p$ for Cole-Cole) for the dielectric spectrum of the MUT, we used Matlab to perform a fitting procedure, based on a genetic algorithm (GA), aimed at minimizing, for the N_f frequencies, in a bounded sense, the absolute difference of the experimental and theoretical values for the unknown coefficients vector $\boldsymbol{x}$, relying on the following cost function [51]:

$$f(\boldsymbol{x}) = \sqrt{\frac{1}{N_f}\sum_{i=1}^{N_f}\left[\left|\frac{\varepsilon'_{m,i} - \varepsilon'_{th,i}}{\varepsilon'_{m,i}}\right|^2 + \left|\frac{\varepsilon''_{m,i} - \varepsilon''_{th,i}}{\varepsilon''_{m,i}}\right|^2\right]} \tag{7}$$

where ε_m is the measured complex permittivity and $\varepsilon_{\rm th}$ is the theoretical complex permittivity. The initial population was set to 30000 individuals, the Tournament selection method was used, and the maximum iteration number was 500, whilst the crossover and mutation probabilities were 0.9 and 0.1, respectively. The constraint tolerance was set to 10^{-6}. The solution space for the parameters of Eq.s (5)-(7) is reported in Tab. 2. The hierarchical relationships between relaxation times and dielectric strengths are respected [50].

The percentage relative error is used as a metric for evaluating the quality of the fitting and then finding the best DS model for the case of Carasau bread doughs.

Once the best DS model is found, the fitting is carried out for all the ten measurements of the three doughs to track the model parameters during the first leavening. Previous solutions are used as initial values for fitting the dielectric properties at time $t > 0$.

H. LEAVENING MODELING AND LINK TO MW PROPERTIES

The correlation between the biochemical reactions, dough microstructure, quality, and electromagnetic properties has not been fully investigated yet. This work aims to contribute by providing a thorough and extensive understanding of the Carasau doughs' dielectric properties. In particular, the modifications which occur during the first leavening step (Fig. 1), relevant to the manufacturing process, were never correlated to modification of the dielectric spectra of the dough.

From a physical point of view, during the leavening, described in Sect. II.B, a dehydration process verifies, i.e., the firmness, index of water content in the dough, can vary during leavening [30]. This loss of moisture can be modeled as a volumetric, first-order kinetic, during which the time-evolution of the moisture content in the dough experiences an exponential decay [44], [52]. The time constant of the dehydration process is a non-linear function of the sample geometry, the velocity of the air flowing around the air-dough interface, the temperature, and the relative humidity in air [44]. Furthermore, the softness (i.e., specific volume, correlated to the air volume in the dough) can increase by about 50% in 2 h, whilst the dough acidity can drop off to 5% of its initial value in the same timeframe [30]. Despite this experimental evidence, the dynamic behavior of the MW properties of dough has never been monitored during leavening [20]–[22]. In this work, we investigate the variation, during the leavening, of the dielectric permittivity of Carasau bread doughs at the specific Industrial, Scientific, and Medical (ISM) frequencies of 0.915, 2.45, 5.8 GHz. Moreover, the variation of the DS model parameters during leavening is analyzed.

III. RESULTS AND DISCUSSIONS

We characterized the raw materials (water with NaCl, yeast, semolina dough) mixed, as described in Sect. II.A, to obtain the Carasau doughs. The real and imaginary part of the relative dielectric permittivities of the starting ingredients (ε) are shown in Fig. 5. Some interesting aspects about the dielectric responses of the dough components can be recognized. At first, the difference between the tap water used for the dough preparation and the salt addiction is relevant, since the salt presence strongly enhances the losses, which are higher than a 0.1M saline solution [44], since the molar concentration in the water used for the dough would be about 0.6. This feature can establish the dielectric signature of the dough and can be a relevant aspect to devise an electromagnetic-based sensor for the monitoring of the product properties during the processes.

From Fig. 5, the real part of the relative dielectric permittivity of the raw unbaked yeast is very close to the value of about 50, for frequencies from 600 MHz to 2.45 GHz, reported by Zuercher *et al.* [21]. As recently found in [36], the yeast, from a MW point of view, can be ascribed to lossy media, such as biological, presenting a relatively high real part of the relative permittivity (from 72 to 48 in the 0.500-8.5 MHz range).

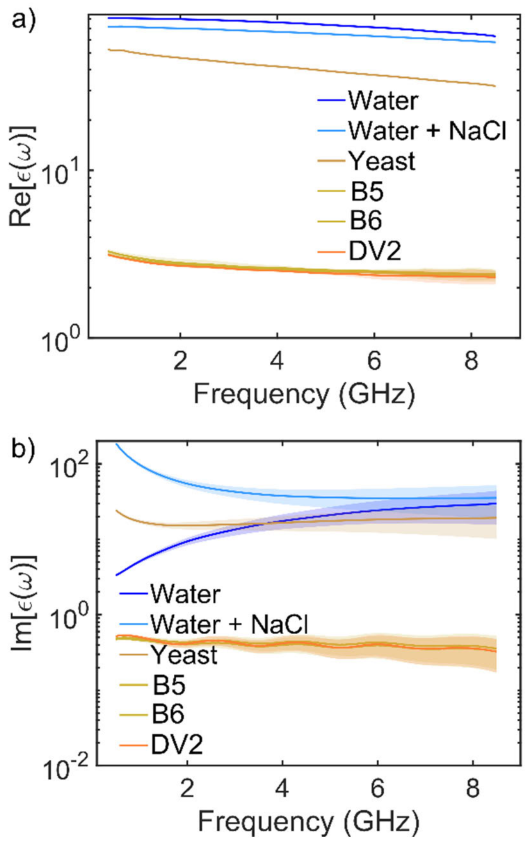

FIGURE 5. a) Real and b) imaginary relative dielectric permittivity over frequency for the raw materials (water, saline, yeast, and semolina) used for the Carasau doughs preparation.

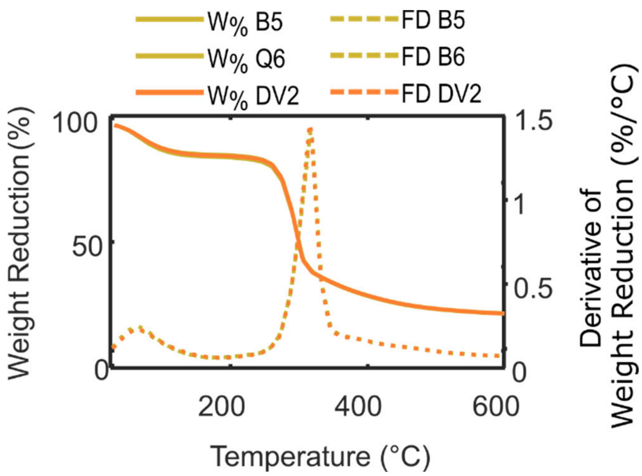

FIGURE 6. Weight reduction ($W_{\%}$, %) and the first derivative (FD) of weight reduction (%·C^{-1}) as a function of the temperature for the three doughs, prepared with three different batches of semolina, with standard composition (Semolina 46%, Water 50%, NaCl 1.5%, Yeast 1.5%). Each curve is the average of three independent TGA runs.

The dielectric properties of semolina dough were never reported in the literature, to the best of the authors' knowledge. To support our dielectric measurements, shown in Fig. 5, and characterization, we considered similar raw flours and powders. For instance, the dielectric properties of wheat flour (Triticum aestivum L, 12.56% water, 11.39% protein, 0.59% fat, 0.64% ash) were investigated from 10 MHz to 3 GHz considering variable moisture content and temperature (from 25°C to 85°C) [53]. Between 500 MHz and 3 GHz, the wheat powders presented a real part of the relative dielectric permittivity between 5 and 2.5, in the temperature range 25°C-35°C, which is comparable to the value of 2.58±0.63 shown in Fig. 5. In [53], the loss factor varied between 2 and 5, whilst for the semolina dough is 0.056±0.03. Therefore, semolina dough has a lower moisture content, compared to other types of wheat. By observing Fig. 5, we can see that the three curves of the real and imaginary parts of semolina patches overlap, thus implying that no significant differences between the semolina powders exist.

This result can be relevant information for the typical bread producer. The different moist content of semolina and the statistically not significant difference can be supported by the TGA curves shown in Fig. 6. From the thermogram analysis, roughly the same behavior and properties of the different dough samples were found, and the weight reduction rate around and just over 100°C is almost the same, meaning that the differences in the starting moisture content of semolina can be neglected for this scope.

The complex dielectric permittivity for a standard Carasau dough, at the initial time of the leavening step, is shown in Fig. 7. For the permittivities of the raw ingredients reported in Fig. 5, we can observe that the real part of the dough permittivity varies from 30 to about 20 in the 0.5-8.5 GHz range, due to high semolina content, which compensates the saline water response.

On the other hand, the imaginary part of the dough permittivity has an intermediate character between saline and yeast, with a marked lossy behavior. Given this comparison, it is reasonable to wonder if the dough properties can be predicted using the formalism of mixing formulas. In Fig. 7, by using the fractions reported in Tab. 1, we present the effective dough permittivity computed using Maxwell Garnett (MG), Bruggeman (B), Lichtenecker (L), and Power Law (PW) models, i.e., Eqs. (1)-(4). The MF, B, and L models fail in the calculation of the dough dielectric behavior, for both the in-phase and out-of-phase components. These 55-73% differences can be due to the nominal percentages may have changed during the dough kneading. Indeed, water can be lost due to the viscous losses and the heat produced by the mechanical stresses generated during the stirring phase [11]–[14]. Furthermore, non-linear phenomena due to the yeast activation and biochemical reaction are occurring, modifying the wheat structure, and hence its final response. In this framework, the PW model, for $\beta = 0.139$, was the best mixing formula for forecasting the dough MW response. Indeed, within the standard deviation of the experiments, the PW model can cover the complex dough permittivity from about 2.45 GHz to 8.5 GHz, with a minimum difference of 2% for the real part and of about 5.5% for the imaginary part.

Given the evidence that the dielectric permittivity of the Carasau doughs cannot be entirely and quantitatively

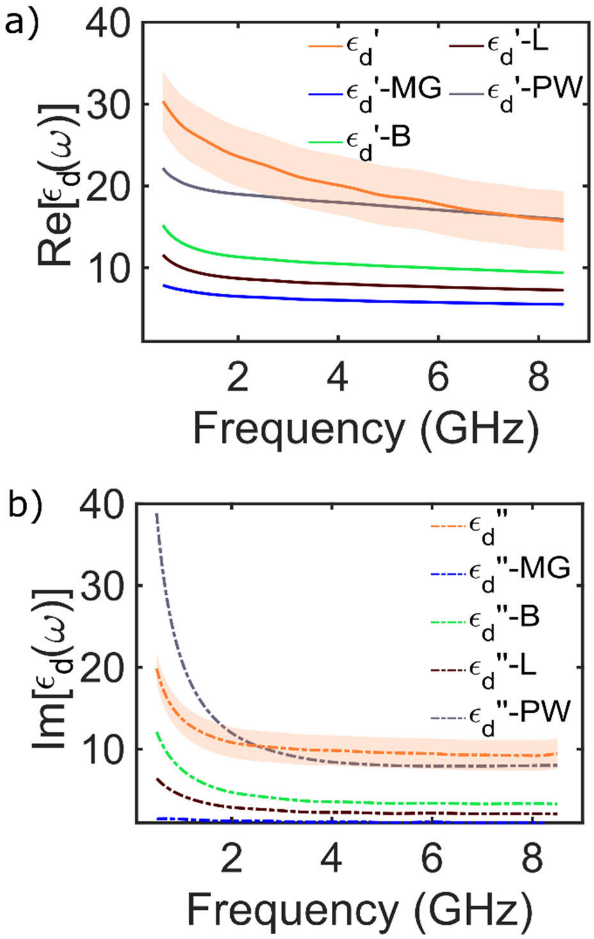

FIGURE 7. **Measured complex relative dielectric permittivity of the standard dough (ε_d), averaged over three measurements, at t = 0 min of leavening, against the effective permittivity computed according to Maxwell Garnett (MG), Bruggeman (B), Lichtenecker (L), and Power Law (PW) models for the nominal recipe of 65.39% of semolina, 33% water, 0.048% yeast and 0.01% air.**

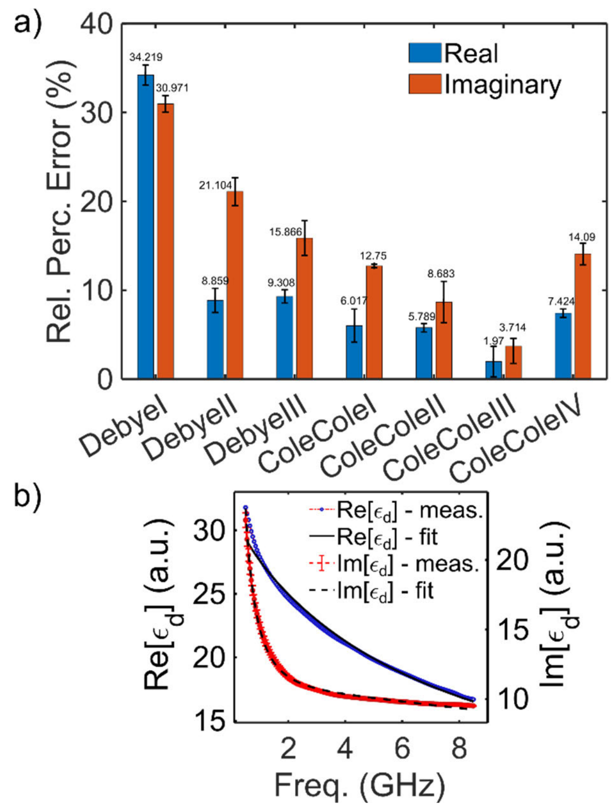

FIGURE 8. **a) Relative percentage error of the real and imaginary parts for the Debye with one pole (DebyeI), two and three poles (DebyeII, DebyeIII), and Cole-Cole models with one to four poles (ColeColeI, ColeColeII, ColeColeIII, ColeColeIV). b) Example of the fitting of the complex dielectric permittivity (ε_d) of the DV2ST20 dough at *t = 0* min obtained with ColeColeIII.**

predicted by using mixing formulas over the entire range of frequencies (Fig. 8), we proposed to frame the MW characterization of Carasau bread dough within a DS model. The dielectric permittivity data at $t = 0$ min, were fitted using modified Debye models with one, two, and three poles, and Cole-Cole models with one to four poles, for a total of seven models in order to find the best model to interpret the dielectric response of this food material. From Fig. 8, it is possible to notice that the Debye models, for any number of poles, present relatively high errors on the real part (from ∼8.8-34%), and very large errors on the imaginary parts. The large discrepancy is due to the resonant behavior of the Debye models [46], [49]. The fitted curves, against the experimental data, are reported in section SM1 in the Supplementary Material. Moving to a single-pole Cole-Cole model strongly reduces the error on both the average, relative percentage error of the imaginary and real parts of the permittivity (Fig. 8.a). The reduced error is due by Eq. (6) which can account for interacting dipoles and for the presence of a distribution of non-exponential relaxation times, which merges and broaden the dispersion curve [50]. By increasing the number of poles, the minimum of both errors is found for the three-pole Cole-Cole model, as shown in Fig. 8.a. Taking into account the combined standard deviation inherent to the dielectric measurement, the agreement with the measured dielectric permittivity is remarkable, as shown in Fig. 8.b.

The comparison of the complex dielectric permittivity of the three doughs samples from Tab. 1, prepared with three different semolina batches, is shown in Fig. 9. The variation during the 40 min of the first leavening step (Fig. 1) is reported. Furthermore, the time variation at the main three ISM frequencies of 915 MHz, 2.45 GHz, and 5.8 GHz is shown. From Fig. 9 we can observe that the three Carasau doughs characterized in this study are very similar. By comparing Fig. 9.a.i-9.c.i, the food materials present a real part of the relative dielectric permittivity of about 32 and 18 at 500 MHz and 8.5 GHz, respectively, whilst the out-of-phase component of the permittivity ranges from ∼25 to about 8, in the same frequency range, at the start of the leavening (t = 0 min). The real part of the dielectric permittivity is very close to the values measured by [20]. In fact, at 1 GHz, values of 28±1 and 26±2 at 2 GHz for normal, wet, and flour-rich doughs were reported [20]. Furthermore, the measured permittivity

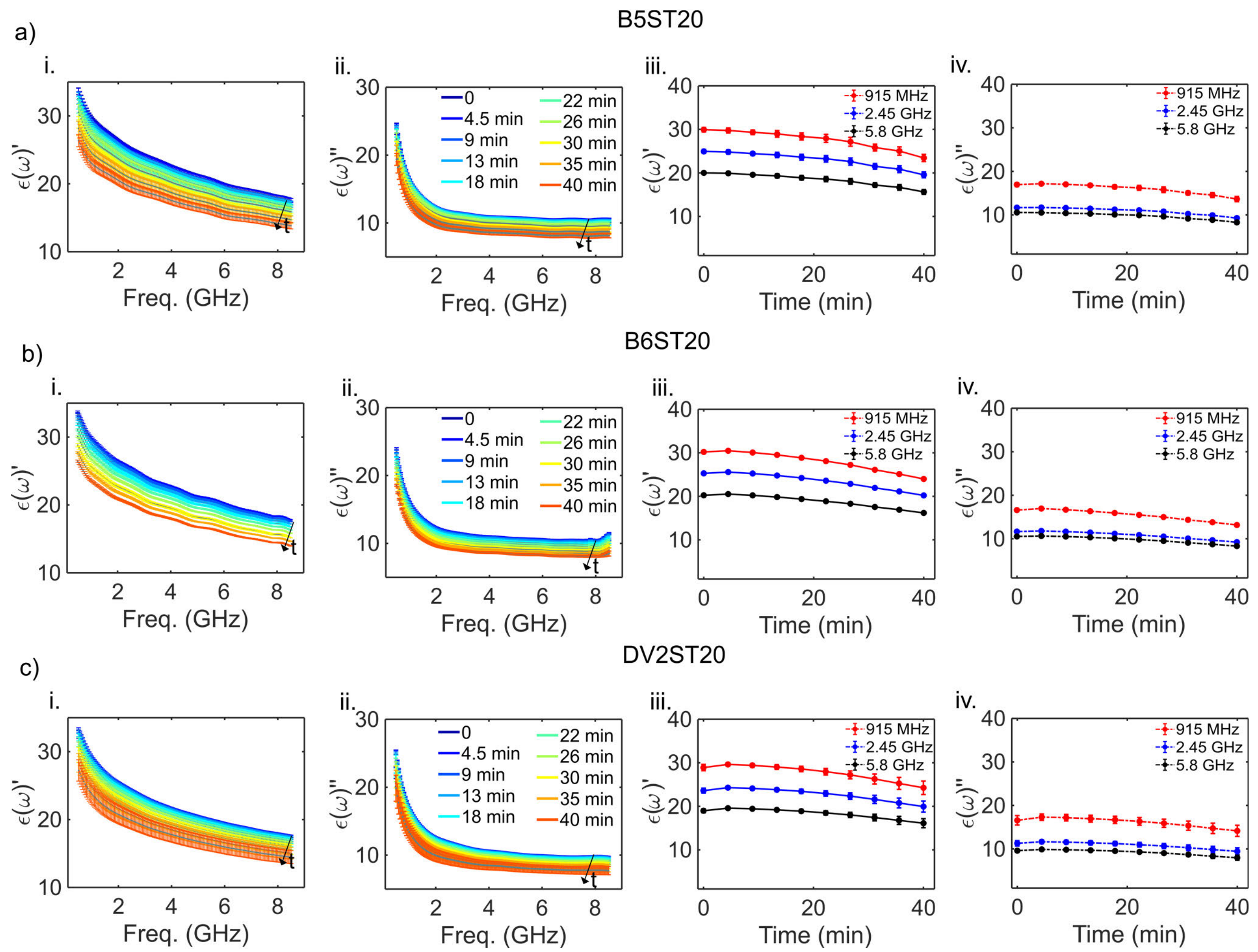

FIGURE 9. For the three Carasau doughs: a) B5ST20, b) B6ST20 and c) DV2ST20 the curves acquired at different time during leavening for the i) real part of the permittivity, ii) the imaginary part of the permittivity are shown, whilst in iii) and iv) the real and imaginary part of the permittivity at the three ISM bands of 915 MHz, 2.45 GHz and 5.8 GHz are reported as a function of the leavening time.

values of Carasau doughs are of the same order of magnitude of the permittivity values of cookies doughs (less than 10% difference) [54], despite the spectra have different shapes and features (Fig. 9.a-9.b).

The model parameters were retrieved for the samples B5ST20, B6ST20, and DV2ST20, by using the fitting procedure described in Sect. II.E are reported in Tab. 1. The standard deviation was evaluated considering three repeated preparations and measurements for each sample. From the perspective of the DS model used to interpret the microwave response of Carasau bread doughs, as can be seen in Tab. 1, we can highlight that the dielectric constant at optical frequencies is 3.354 ± 0.360, which tends to the real permittivity values of the raw semolina wheat (Fig. 5). The dielectric strengths of the three poles ($\Delta\varepsilon_1$, $\Delta\varepsilon_2$, $\Delta\varepsilon_3$) are quite similar. Indeed, the first two values are equal to, respectively, 36.785 ± 2.994 and 41.264 ± 2.856, on average (Tab. 1). The third pole has a lower value of 29.268 ± 0.160. It is worth noting that the real permittivity of foods is almost

TABLE 3. Coefficients for the three-pole cole-cole model AT T = 0 MIN.

Param.	B5ST20	B6ST20	DV2ST20	Mean ± Std.
ε_∞	3.113 ± 1.016	3.298 ± 1.180	3.434 ± 0.392	3.354 ± 0.360
$\Delta\varepsilon_1$	32.273 ± 4.080	33.870 ± 6.152	35.809 ± 9.986	36.785 ± 2.994
τ_1 (µs)	57.29 ± 5.152	60.325 ± 3.44	65.790 ± 9.423	60.124 ± 5.353
γ_1	0.256 ± 0.021	0.251 ± 0.034	0.261 ± 0.031	0.255 ± 0.017
$\Delta\varepsilon_2$	44.312 ± 1.411	38.08 ± 1.958	39.167 ± 2.358	41.264 ± 2.856
τ_2 (ns)	526.784 ± 15.752	539.173 ± 17.921	555.575 ± 16.595	541.714 ± 16.105
γ_2	0.518 ± 0.013	0.447 ± 0.153	0.535 ± 0.053	0.523 ± 0.029
$\Delta\varepsilon_3$	29.115 ± 1.274	30.349 ± 2.588	29.574 ± 1.552	29.268 ± 0.160
τ_3 (ps)	20.397 ± 0.879	22.241 ± 0.789	26.953 ± 0.630	22.582 ± 3.785
γ_3	0.320 ± 0.016	0.334 ± 0.025	0.381 ± 0.081	0.341 ± 0.010
σ_{dc} (S/m)	0.558 ± 0.022	0.548 ± 0.025	0.557 ± 0.032	0.544 ± 0.005

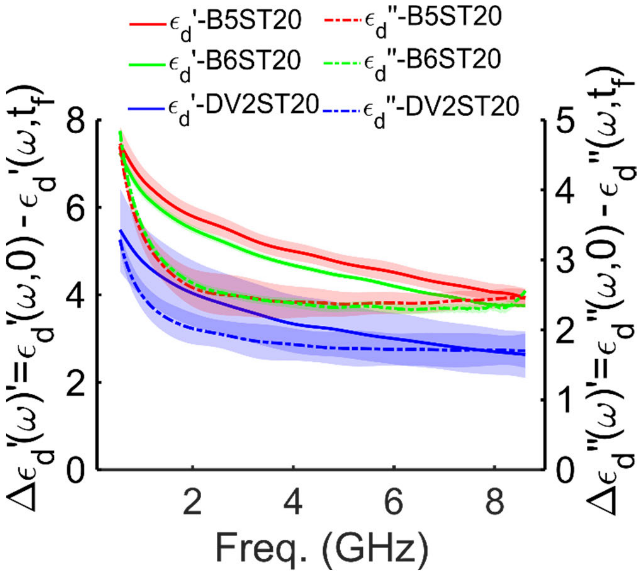

FIGURE 10. Differences in the real and complex Carasau dough spectra at the initial time of leavening (t = 0, i.e., $\varepsilon'(\omega, 0)$ and $\varepsilon''(\omega, 0)$) and at the final time ($t = t_f = 40$ min, i.e., $\varepsilon'(\omega, t_f)$ and $\varepsilon''(\omega, t_f)$) for the samples B5ST20, B6ST20 and DV2ST20.

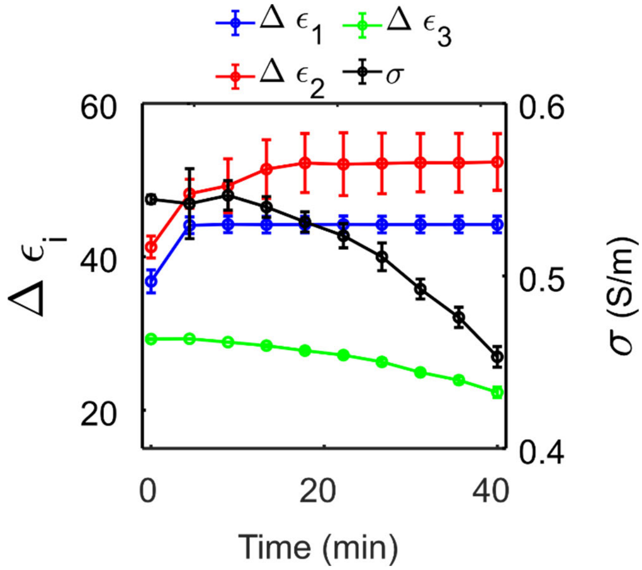

FIGURE 11. Variation of the parameters ($\Delta\varepsilon_1$, $\Delta\varepsilon_2$, $\Delta\varepsilon_1$, and σ, in S/m) of the three poles Cole-Cole model proposed for the Carasau doughs during the first leavening.

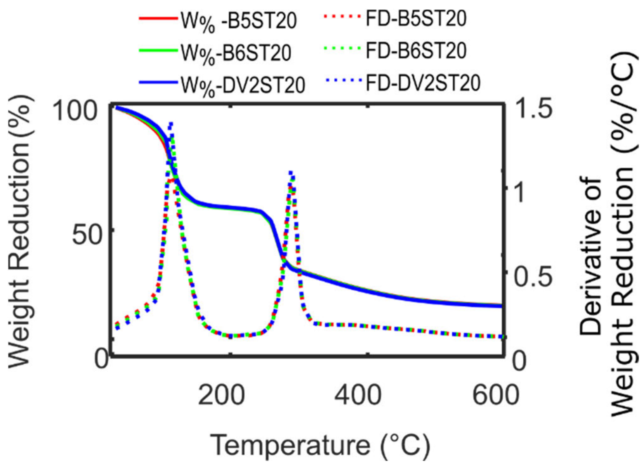

FIGURE 12. Weight reduction ($W_{\%}$, %) and the first derivative of weight reduction ($\%\cdot C^{-1}$) as a function of the temperature for the three doughs (B5ST20, B6ST20, DV2ST20), prepared with three different batches of semolina, with standard composition (Semolina 65.36%, Water 33%, Yeast 0.048%). Each curve is the average of three independent TGA runs.

exclusively defined by water content and its interaction with other components in foods (i.e., protein, starch, etc.) [21], therefore, the $\Delta\varepsilon$ values can be related to the microstructure and texture of the dough (Fig. 1). Indeed, the static permittivity decreases due to the action of the depolarizing internal electric fields, the dielectric saturation, as well as following the permanent dipole orientations in presence of the strong Coulombic field of the small ions dissolved in the liquid dough phase (i.e., Na+, Cl-) [55]. Despite this evidences, the main differences are due to the diverse relaxation characteristic times (τ_1, τ_2, τ_3) and the broadening parameters (γ_1, γ_2, γ_3). In fact, from Tab. 1, we can notice that the first relaxation mechanism is 60.124 ± 5.353 μs, whilst the second time is around 541.714 ± 20.105 ns, but the last damped relaxation mechanism is on the order of 22.582 ± 3.785 ps. The dipolar relaxation times can be related to the moisture content and, hence, to the existence of free or bound water in the analyzed doughs [41], [44]–[46], [50], [58]–[60]. In the γ-dispersion region (i.e., around 2.45 GHz) [21], [45], the signature of the dielectric properties of the dough can be ascribed to the water content (Fig. 9.iii and 9.iv). At these MW frequencies, the larger relaxation times (τ_1, τ_2) can be associated with bound water, i.e., water molecules trapped, adsorbed in regions where the content of hydrophilic components are highly concentrated (Fig. 1) [55]. The lower relaxation time, of tens of ps, could be correlated to the rapid rotation and orientation mechanism of water dipole in the hydration region, far from the interfaces (Fig. 1), as done with biological tissues [56]. Further analysis is needed to fully elucidate these contributions. As regards the broadening parameters, any of the Cole-Cole models do not fall in the limiting Debye case, since values of about 0.255 ± 0.017, 0.523 ± 0.029, and 0.341 ± 0.010 are obtained (Tab. 1). Therefore, from these findings, we can infer that the dispersion characteristic of the Carasau bread doughs can be effectively interpreted, from a macroscopic point of view, as a complex system with non-exponential relaxation laws [50]. Finally, the static conductivity of the doughs is 0.544 ± 0.005 S/m. Since the starch and gluten present in the dough microstructure are known to have a poor contribution to the electrical conductivity and the measured loss [20]–[24], this value is imputable to the presence of water and free salts. This electrical response is also reported by ref. [57].

Although having presented the MW DS model for interpreting the electromagnetic behavior of Carasau bread doughs, the material under test is not a static one. A goal of this work is also to evaluate if the properties of the dough undergo relevant changes during the crucial first leavening step, and then to study and model the variations to develop a framework to design an electromagnetic device for monitoring and empowering the bread manufacturing process. From Fig. 9 we can observe that the MW properties of the Carasau bread doughs change during the first 40 min of leavening. In detail, from Fig. 9, we can see that both the real and imaginary parts of the dielectric permittivity decrease as time increase. The decrease in both the real and imaginary part of the dielectric permittivity is coherent to the results of Zuercher *et al.* [20], who reported a difference of 1 unit of permittivity after 175 s and a larger decrease of 2 units of the imaginary part in the same timespan for wet doughs. Our measurements are performed in a larger time span and therefore the variation is more significant, as shown in Fig. 10 for the three samples.

The decrement of the properties can be ascribed to several mechanisms. In fact, since the proportion of carbohydrate to water increases, as the microstructure is changing [11]–[14], [30], a decrease in permittivity of the mixture, according to the power-law model, can justify this trend. Since high moisture-content foods are known to exhibit $d\varepsilon/dT < 0$, for temperatures between 0°C and 60°C [46], it is questionable to ask if the variations are due to temperature changes. However, the temperature of the dough sample, in the considered timespan, slightly increases (of ∼1°C) from room temperature [11]–[14], [30]. The temperature variation is not sufficient to justify the changes observed in the dielectric permittivity curves shown in Fig. 9 and Fig. 10.

The loss of water due to dehydration could be another factor for the permittivity decrease [44]. However, from the permittivity spectra from Fig. 9.iii and Fig. 9.iv, the trend of variation of the MW properties with time has an almost linear or a very slow exponential trend. Furthermore, it is known that the dough undergoes volume change [30], porosity modification from 20% to 60% values in 60 min, due to the formation of air bubbles [61].

To elucidate these aspects, we performed the fitting for every time of measurements (0, 4.5, 9, 13, 18, 22, 26, 30, 35, 40 min) and evaluate in terms of the proposed Cole-Cole model how the changes could be described by its parameters. The findings are reported in Fig. 11. We noticed that the dielectric constant at optical frequencies, the relaxation times, and the broadening parameters can be assumed to be constant, as well as that the variation of the dielectric properties can be effectively interpreted by the variation of the static dielectric permittivities of the poles and by a decrease in the electrical conductivity, as shown in Fig. 11. In detail, the terms $\Delta\varepsilon_1$ and $\Delta\varepsilon_2$ increase by ∼19% and 16%, respectively, and saturates after ∼10 min after the first leavening started. The parameter $\Delta\varepsilon_3$ presents a larger variation, which is fit to a linear decrease described by the linear law $\Delta\varepsilon_3(t) = 29.799\text{-}0.1748\cdot t$ ($R^2 = 0.9747$). Despite this narrow variation, the electrical conductivity has a larger variation, reducing its value up to 0.4531 S/m in 40 min, and with a non-linear trend. The conductivity decrease, relying on the average values of the three doughs, was modeled as $\sigma_c(t) = 0.5455\cdot\exp[(t-t_0)^2/2c^2]$ ($R^2 = 0.9554$), where $t_0 =$ 2.584 min and $c = 60.798$ min. The exponential reduction of the electrical conductivity can be related to the dehydration process [44] and the carbohydrates network formation, i.e., the conductivity decrease can be justified by the reduction of the relative amount of the free water, in which ions are dispersed, in the dough volume. The reduction of electrical conductivity at MW frequency can be the more relevant parameter for driving the design of an electromagnetic device for monitoring and assessing the first leavening of Carasau doughs, providing an engineering tool for empowering the industrial process.

From Fig. 10, we can observe that the average variation of the electromagnetic properties of Carasau dough are very similar for the B5ST20 and B6ST20 samples, which were manufactured from the same brand of semolina, but in different batches. In particular, the imaginary permittivities of the two samples are almost identical (<1%), within the experimental uncertainty. However, the average variation of the dielectric permittivity of the samples DV2ST20 is quite different from the B5ST20 and B6ST20. The discrepancy in the real and imaginary part of the permittivity is concerning, given that the raw, unmixed semolina batches presented very similar MW properties (Fig. 5) and the thermogravimetric analysis supported these findings (Fig. 6).

In this framework, we performed the TGA on the doughs, after 40 min of leavening, and the findings are reported in Fig. 12. In a way similar to the thermogram of Fig. 6, the average curves of the three different samples are very similar, thus indicating that the batches, from a thermal point of view, are almost identical.

IV. CONCLUSION

This work dealt with the characterization of Carasau bread doughs during leavening using microwave spectroscopy. The traditional Carasau bread is a typical product whose industrial production is facing the problem of keeping pace with the transition to the Industry 4.0 paradigm. The more critical aspects in the manufacturing process were identified in the raw materials kneading and the dough first leavening [8], [11]–[14]. To this aim, we performed open-ended coaxial probe measurements from 0.5 to 8.5 GHz on the raw materials and the Carasau bread doughs, monitoring the leavening. Our measurements were used to investigate the feasibility of predicting the dough properties by using the raw material properties. We tested different mixing formulas, i.e., Maxwell Garnett, Bruggeman, Lichtenecker, and the Power-Law model, and found that the Power-Law model could describe the dough permittivity from about 2.45 GHz to 8.5 GHz, with a maximum error of about 5%. We also proposed and tested Debye and Cole-Cole models with a different number of poles, for predicting the dielectric dispersion of Carasau dough. The best model was found to be a three-pole Cole-Cole model, covering the entire frequency range with an average error of about 2%. The modeling was extended by fitting the variation of the dielectric spectra of the doughs during the leavening. We found that the overall decrease in the electromagnetic properties can be justified by a slight reduction of the static dielectric permittivity and a stronger decrease in the electrical conductivity. These changes were associated with the decrease in water content and the carbohydrates network formation, as confirmed by thermogravimetric analysis. The gained knowledge can foster the development of an electromagnetic device for empowering the Carasau bread manufacturing process, in terms of quality control or process scale-up. In particular, dedicated microwave sensors, possibly miniaturized [38], could be designed [23]–[25]. For instance, the problem of designing and testing planar, microstrip resonators for the accurate determination of the leavening or wideband devices to track water content in the dough could be faced in future works.

Next works must deal with the problem of characterizing how the dough composition and the variation of raw materials (e.g., amount of salt, yeast) would affect the dielectric spectra at microwave frequencies, while also considering more types of semolina batches to better understand the product variability. Future work could also deal with the exploration of higher frequency range (e.g., from 10 GHz to millimeter-wave range).

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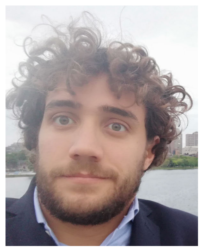

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