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1 TITLE OF THE PAPER:

2 "Dynamic measurement of dielectric properties of food snack pellets during microwave expansion"

3 by

4 José D. Gutiérrez [a*], José M. Catalá-Civera [a], John Bows [b], Felipe L. Peñaranda-Foix [a]

5 [a] Instituto ITACA. Universitat Politècnica de València. Camino de Vera s/n.46022 Valencia, Spain.

6 E-mail: jdgutierrez@itaca.upv.es, jmcatala@dcom.upv.es, fpenaran@dcom.upv.es

7 [b] PepsiCo International UK & Europe R&D, Feature Road, Thurmaston, Leics. LE4 8BS, UK.

8 E-mail: john.bows@intl.pepsico.com

9 * Corresponding Author. E-mail: jdgutierrez@itaca.upv.es

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13

14 ABSTRACT

15 *The in situ dielectric properties of a starch-based food pellet have been measured during microwave*
16 *expansion. A dual-mode cylindrical cavity allowed simultaneous microwave heating and dielectric*
17 *measurements of a single pellet inside a quartz tube, ensuring uniform heating during microwave*
18 *processing. The cavity included additional measurement devices to correlate the dielectric properties*
19 *with the main parameters of the expansion process, such as temperature, expansion time, pellet volume*
20 *and absorbed power.*

21 *A commercially available snack food pellet was used as the test material for expansion experiments.*
22 *Results indicated that dielectric constant (ε') and loss factor (ε'') increased during heating, reaching a*
23 *threshold value of ε'=12.5 and ε''=5.2, around a temperature of 115ºC when the material expanded and*
24 *the dielectric properties dropped abruptly due to the loss of water content and the increase in size.*

25 *This measurement procedure may provide useful material science information to improve the overall*
26 *design of starch-based food pellets processed by microwaves.*

Vector network analyzer (VNA), expansion index (EI), cavity perturbation method (CPM), dielectric constant (ε'), loss factor (ε'').

Keywords: food pellets, microwave heating, microwave expansion, foaming, dielectric properties, cavity perturbation method

1. INTRODUCTION

Next generation snack foods offer manufacturers the ability to deliver new and improved consumer experiences through control of texture, shape and colour, and to reach consumers by non-traditional channels, such as food service, street food and vending. The manufacture of the food snack in an intermediate form, a shelf stable, glassy half-product termed a pellet, enables high volume/low cost manufacturing to be separated from finish drying of the consumer-ready final product. These two stages (Riaz, 2006) typically comprise: (1) manufacture of the shelf-stable glassy pellet, formed at low pressure to prevent expansion, to a moisture content 10–12% (hereinafter expressed in wet basis), and (2) finish drying, causing expansion to a moisture content 1–2% (the "finished" product). To obtain the finished product from the pellet, the expansion or foaming procedure is typically accomplished commercially by baking (Chen and Yeh, 2000), hot air puffing (Nath et al., 2007) or immersion of the pellets in frying oil (Osman et al., 2000). Domestic microwave ovens are also used for home finishing of some pellet forms such as pappadums.

Thermal technology is one of the most delicate aspects of food processing (Meda et al., 2005). In conventional heating, the material absorbs energy as a result of thermal gradients through convection, conduction and radiation. By contrast, microwave energy interacts directly with the material molecules, leading to an energy conversion to heat rather than heat transfer. Consequently, microwave energy can reduce processing times and energy consumption, thereby improving energy efficiency. When microwave energy is applied to starch-based materials, the factor that initiates the heating process is the water content (Boischot et al., 2003). Accordingly, as the starch matrix heats up and the temperature increases, water molecules are transformed into superheated steam, creating local high pressure. If the temperature is sufficiently high, the pellet matrix experiences a phase transition from a glassy to a rubbery state and, combined with the high superheated steam pressure, expands (Moraru and Kokini, 2003). If microwave heating is terminated at the appropriate time, the matrix reverts to a glassy state and the foamed air cells are retained due to the mechanical resistance of the matrix in the glassy state, generating a crispy or crunchy texture that appeals to consumers. Conversely, the matrix might burn if microwave heating is not terminated timely (Gimeno et al., 2004).

The detailed study of this singular heating process is particularly challenging due to the complex and rapid transformations that occur during the short time period in which the microwave expansion takes

place, even under irregular distribution of temperature and moisture. Despite this difficulty, there are several studies in the literature that can provide relevant information on the process. For example, Lee et al. (2000) analyzed the effect of gelatinization and moisture content of extruded starch pellets on some morphological and physical properties of the expanded products (puffing efficiency and bulk density, among others), finding that the optimal expansion by microwave heating was achieved when the starch was approximately half-gelatinized and the moisture content in the pellets was ~10%. Kraus et al. (2014) investigated the influence of microwave power, system pressure and sample mass on the volume expansion, moisture ratio and pore distribution of starch pellets during microwave vacuum processing. They determined that the surface temperature of the pellet largely depends on the moisture content, observing that as microwave absorption increases, the water removal and the kinetics for heat and mass transfer occur faster, inducing a higher number of nucleated vapor bubbles. A study carried out by Gimeno et al. (2004) analyzed the effect of xanthan gum and carboxymethyl cellulose addition to improve the mechanical and structural properties of extruded glassy corn pellets expanded by microwave heating. The authors concluded that a small addition (~1%) of these substances could improve the shape, structure, and texture of microwave-expanded corn pellets.

The majority of the experiments performed in these studies and other reported expansion trials made use of multimode microwave equipment, such as domestic home equipment (Camacho-Hernández et al., 2014; Lee et al., 2000; Zhou et al., 2006) or specialized laboratory equipment (Boischot et al., 2003; Gimeno et al., 2004). Multimode microwave chambers are known to produce uneven temperature distributions, especially in low moisture content products, which are affected by factors including oven cavity geometry, location of the sample and size of the workload. Moreover, multimode applicators lack specific information about the absorbed power by the samples. For example, Lee et al. (2000) reported a power of 700 W to expand 30 g of pellets for 70 s in a commercial furnace (RE-552N, Samsung), Zhou et al. (2006) applied 1000 W to expand 10 g of pellets in a microwave oven (R-8720M, Sharp), and Gimeno et al. (2004) expanded single pellets inside a laboratory apparatus (AVC-80 moisture-solids analyzer, CEM Corporation) at 600 W for different periods of time, up to 60 s. On the other hand, experimental analysis relies on measurements before and after the expansion process, without monitoring the behaviour during the heating process.

The precise knowledge of the food pellet properties and process-related parameters is important to fully understand the heating of such materials by microwave energy. In particular, the dielectric properties

define the interaction of dielectric materials with microwaves and, consequently, these parameters are an essential variable to describe the heating behaviour of food pellets when applying microwave fields (Nelson and Datta, 2001). The permittivity is defined as a complex value. The real part (or dielectric constant) is related to the ability of a material to store energy when it is polarized under alternating electric fields. The imaginary part (or loss factor) quantifies the capacity of the material to absorb this stored electromagnetic energy and dissipate it into heat. Since the loss factor of food materials is highly correlated with the amount of water (Meda et al., 2005), the moisture content of pellets will have a significant effect on the microwave heating process.

The influence of moisture content on the dielectric properties of ground pellets has been reported recently by Kraus et al. (2013), who used a cylindrical resonant cavity and the cavity perturbation method (CPM). Some attempts have also been made to measure dielectric properties of pellets during foaming for packaging applications, as described in Peng et al. (2013). In the latter work, the microwave instrument described in Nesbitt et al. (2004) was also used for thermal and dielectric measurements. The volume of pellets was calculated before and after the expansion, however, the influence of volume changes in the dielectric calculations was not provided.

The aim of the present study was to determine in situ the evolution of dielectric properties of starch-based food pellets during expansion as they are heated by microwave energy. The microwave apparatus used in the study was based on the dual mode cylindrical cavity described in (Catalá-Civera et al., 2015), which is able to heat and measure simultaneously with two different microwave sources. This setup was modified including some additional devices to fit the specific needs of the expansion procedure. The experimental study was carried out using a single pellet placed in the uniform field of the cavity to maximize the even absorption of microwave energy during heating and expansion. Unlike previous approaches, the temperature, microwave absorbed power, volume and processing time was also monitored in situ during the expansion process and correlated with the dielectric properties of the pellet. The calculation of dielectric properties made use of an enhanced CPM with calibration coefficients of different volume to increase the accuracy of in situ measurements.

The findings obtained using the experimental system described here may be useful for a better understanding of the kinetics and processing conditions of the expansion process under microwaves. The dielectric properties may be valuable parameters for the modeling and design of energy efficient

119 microwave heating chambers. Moreover, these results may help food researchers to further adapt the
120 overall properties of this type of snack to the conditions needed during microwave heating.
121
122 ## 2. Materials and Methods
123 ### 2.1. Food pellets
124 A pellet used to make a commercially available snack food was used as the test material. The pellet (11–
125 13% wet basis moisture content) was formulated primarily from potato flakes and was designed to be
126 finished by factory frying in hot oil to a final moisture content of 2%. Although not formulated for
127 microwave heating, the pellet has been found to expand reasonably well in domestic microwave ovens.
128 For example, 100 g of pellets of approximately 30 mm in length and 3 mm in diameter (placed in a plastic
129 tub), when heated at full power for 120 s yielded around 70% of pellets which were fully expanded, with
130 the remaining pellets under-expanded or burned. The finished product when fully expanded was 50–60
131 mm in length and 6 mm in diameter.
132 Prior to expansion with the microwave system described below, pellets of circular cross-section ~3 mm in
133 diameter were cut into pieces of 10–11 mm in length with flat sides and equilibrated to room temperature
134 (~23ºC). The approximate weight of a pellet was 0.09 g. The moisture content of pellets was determined
135 from the weight loss after heating to 103ºC in a convection oven (Heraeus WU 6100) for 72 h.
136
137 ### 2.2. Experimental microwave set-up
138 The microwave experiments were conducted using the instrument described in Catalá-Civera et al.
139 (2015), with appropriate modifications to account for the singular shape and behaviour of the pellet.
140 The microwave applicator consisted of a cylindrical cavity designed to simultaneously operate with two
141 different resonant modes. The Transverse Electric (TE_{111}) mode was used for heating the pellet sample
142 (heating mode) using high-power microwave signals (~150 W), and the Transverse Magnetic (TM_{010})
143 mode provided the information to perform the calculation of the dielectric properties (testing mode). Both
144 operating modes have a uniform electric field in the cavity center where the pellet sample is placed.

Figure 1 shows a schematic view of the microwave applicator. The cavity has a diameter of 104.92 mm and a height of 85 mm and includes several apertures for feeding the cavity in each mode and placing infrared thermal and video cameras.

The microwave source used to feed the heating mode of the cavity comprised a vector network analyzer (VNA) and a 50 dB gain solid-state amplifier working from 2.2 to 2.6 GHz. The output signal of the VNA was guided to the amplifier and introduced into the cavity through an N-connector port placed at the sidewall and terminated in an electric probe. The VNA was able to perform several frequency sweeps per second, allowing about 5 heating cycles per second depending on the bandwidth of the signal. The average absorbed microwave power was determined by measuring the forward and reflected microwave signals in this port of the cavity within the range of frequencies delivered by the microwave source, as described in Catalá-Civera et al. (2015).

To ensure efficient power delivery to the microwave cavity during processing of the dielectric sample, an automatic procedure adjusted the sweep frequency band of the input VNA source to provide the desired sample heating rate, as described in Catalá-Civera et al. (2015).

The testing mode used a second VNA calibrated in the frequency range 1.9–2.2 GHz and a cross-coupling filter to avoid disturbance signals received from the heating mode. The low-power signal was coupled to the cavity by another electric probe placed at the central position of the bottom wall by an SMA connector. The input return loss measured in this frequency range was employed to determine the resonance parameters required for dielectric calculations according to Canós et al. (2006).

A single pellet sample was positioned vertically at the bottom of a quartz tube holder of 10 mm internal diameter, which was introduced into the reactor through a non-radiating cylindrical access at the top of the cavity. To prevent water condensation on the quartz holder that might influence dielectric measurements when the moisture of the pellet was released during expansion, a venturi-based suction device was placed on the top of the upper cover in the cavity as shown in Figure 1. An infrared thermal camera with an accuracy of ±2ºC (Optris PI 160, Optris, Berlin, Germany) was also placed at the top of the suction system to monitor the surface temperature of the pellet during microwave processing. The emissivity was fixed to 0.96 in the camera by comparing the temperature measured by the camera with the temperature of a thermocouple sensor in contact with the sample. The configuration of the camera was set to measure the maximum surface temperature on the top of the material.

To evaluate the volume changes during the heating process, the volume expansion index (EI) was defined as

$$\mathrm{EI} = \frac{V_i}{V_0} \qquad (1)$$

where V_0 is the initial volume of the preprocessed pellet and V_i is the time-dependent volume of the pellet by microwave processing.

The pellet volume V_i evolution during the heating process was calculated from the video signal of a miniature video camera (15 × 15 × 8 mm dimension) with a 2.2 μm pixel size (MU9PC-MH, Ximea, Münster, Germany) attached to the lateral side of the cavity. Analysis of the 2D frames recorded during microwave heating allowed the in situ identification of the pellet expansion profile by in-house image process software with MATLAB libraries, including multi-point edge detection and filtering functions. The total height of the pellet was divided into 30 circular cross-section cylinders with diameters calculated from the profile of the video frames, and the final volume was determined by adding the separate volumes of the small cylinders together.

All components of the measurement set-up system, including network analyzers, video camera and thermal camera, were controlled by in-house developed LabVIEW software (LABVIEW 5.1, National Instruments, Austin, USA) to achieve a full automatic process for heating and testing operation modes.

2.3. Cavity Perturbation Method (CPM)

The CPM is possibly the most popular technique for measuring the dielectric properties of materials, among them food products, at microwave frequencies (Risman and Bengtsson, 1971; Lyng et al., 2014).

The method entails the resonance measurement of a microwave resonant cavity before and after the insertion of a sample of the material under test. The fundamental concept of this technique is that the introduction of a small sample in the cavity barely perturbs the electromagnetic field around the material. Under this assumption and making use of the quasi-static approximation (Altschuler, 1963), the dielectric constant ε' and the dielectric loss factor ε'' of a material under test (here, the food pellet) can be related to

199 the relative shift in the resonant frequency $\Delta f/f$ and the change in the Quality factor term $\Delta(1/2Q)$ by

200 (Khanna et al., 1974),

201

202 $$\varepsilon' = 1 + \frac{-\frac{\Delta f}{f}\left(\eta + N\frac{\Delta f}{f}\right) - N\left[\Delta\left(\frac{1}{2Q}\right)\right]^2}{\left(\eta + N\frac{\Delta f}{f}\right)^2 + N^2\left[\Delta\left(\frac{1}{2Q}\right)\right]^2} \quad (2)$$

203 $$\varepsilon'' = \frac{\eta\Delta\left(\frac{1}{2Q}\right)}{\left(\eta + N\frac{\Delta f}{f}\right)^2 + N^2\left[\Delta\left(\frac{1}{2Q}\right)\right]^2} \quad (3)$$

204

205 where: ε' = dielectric constant (dimensionless); ε'' = loss factor (dimensionless); f = resonant

206 frequency (s^{-1}); Q = quality factor (dimensionless); η = sample filling factor (dimensionless); N =

207 sample depolarization factor (dimensionless).

208 In (2) and (3), Δf refers to the difference of the resonant frequency of the cavity with the sample with

209 respect to the resonance without the sample. The shift in the resonant frequency and the change in the

210 Quality factor are written, respectively (Catalá-Civera et al., 2015),

211

212 $$\frac{\Delta f}{f} = \frac{f_{uts} - f_{ut}}{f_{uts}} \quad (4)$$

213 $$\Delta\left(\frac{1}{2Q}\right) = \frac{f_{ut}}{f_{uts}} \cdot \frac{1}{2} \cdot \left(\frac{1}{Q_{uts}} - \frac{1}{Q_{ut}} - \frac{1}{Q_{u0}}\frac{f_{uts}^2 - f_{ut}^2}{f_{u0}^2}\right) \quad (5)$$

214

215 where f_{uts} and Q_{uts} are the resonance frequency and Quality factor of the cavity, respectively, with the

216 sample holder containing the sample, and f_{ut} and Q_{ut} with the empty sample holder.

The presence of apertures in the cavity for sample insertion or video visualization requires additional corrections to the cavity resonance. However, in the microwave cavity shown in Figure 1, the fields in the holes remain virtually unaltered in the perturbed cavity because the dielectric sample is far from the holes. The effect of the insertion holes is therefore assumed to be canceled by the perturbation expressions (2) and (3) making use of equations (4) and (5), which involve relative deviations, and then f_{ut} refers to the resonant frequency including the insertion holes.

Parameters η and N ($N \in [0.1]$) represent the sample filling factor (which quantifies the relative electric volume sample/cavity) and the sample depolarization factor, respectively. These parameters depend on the specific geometry of the cavity, resonant mode and dielectric sample, and they are typically determined through calibration procedures by measuring reference materials with known permittivity (Roussy et al., 2001). In the case of food pellets, the samples are expected to reshape and expand considerably during microwave processing, thus the calibration procedure has to take into account reference samples of different size.

Three different samples with known dielectric properties, polytetrafluoroethylene (ε'= 2.03, ε''=0.0007), polyvinyl chloride (ε'= 2.93, ε''=0.023) and marble (ε'= 7.36, ε''=0.024) were used to perform the calibration. For each reference material, nine cylinders were machined in different sizes by varying diameters and lengths to achieve volumes from 30 mm^3 (10 mm length and 1.95 mm diameter) to 610 mm^3 (25.96 mm length and 5.47 mm diameter). Each material was introduced vertically into the quartz tube and positioned in the cavity for resonant frequency and quality factor measurements in the testing mode. For each volume, η and N parameters were minimized using equations (2) and (3), with the resonance measurements and their respective values of permittivity, and they are represented in Figure 2. As the figure shows, a straight line was sufficient to find this relationship despite the difficulty to machine precise samples of very small and specific sizes.

To verify the accuracy of the permittivity calculations by the CPM described above, different trials with cylindrical and non-cylindrical samples (circular cross-section with different diameters in height) with known permittivity and with a volume within the range 100–700 mm^3 were measured in the cavity at room temperature. From the discrepancies observed in the results with respect to the reference values, the average accuracy of the CPM was estimated to be in the range of 5% for the dielectric constant and ~10% in the loss factor within the range 10^{-2} through 10^{1}.

246 The accuracy was significantly higher as the precision in the calculation of the sample's volume was
247 improved. The volume measurement of the sample was therefore of the utmost importance to properly
248 determine dielectric properties from calibration parameters.

249

250 ## 3. Experimental Results and Discussion

251 Extensive experimental trials were carried out with the microwave system by heating pellets under
252 different microwave conditions and measuring the evolution of dielectric properties during microwave
253 expansion. The input source was configured to provide an average absorbed microwave power from 8 W
254 to 20 W to achieve approximate heating rates in the pellets from 2ºC/s to 24ºC/s.

255

256 ### 3. 1. Temperature and Volume (expansion index) during Microwave Expansion

257 Figure 3 shows the surface temperature and the evolution of the expansion index (EI) of two pellets as a
258 function of the processing time for two different microwave trials. The average microwave power
259 absorbed by the pellet samples and test cell (by ohmic or wall losses) was 12.4 W and 15.3 W, and the
260 heating rates were 3.6ºC/s and 7.1ºC/s, respectively.

261 At the beginning of the process, microwave energy heated the matrix through vibration of water
262 molecules and the temperature of the sample increased progressively (Moraru and Kokini, 2003).
263 Depending on the amount of moisture in the product, as the temperature increased the water molecules
264 were transformed to vapor (superheated steam) and accumulated at nuclei in the glassy matrix, and the
265 matrix underwent a phase transition from a glassy to a rubbery state (Boischot et al., 2003).

266 When the surface temperature was ~115ºC in both experiments, independently of the applied microwave
267 power the local water vapor pressure inside the nuclei exceeded the binding energy of the pellet matrix
268 and it was abruptly released from the matrix, causing expansion of the pellet. The expansion is clearly
269 appreciated in Figure 3 by an increase in the EI. At this point, the surface temperature of the pellet rapidly
270 increased to ~140ºC due to the higher inner temperature of the released superheated steam, and
271 subsequently increased at a slower rate during the short expansion process. Once the moisture was lost,
272 the final volume (EI) of the pellet remained constant and the surface temperature continued to rise
273 (~170ºC) until microwaves were ceased. At this stage, the matrix cooled and passed into the final

structure of a glassy state. If microwave heating continues long after the expansion is complete, samples begin to burn (Boischot et al., 2003). The temperatures measured during microwave expansion agree well with the surface temperature observations of expanded starch-based pellets in Boischot et al. (2003), Gimeno et al. (2004) and Peng et al. (2013). In these studies, expansion was reported to take place above its glass transition temperature (Tg) when the water vapor pressure inside the bubble is sufficient to overcome the resistance to its expansion (Boischot et al., 2003).

As shown in Figure 3, the time required for expansion, the duration of the expansion process and the calculated EI of the expanded sample clearly depended on the microwave absorbed power provided to generate the superheated steam necessary to overcome the constraining starch matrix. Whereas the duration of the expansion process in the sample processed with 12.4 W absorbed power was ~9 s and the final EI was around 6.2, the sample processed by 15.3 W required only 5 s to expand and the increase in EI was around 7.1. Numerous experiments were performed to establish the relationship between applied microwave power and expansion process kinetics. Figure 4 shows the EI and the time required to expand (i.e., from when microwave energy initiates until the material begins to expand) as a function of the average absorbed microwave power by the sample and test cell. When absorbed power levels around 10 W were supplied from the power source, pellet expansion began at around 60 s (energy E=600 J), achieving an EI of four times the initial volume and a moisture content of around 5%. When the absorbed power provided to the sample was increased to 20 W, the EI achieved was eight times the starting volume in only 6 s, with a final moisture content around 1–2% (E=120 J).

These results indicate that rapid heating (high heating rate) is essential for the microwave expansion process, which is consistent with the results reported in Camacho-Hernández et al. (2014) but contrast with the results of microwave foaming of extruded starch-based pellets reported in Boischot et al. (2003) with more emphasis in the energy threshold.

Figure 5 shows representative frames of the recorded expansion process and the profile of the pellet recognized by the edge-detection software during volume calculations. Microwave energy was switched on 10 s after the video started and the first frame (10 s) shows the pellet before processing. The pellet shape remained constant over the next 17 s and then the pellet began to increase in size when expansion commenced, as shown in the second frame (27 s). Depending on the experiment, expansion commenced in the material from the bottom, from the top or from the entire volume. The size of the pellet illustrated

303 in the last frame of Figure 5 (31 s) had increased from 10.7 mm in length and 2.95 mm in diameter to 17.6
304 mm in length and 6.26 mm in diameter, resulting in an EI greater than 7.

305

306 ### 3. 2. Dielectric Properties during Microwave Expansion

307 Concurrent with the microwave heating, temperature and volume measurements, the evolution of the
308 resonant frequency and Quality factor of the cavity in the testing mode was measured and employed to
309 determine the dielectric properties of the pellet sample during expansion according to the CPM procedure
310 described in the previous section.

311 Preliminary measurements were performed, progressively applying microwaves for short periods of time
312 (of several seconds) to analyze in detail the evolution of the superheated steam necessary for expansion.
313 Measured dielectric constant and loss factor of pellets at room temperature gave values around $\varepsilon'=5.7$ and
314 $\varepsilon''=0.9$, which are sufficiently high for efficient coupling of microwave energy.

315 Figure 6 shows the time evolution of the dielectric properties of a pellet when a microwave absorbed
316 power of 13.5 W was applied for periods of 2, 6, 11, 19 and 24 s. The heating rate in all cases was
317 approximately 5.5ºC/s.

318 When the microwave heating was applied for short periods (i.e., 2–19 s), the dielectric properties
319 (dielectric constant and loss factor) of the pellet increased mainly as a consequence of the increase in the
320 temperature of the water content in the matrix (Gimeno et al., 2004), as illustrated in Figure 6. If the
321 duration of the heating period was sufficiently short, the dielectric properties reverted to their initial value
322 when microwave heating ceased and the pellet cooled to room temperature. Thus, during these periods,
323 for pellet samples that were heated only with no expansion, the dielectric properties showed a direct
324 dependence on the temperature. The stabilization of the loss factor to the same value after each heating
325 period indicated that no water was released from the material therefore drying was negligible.

326 When the duration of the heating period was longer (i.e., >19 s), the dielectric properties increased with
327 the temperature of the pellet as before; however, if the temperature reached was higher than the threshold
328 temperature (~115ºC), glass-to-rubber transition occurred and the superheated steam had sufficient energy
329 to overcome the cell resistance, causing expansion of the pellet (Moraru and Kokini, 2003) accompanied
330 by a sudden decrease of the dielectric properties. When microwaves were ceased, the dielectric properties

were stabilized at very different values to those detected when microwave power was applied for short intervals since the final material had lost most of its moisture after expansion and its size was considerably greater (Sjöqvist and Gatenholm, 2007). For example, the loss factor was reduced to $\varepsilon''=0.015$.

Figures 7 and 8 show the dielectric constant and loss factor of the pellet samples for different microwave power heating continuous cycles. The expansion index of the trial corresponding to 9.8 W is also shown in Figure 7.

As shown in Figures 7 and 8, when the applied microwave power was low (i.e., power absorbed =8 W), the absorbed thermal energy was not sufficient for pellet expansion and the pellets were only heated. In this case, both dielectric constant and loss factor increased in value with the processing time and temperature, reaching saturation around $\varepsilon'=10$ and $\varepsilon''=3$.

When the applied microwave power was sufficiently high (power absorbed >9.5 W) the thermal energy increased the molecular friction of water dipoles such that the threshold temperature was reached (~115ºC, as shown in Figures 3 and 6), the vapor pressure overcame the matrix constraint and the pellet expanded (Moraru and Kokini, 2003). This is shown in Figures 7 and 8 as a progressive increase of both the dielectric constant and the loss factor with the processing time, and a fast decrease in both values during foaming.

Figure 7 also shows the EI of the trial corresponding to an absorbed power of 9.8 W, where it is possible to associate the duration of the expansion time represented by the period when EI changes (17.3 s) with the duration of the fast decrease of dielectric properties.

It can also be appreciated in Figure 7 and 8 that the application of higher microwave power levels led to a higher heating rate (Kraus et al., 2014), and the increase of dielectric constant and loss factor prior to expansion occurred more rapidly. Nevertheless, the dielectric properties of the pellets before expansion were very similar in most of the experiments, indicating also a threshold in these values ($\varepsilon'=12.5$ and $\varepsilon''=5.2$) below which expansion did not occur.

Especially relevant are the changes to the loss factor observed in Figure 8, lowering the value of $\varepsilon''=5.2$ to around $\varepsilon''=0.02$ after expansion. Because the loss factor of a food substance is closely related to its moisture content (Meda et al., 2005), the differences in the loss factor after expansion suggests that in

pellets expanded with lower absorbed microwave power more water molecules might have remained in the matrix during the expansion phase.

Figure 9 shows the final EI obtained in several trials at different power levels as a function of the measured loss factor just after expansion (around 160ºC) and cooled to room temperature. A clear relationship can be seen between these two parameters, with pellets with lower loss factor after expansion achieving higher EI, indicating that more moisture was expelled from the pellets in the expansion process and that the volumes are larger (moisture content from around 5% to 1–2%). If both curves are extended, there appears to be an EI trend to around 9 and 10 for this type of pellet.

The present study shows that the in situ measured dielectric properties of pellets and their relationship with microwave absorbed power, temperature, processing time, volume and moisture content are critically important to understand the microwave expansion of the starch-based food pellet.

In the first stage, superheated water was indicated by an increase of both dielectric constant and loss factor, reaching a threshold value of approximately $\varepsilon'=12.5$ and $\varepsilon''=5.2$. Below this value, the energy provided by the microwave system was not sufficient to overcome the energy required to generate steam, break the molecular bonding of water to the substrate and expand (Boischot et al., 2003)

The expansion process was observed as a fast decrease of dielectric properties, particularly the loss factor, indicating the release of water from the matrix and a change in size (Sjöqvist and Gatenholm, 2007). The expansion time, calculated from the duration of the decrease in dielectric properties, directly correlated with the duration of EI changes.

The achieved EI was found to be directly dependent on the loss factor after expansion, which appeared to indicate the amount of moisture released during the process (Kraus et al., 2013). With this relationship, it is possible to estimate the maximum EI that can be reached in the pellet.

4. Conclusions

The in situ and real time observation of dielectric properties of a potato-based snack food pellet during microwave expansion together with the simultaneous measurement of other parameters such as temperature and volume, have provided for the first time a solid experimental platform for investigation of the interaction of these materials with microwaves and the resulting expansion kinetics.

Dielectric measurements have been carried out in a dual-mode cylindrical microwave cavity with simultaneous heating and testing modes. The cylindrical shape factor of the pellet and finished product enabled relatively straightforward generation of calibration factors required for accurate dielectric measurements by the cavity perturbation technique.

The main process-related parameters are the minimum absorbed microwave power required to expand the pellet and the pellet expansion time. The expansion index has been correlated with dielectric measurements of the finished product.

The insights developed for microwave-based finish drying of the snack pellet can be used to refine the pellet expansion performance (e.g., to reduce the amount of under-expanded or over-expanded product), as well as to investigate how the pellet formulation itself is linked to microwave expansion performance.

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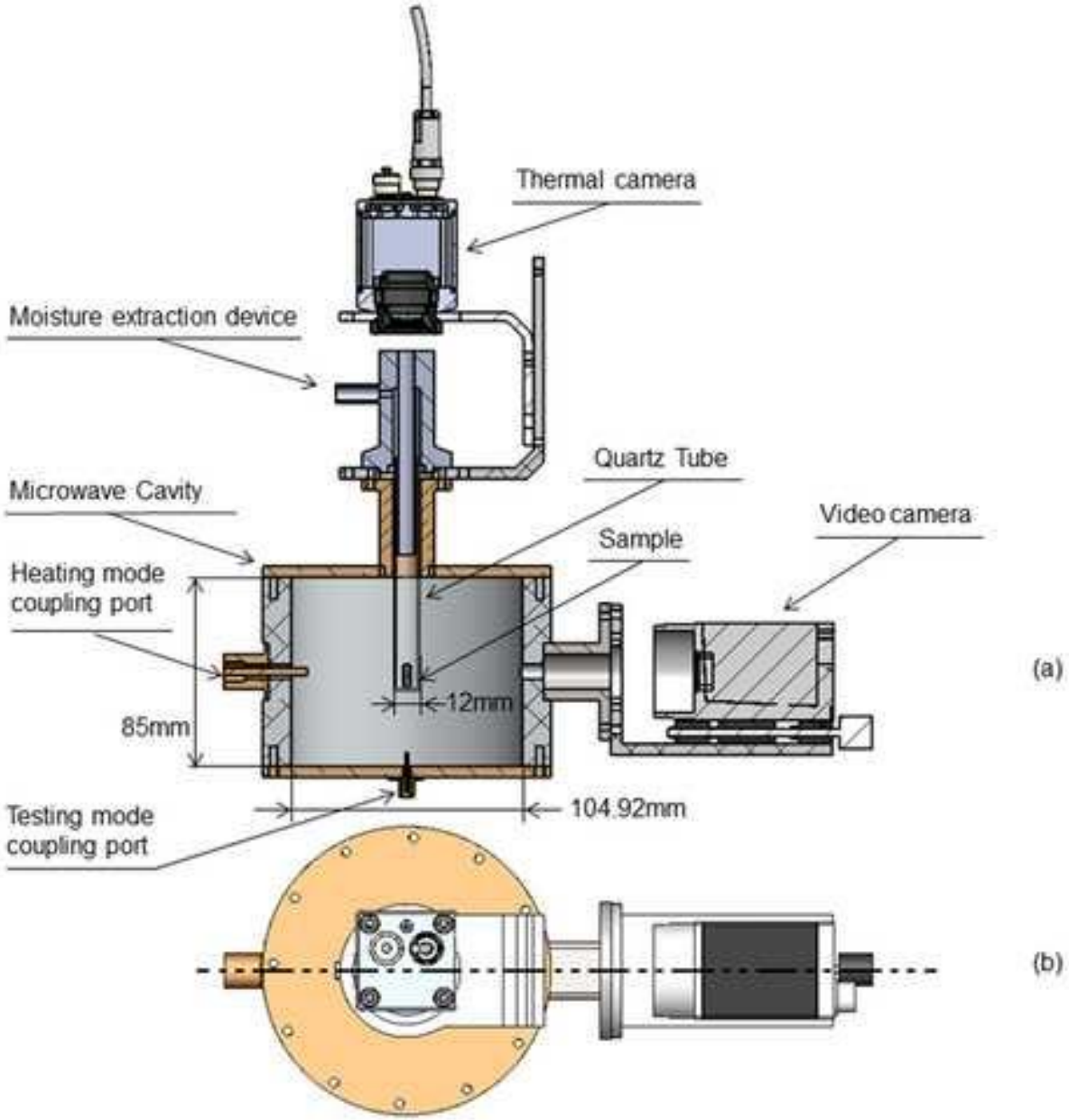

Fig. 1. Schematic view of the dual-mode cylindrical microwave cavity. (a) Front view of the cavity. (b) Top view of the cavity.

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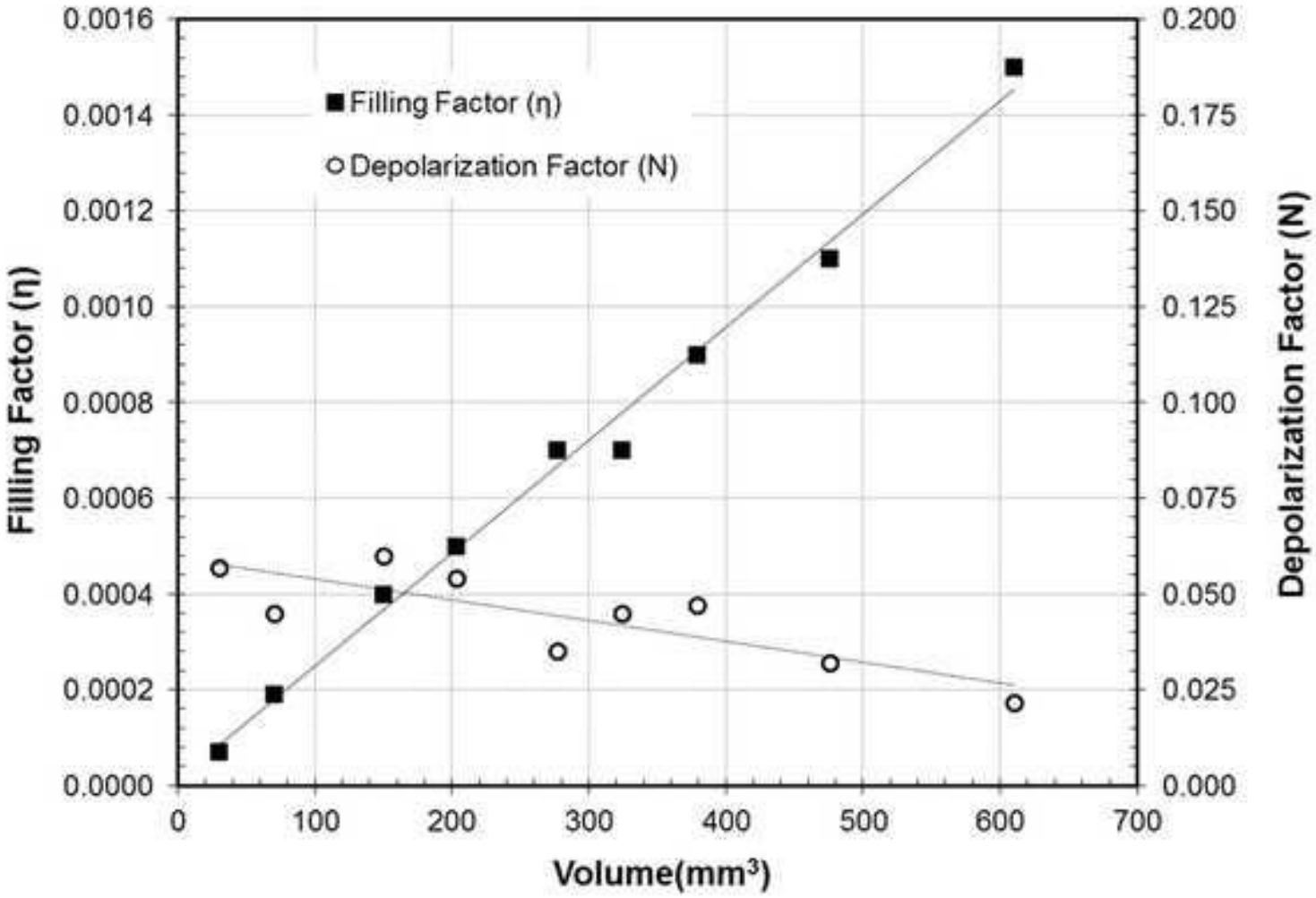

Fig. 2. Cavity perturbation method calibration parameters, η and N, determined with reference samples of different volume and known permittivity

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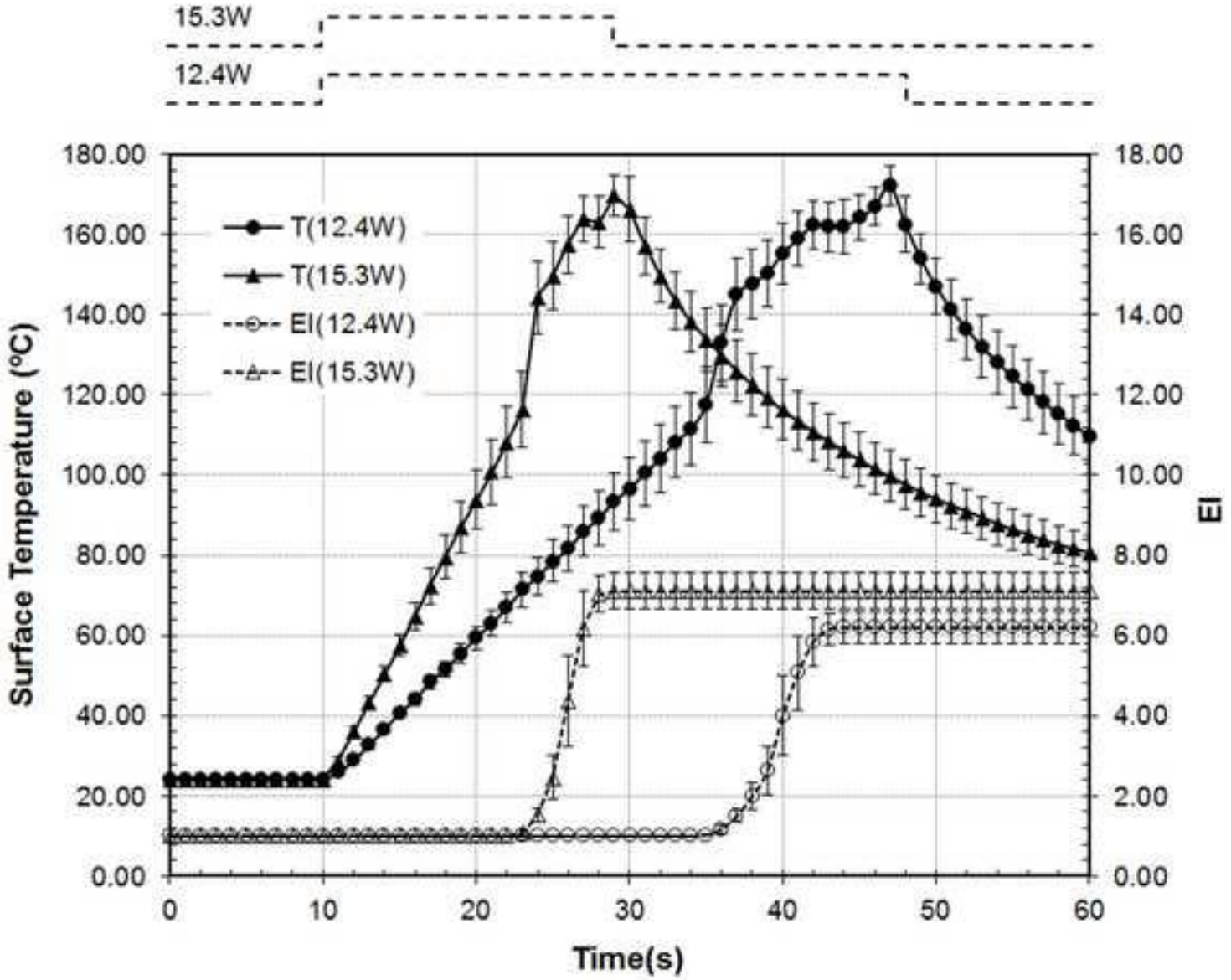

Fig. 3. Temperature and Expansion Index (EI) of pellet samples during two different microwave expansion conditions (MW power absorbed 12.4 W and 15.3 W and heating rates 3.6°C/s and 7.1°C/s).

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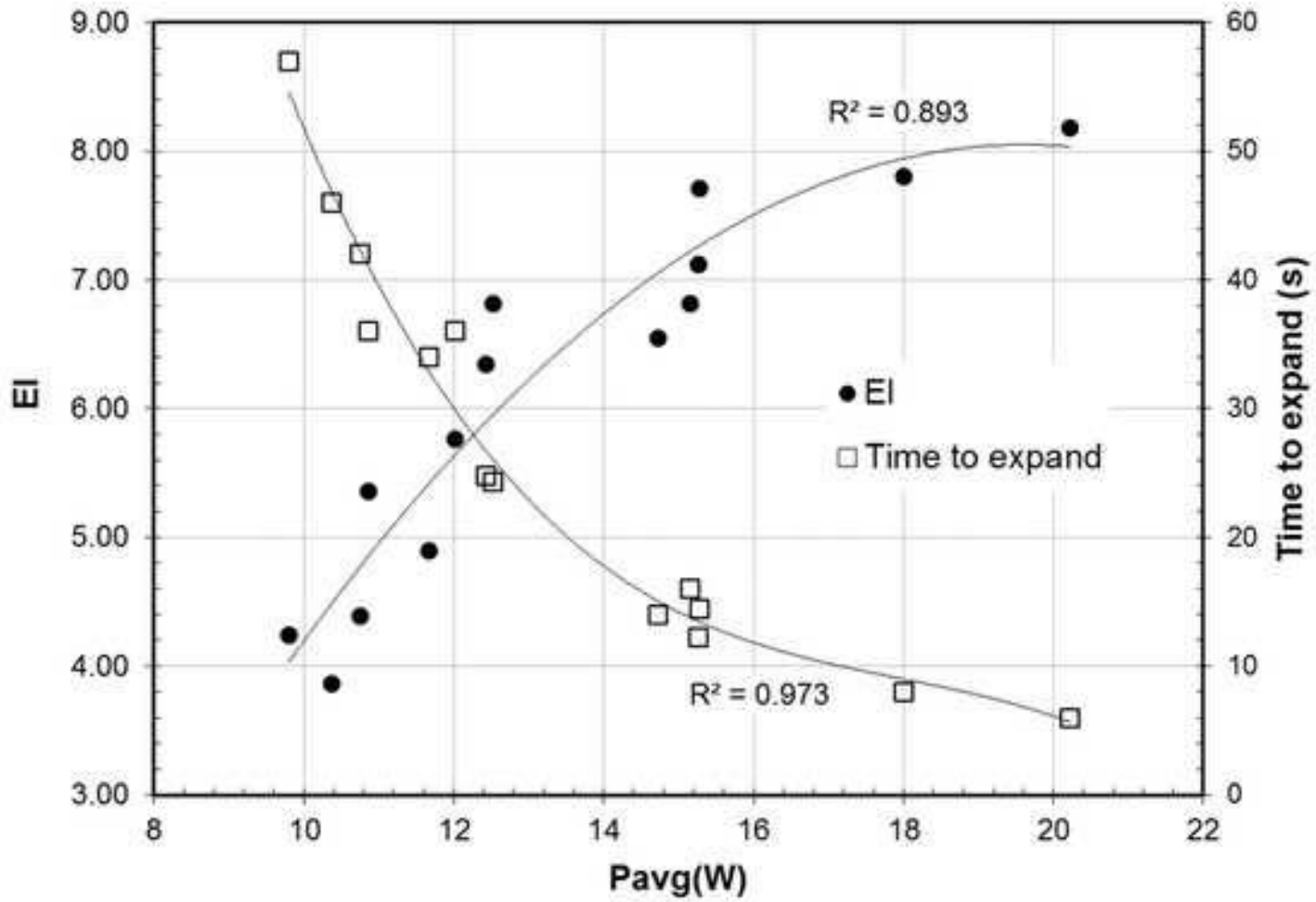

Fig. 4. Expansion Index (EI) and required time to expand as a function of the average microwave absorbed power applied to the pellet sample and test cell.

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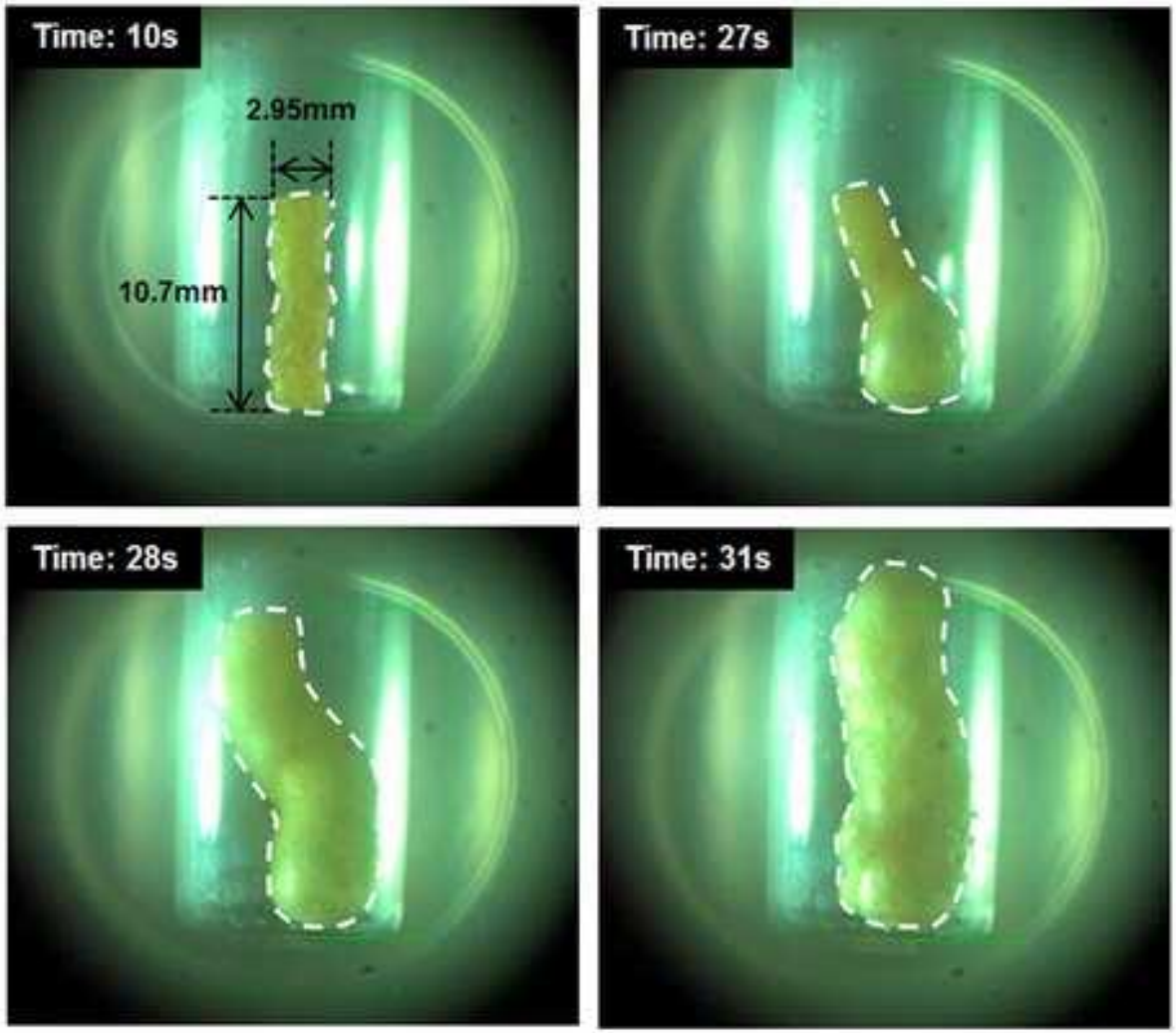

Fig. 5. Evolution of the profile and volume of the pellet during microwave expansion (MW absorbed power 14.1 W)

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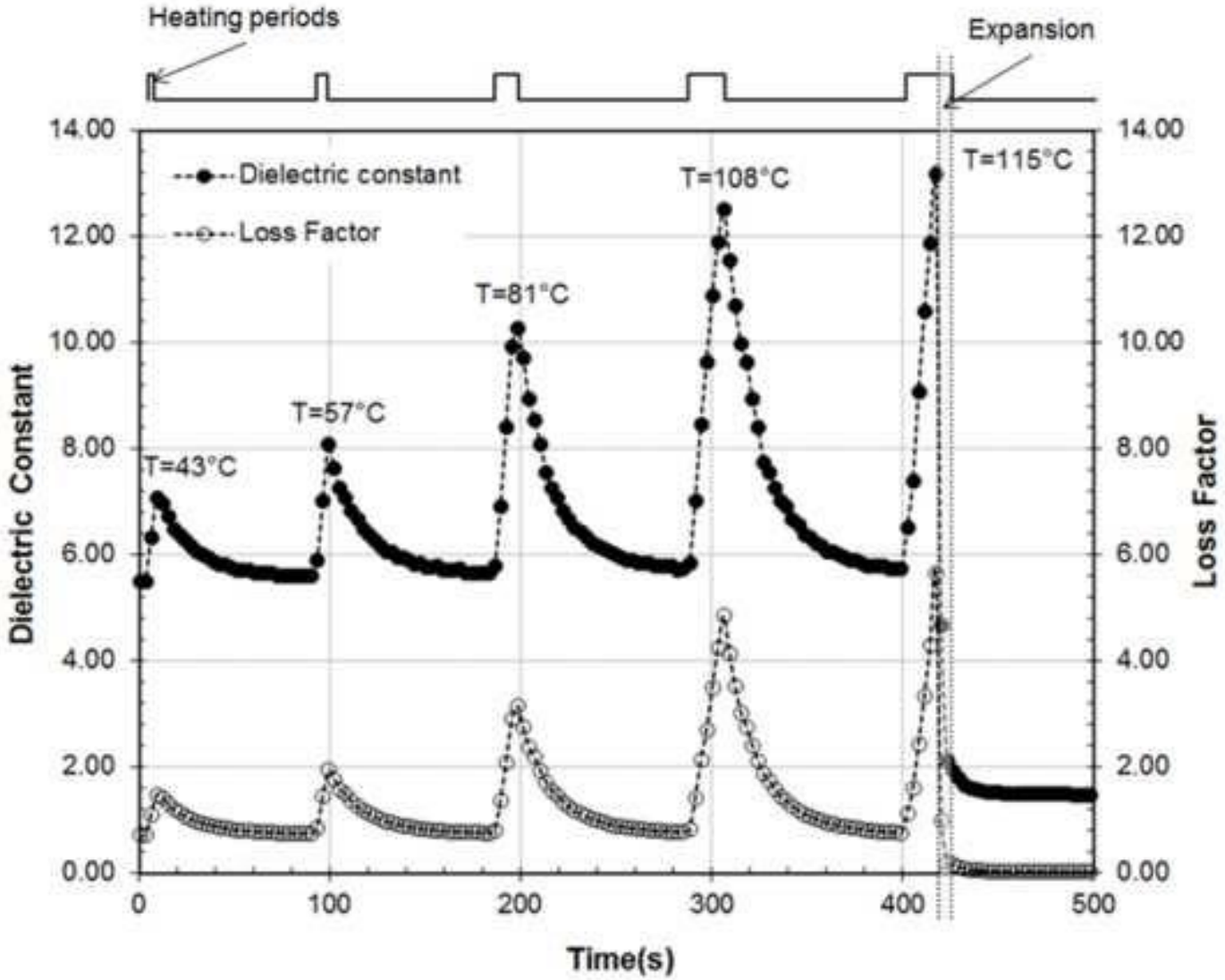

Fig. 6. Time evolution of dielectric properties of a pellet sample processed by microwave heating periods (2, 6, 11, 19 and 24 seconds)

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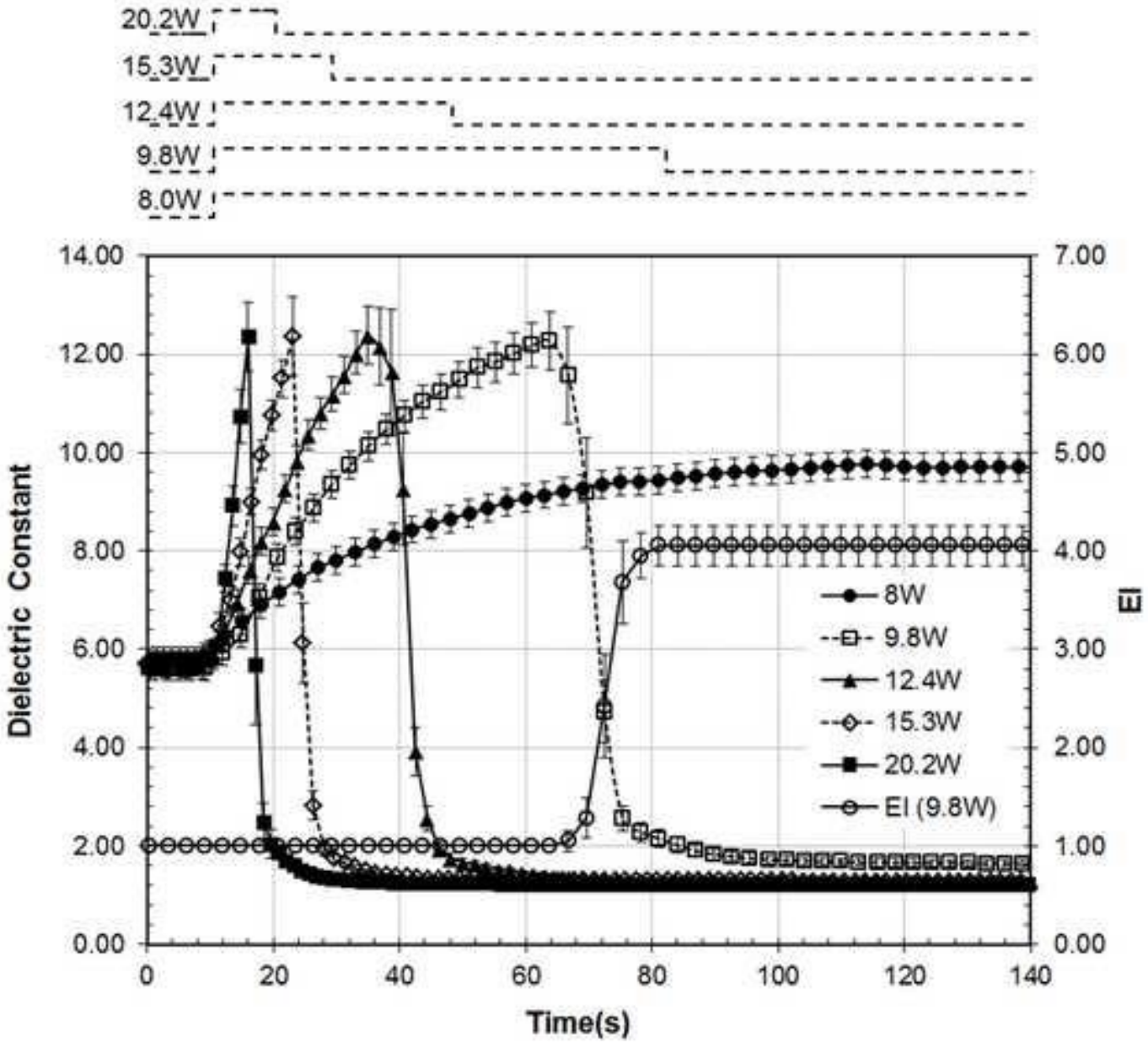

Fig. 7. Dielectric constant of pellet samples during microwave expansion at different absorbed power levels (left axis). EI of trial 9.8W (right axis). Period of time with microwaves on (top).

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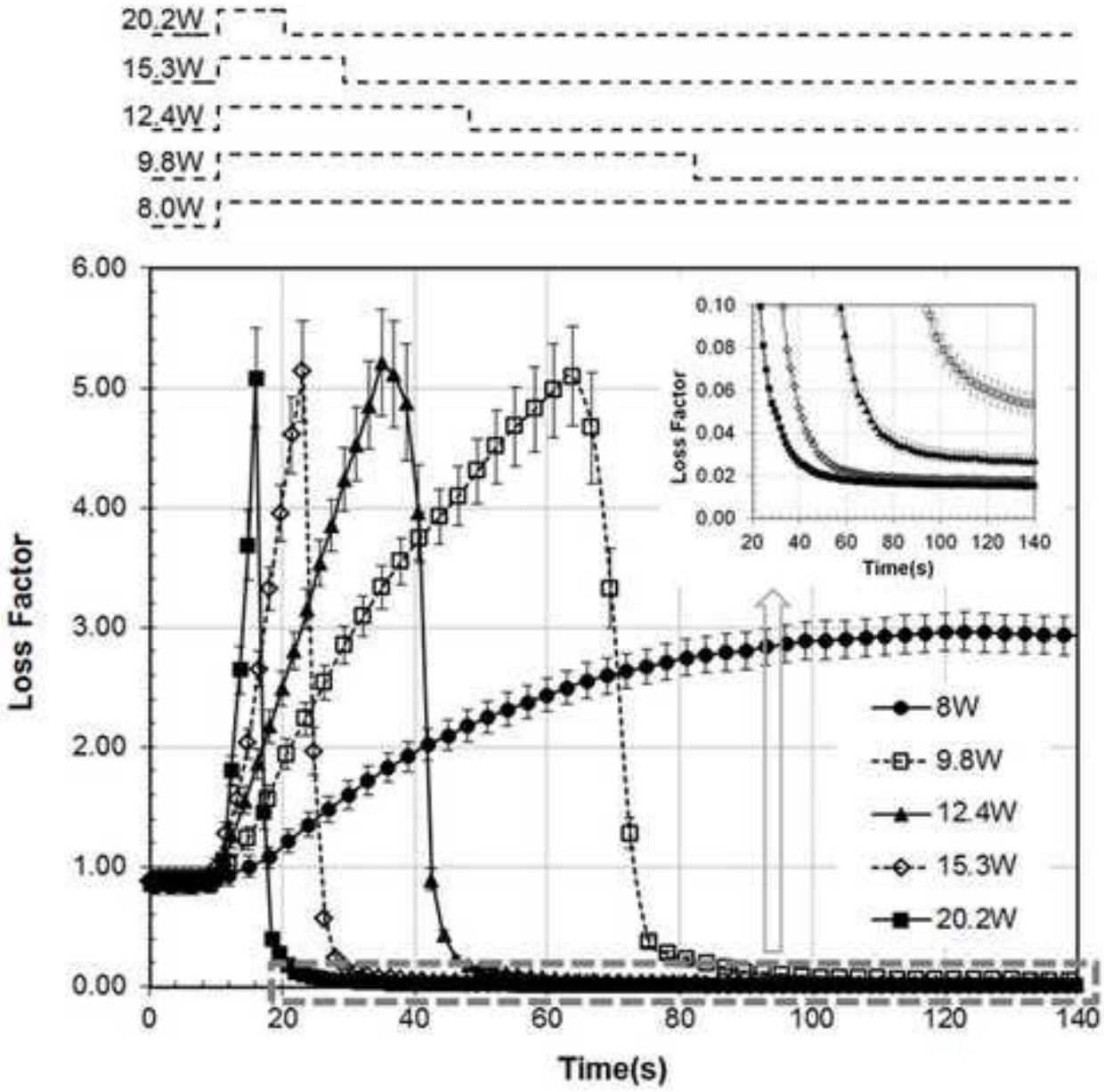

Fig. 8. Loss factor of pellet samples during microwave expansion at different power levels. The inset shows more details in the final loss factor value. Period of time with microwaves on (top).

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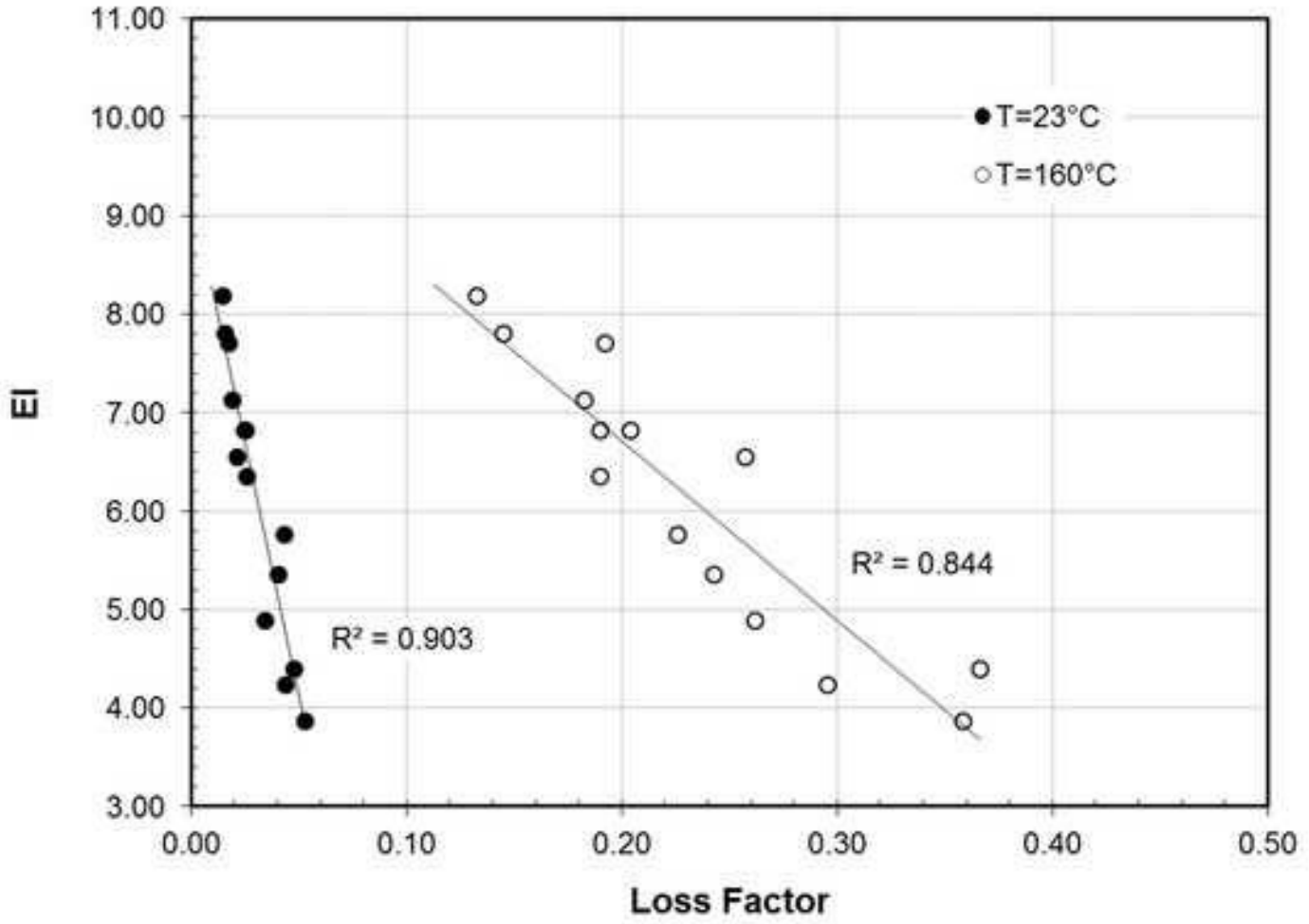

Fig. 9. Expansion Index of several pellet samples after microwave expansion as a function of the loss factor at different temperatures

Figure Captions

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