Gutiérrez Cano, JD.; Hamilton, IE.; Catalá Civera, JM.; Bows, J.; Penaranda-Foix, FL. (2018). Effect of water content on the dynamic measurement of dielectric properties of food snack pellets during microwave. Journal of Food Engineering. 232:21-28. https://doi.org/10.1016/j.jfoodeng.2018.03.018

Elsevier Editorial System(tm) for Journal of Food Engineering
Manuscript Draft

Manuscript Number:

Title: Effect of Water Content on the Dynamic Measurement of Dielectric Properties of Food Snack Pellets during Microwave Expansion

Article Type: Research Article

Keywords: food pellets; microwave heating; microwave expansion; dielectric properties; cavity perturbation method; water content.

Corresponding Author: Mr. José D Gutiérrez-Cano,

Corresponding Author's Institution: Universitat Politècnica de València

First Author: José D Gutiérrez-Cano

Order of Authors: José D Gutiérrez-Cano; Ian E Hamilton; José M Catalá-Civera, Full Professor; John Bows; Felipe L Peñaranda-Foix, Full Professor

Abstract: The evolution of dielectric properties of starch-based food pellets with different moisture contents was measured during microwave expansion to determine the effect of water content on the expansion dynamics.

Dynamic dielectric measurements were found to be an excellent procedure to in situ monitor and characterize the different stages in the material transformation of food pellets during microwave expansion.

Although the maximum bulk expansion of pellets was achieved at a moisture content of approximately 8% (wet basis), comparative analysis showed that a moisture content 10-11% produced the best results considering the tradeoff between the foaming and expansion temperature. This was due to the high expansion index and an expansion temperature that was sufficiently lower than the onset temperature for pellet scorching, which provides an operating window to maximize expansion and minimize the likelihood of burning.

Dielectric measurements during microwave heating in short on/off cycles prior to pellet expansion suggested that the water was not as dielectrically bound for high moisture content pellets

Cover Letter

Dear Editor,

Please find attached the paper

"Effect of Water Content on the Dynamic Measurement of Dielectric Properties of Food Snack Pellets during Microwave Expansion", by José D. Gutiérrez-Cano, Ian E Hamilton , José M. Catalá-Civera, John Bows, Felipe L. Peñaranda-Foix

to be considered as a contribution to the journal of food engineering.

Do not hesitate to contact me if you need any additional information.

Sincerely,

José D. Gutiérrez-Cano
Address: ITACA Institute, Universidad Politécnica de Valencia,
Camino de Vera s/n, 46022, Valencia (Spain)
Phone:+34-963879742
E-mail: jdgutierrez@itaca.upv.es

Highlights:

1. The dielectric properties of food pellets with different moisture contents were measured during microwave expansion.

2. Dielectric properties were measured simultaneously with microwave heating without interference.

3. Scorching in the pellet samples was identified from the time evolution of loss factor after expansion.

4. Pellets of 10–11% moisture content showed a good expansion index and a moderate scorching risk.

TITLE OF THE PAPER:

"Effect of Water Content on the Dynamic Measurement of Dielectric Properties of Food Snack Pellets during Microwave Expansion"

by

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

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

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

[b] PepsiCo R&D, Leicester, UK.

E-mail: ian.hamilton@pepsico.com, john.bows@pepsico.com

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

ABSTRACT

The evolution of dielectric properties of starch-based food pellets with different moisture contents was measured during microwave expansion to determine the effect of water content on the expansion dynamics.

Dynamic dielectric measurements were found to be an excellent procedure to *in situ* monitor and characterize the different stages in the material transformation of food pellets during microwave expansion.

Although the maximum bulk expansion of pellets was achieved at a moisture content of approximately 8% (wet basis), comparative analysis showed that a moisture content 10–11% produced the best results considering the tradeoff between the foaming and expansion temperature. This was due to the high expansion index and an expansion temperature that was sufficiently lower than the onset temperature for pellet scorching, which provides an operating window to maximize expansion and minimize the likelihood of burning.

Dielectric measurements during microwave heating in short on/off cycles prior to pellet expansion suggested that the water was not as dielectrically bound for high moisture content pellets

Wet basis (wb), cavity perturbation method (CPM), moisture content levels (MC), expansion index (EI)

Keywords: food pellets, microwave heating, microwave expansion, dielectric properties, cavity perturbation method, water content.

1. INTRODUCTION

Third-generation starchy snacks provide a new way to appeal to consumers by offering the possibility to finish the snack at home from an intermediate pellet form. To create a shelf-stable glassy product, food pellets are first extruded at low pressure at the die to prevent expansion, and are subsequently dried-down to the desired moisture content (Riaz, 2006), which is typically 10–13%. This process route allows storage without refrigeration, which simplifies transport and improves marketability by affording a high bulk density. The final dried product requires an expansion (puffing) step that can be accomplished by baking, hot air puffing, immersion frying in oil, or microwave heating (Moraru and Kokini, 2003; Nath et al., 2007; Osman et al., 2000).

Compared with other heating technologies, microwave expansion of snacks has been reported as more efficient, faster, and eliminates the need for additional fat (Lee et al., 2000; Moraru and Kokini, 2003). Nevertheless, pellet expansion in the domestic microwave oven is a more complicated process than traditional frying and remains a focus of investigation to overcome challenges related to condensation brought about by the cooler surrounding regions, which can cause clumping after expansion, low and uneven rates of pellet expansion, and/or burning (van der Sman and Bows, 2017).

Starchy pellets undergo several stages during microwave expansion (Boischot et al., 2003; Moraru and Kokini, 2003): (I) the absorption of energy increases the temperature of water molecules to produce superheated steam in the glassy matrix and the material undergoes a state transition from a glassy to a rubbery state, provided that the temperature exceeds the glass transition temperature (Tg); (II) pellet expansion occurs when the vapor pressure of the superheated steam is sufficient to overcome the resistance of the rubber-like matrix; (III) after expansion, if the microwave energy is turned off, the pellet cools down and reverts to a glassy state; (IV) if, however, the energy is maintained for a long enough period, the pellet experiences scorching and then burning.

The moisture content of the food product is a critical element during the successive stages of the microwave process and influences the physical and mechanical properties of the expanded material (van der Sman and Bows 2017). Consequently, several studies have investigated the effects of moisture content for the microwave performance of snack pellets and other related materials.

Lee et al. (2000) analyzed the effect of moisture content and gelatinization on the puffing efficiency and expansion volume of corn-starch pellets, finding an optimal expansion for half-gelatinized starch when the pellet moisture content was ~10% wet basis (wb). By studying the microwave expansion of glassy amylopectin extrudates for five different water activities, Boischot et al. (2003) concluded that a maximum expansion was achieved with moisture losses in the range 10–12% wb. Similarly, Sjöqvist and Gatenholm (2007) examined the influence of the moisture content in the expansion of high amylopectin starch extrudates for packaging applications processed by microwave energy, determining that the largest expansion was attained at moisture levels of 11.2 and 13.4% for extrudates conditioned at a relative humidity of 33% and 54%, respectively.

An important caveat to the aforementioned studies is that they were conducted using multimode domestic microwave chambers, which could result in misleading conclusions because of the uneven electric field configuration that is highly dependent on the chamber configuration, the sample location inside the chamber and the size of the workload, in addition to limited process control (e.g., inability to measure power absorbed by the sample).

Water content has a profound influence on the dielectric properties of food materials and in the glass/rubber transition. Accordingly, the measurement of the dielectric properties can provide valuable information about the water activity and consequently the heating performance of starch pellets during microwave expansion (Nelson and Datta, 2001).

Studies in the literature have reported dielectric properties values for similar materials as a function of the moisture content level. For example, Ling et al. (2015) used an open-ended coaxial-line probe to obtain the (off-line) dielectric properties dependencies of pistachio kernels with regards to radio frequency energy, temperature and moisture content. They found that the loss factor increased with increasing temperature and moisture. Bansal et al. (2015) employed a coaxial probe placed at the bottom of a sample holder to obtain the dielectric properties of corn flour for different moisture contents ranging from 8.8% to 22.7% wb, which again showed a clear correlation of both parameters. Finally, Kraus et al. (2013) used a cylindrical cavity and the cavity perturbation method (CPM) to calculate the dielectric properties of starch-based food materials for different moisture values at room temperature, also reporting a direct correlation between dielectric properties and the moisture content of the samples.

A better understanding of the mechanisms involved during microwave expansion requires the development of fast measurement devices that can provide *in situ* information in the short time period that the expansion process takes place. This knowledge could be used to improve the palatability and texture of snack foods in addition to providing a framework for the development of new products. We recently described a new procedure capable of measuring, for the first time, the *in situ* evolution of dielectric properties and other process-related variables along the different stages of the microwave expansion of starch-based materials (Gutiérrez et al., 2017). This method enabled us to analyze the dynamics of the expansion process and its relationship with process parameters such as the expansion time and the expansion index (EI) during the rapid process of expansion.

The aim of present study was to analyze the effect of the moisture content of starch-based food pellets during microwave expansion by *in situ* dynamic measurement of dielectric properties. The findings from this work may increase our understanding of the kinetics and processing conditions of the expansion process to further improve the overall properties of these types of snacks finished by microwave heating.

2. MATERIALS AND METHODS

2.1. FOOD PELLETS

An intermediate half-product pellet for commercially available snack foods was used as the test material (identical to that used in Gutiérrez et al., 2017). During the production process, these pellets (primarily based on potato flakes) leave the extruder at 35% moisture and are hot air-dried to ~12% moisture in a humidity-controlled environment in preparation for later finish drying in the commercial production process. Although not formulated for microwave heating, the pellets show favorable expansion in a domestic microwave oven. The pellets used in this study were cylindrical in shape and were approximately 30 mm in length and 3 mm in diameter. When fully expanded, the finished product is approximately 50–60 mm in length and 6 mm in diameter.

The pellets were conditioned at three different levels of relative humidity in order to achieve three different moisture content levels (MC) in the range of 8%, 11% and 15% MC. The initial moisture content of the pellet was calculated by heating 60 g of product in a convection oven (Heraeus WU 6100) for 72 h and measuring the weight loss. The initial moisture obtained was 10.31% (wb).

For the additional moisture level measurements, fragments of pellets were placed inside a dessicator containing a saturated solution of potassium acetate (791733 Fluka) and potassium chloride (P9541 Sigma), for six weeks, to allow the pellets to reach a state of equilibrium. The samples were weighed before and after this conditioning period and the final moisture content of the samples was obtained from the difference in weight. The moisture content achieved was 7.91% and 15.67% MC.

Prior to microwave heating, the conditioned pellets were equilibrated to room temperature. The pellets presented a circular cross-section (~3 mm in diameter) and were cut into small pieces of 10 mm in length with flat sides, to allow the vertical placement into the reactor.

2.2. EXPERIMENTAL SET-UP

Microwave expansion of one single pellet placed inside a quartz tube was conducted in the dual-mode microwave cavity as described in Catalá-Civera et al. (2015), where simultaneous microwave heating and dielectric properties measurements are feasible without interference.

The cavity was conditioned for the specific application of microwave expansion of pellets as described in Gutierrez et al. (2017) by installing a venturi-based suction system at the top of the cavity to prevent water condensation in the quartz tube during expansion. The temperature of the pellet was measured from the top of the cavity with an infrared thermal camera (Optris PI 160, Optris, Berlin, Germany). A video camera (MU9PC-MH, Ximea, Münster, Germany), placed at the side of the microwave cavity, recorded the expansion of the pellet for EI calculations.

Figure 1 reproduces the schematic of the experimental setup of the microwave reactor with the pellet sample inside the quartz tube (Gutiérrez et al., 2017).

The microwave heating operation allowed for providing the desirable level of heating to the pellet from temperature and microwave power measurements in a close-loop feedback computer-programmed control system (Gutiérrez et al., 2017). In the experimental set-up, microwave power was applied to obtain a constant heating rate of 2ºC/s for each experiment, which leads to an expansion time of 45–60 s (which is approximately the time it takes for 50–100 g pellets to begin to expand in a domestic microwave oven).

The venturi device was activated only once the expansion was completed and microwaves were switched off. This mode of operation was different to that described previously (Gutiérrez et al., 2017) and minimized the effect of suction on the heating and measurement (temperature and foaming) operations, since the constant suction may exert a small influence on temperature (cooling of the upper pellet surface) and volume during expansion.

In some experiments, samples were also processed in short cycles, where microwaves were applied and rapidly stopped to ensure that the pellet heated below the expansion temperature. After the short cycle heating period, the samples were stabilized to room temperature inside the cavity; they were then removed and weighed. The process was repeated for each sample until the weight variations from two consecutives heating pulses was negligible. At this state, the subsequent heating pulse duration was increased for a sufficient time to cause the expansion of the sample.

2.3. DIELECTRIC MEASUREMENTS

Dielectric properties were calculated from *in situ* resonance measurements of the testing mode in the dual mode cavity by the CPM. The shift of the resonant frequency of the cavity during microwave heating defined the changes in the dielectric constant (real part), whereas the variations of Q-factor determined the loss factor (imaginary part). Since the volume of the sample pellet changes during expansion, CPM coefficients such as the filling factor and the depolarization factor were calibrated with known samples of different volume, as described in Gutiérrez et al. (2017).

From comparative analysis of measurements of reference materials with more accurate methods, the accuracy of dielectric calculations with this procedure was estimated to be around 3% for the dielectric constant and 5% for the loss factor.

2.4 GLASS TRANSITION DETERMINATION

The determination of the Tg range of the samples at varying moisture contents was performed using differential scanning calorimetry (DSC 3+; Mettler Toledo, Leicester, UK) and analyzed using STAR SW 15 Software (Mettler Toledo). Samples (9 ± 1.5 mg) were sealed in crimped stainless steel pans and subjected to a heating ramp of 40 ºC/min from 25 to 220 ºC.

3. EXPERIMENTAL RESULTS AND DISCUSSION

To investigate the influence of moisture level in the expansion process, pellets with approximate moisture content of 8%, 11% and 15% were subjected to microwave expansion in the microwave reactor.

3. 1. TEMPERATURE AND VOLUME (EI) DURING MICROWAVE EXPANSION

Figure 2 shows the surface temperature of three pellets with different moisture content as a function of processing time in the microwave reactor (heating rate 2ºC/s). The EI, calculated from the recorded video signal of the pellet, is also shown to identify the foaming evolution of the samples.

Samples with lower moisture levels required longer times to expand. Accordingly, the time to expand for the ~8% MC sample was 60 s, whereas it was reduced to 45 s for the 15% MC sample. Moreover, the 15% MC sample showed lower foaming capabilities (EI ~4.7) than the other samples (EI ~7.5).

The differences observed in the time required for expansion had a direct relationship with the expansion temperatures measured at 119ºC, 140ºC and 152ºC for the 15%, 11% and 8% MC pellets, respectively.

According to the information given by Moraru and Kokini (2003), maximum expansion occurs at a temperature in the approximate range of the Tg and 100ºC and above. The work of van der Sman and Bows (2017) described the desired optimal state for expansion to be the point at which the boiling line crosses the glass transition line; however, DSC analysis of the pellets with varying moisture levels showed that as the moisture content increased the Tg tended to be closer to 100ºC. The reduction of the Tg resulted in a reduction in the time to develop sufficient superheated steam (and pressure) to drive good expansion, leading to the potential of under expansion of pellets (van der Sman and Broeze, 2013).

Table I describes the measured Tg temperature ranges, which are inversely related to the moisture content of the pellet, and are in concordance with the different temperatures observed during expansion.

Other parameters during the four stages of the process were measured and are shown in Table I. To measure the density before expansion and at other stages, microwave energy was switched off and the sample was weighed outside of the apparatus once it had stabilized at room temperature.

		Sample 8%mc	Sample 11%mc	Sample 15%mc
Before microwaves	mc(%)	7.91 ± 0.15	10.31 ± 0.15	15.67 ± 0.15
	density (gr/mm3)	1.056 ± 0.007	1.119 ± 0.006	1.168 ± 0.006
	Tg (ºC) (Onset)	155.6	108	84
	Tg (ºC) (Mid-point)	164.8	112	108
	Tg (ºC) (End set)	182	128.3	122

Stage I	mc(%)	7.53 ± 0.15	9.85 ± 0.15	14.79 ± 0.15
(Before Expansion)	density (gr/mm3)	1.051 ± 0.007	1.113 ± 0.007	1.155 ± 0.006
	Expansion Tsurf (ºC)	152.2 ± 3.1	140.13 ± 2.8	119.6 ± 2.1
	Time to Expand(s)	58.9 ± 2.5	53.9 ± 2.3	45.6 ± 1.1
Stage II	EI	8.03 ± 0.39	7.33 ± 0.35	4.72 ± 0.32
(After Expansion)	Expansion Time (s)	31.5 ± 2.7	32.7 ± 3.0	32.4 ± 2.6
	mc(%) (Time=90s)	2.15 ± 0.22	3.39 ± 0.32	6.17 ± 0.46
	mc lost (%)	5.76 ± 0.37	6.92 ± 0.47	9.50 ± 0.61
	density (gr/mm3)	0.160 ± 0.004	0.170 ± 0.003	0.226 ± 0.005
Stage III & IV	mc(%) (Time=105s)	1.42 ± 0.18	2.25 ± 0.24	4.23 ± 0.37
	density (gr/mm3)	0.158 ± 0.004	0.167 ± 0.003	0.223 ± 0.005
	Burning Tsurf (ºC)	177.17 ± 6.51	175.47 ± 5.19	171.98 ± 4.32
	Burning Time (s)	100 ± 5.2	115 ± 5.8	130 ± 6.6

Table I. Critical factors measured during microwave treatment of food pellets.

The observation of expansion temperatures in the experiments described above differs slightly from the measured temperatures in Gutiérrez et al. (2017) due to the different application of the venturi suction system, as explained previously. An expansion temperature for the 11% MC sample was measured here as 140ºC, whereas a sample with the same moisture level was measured as 115ºC in Gutiérrez et al. (2017). Furthermore, the final expansion volumes obtained were greater than those from our previous study because the sample was not forcibly cooled by the action of the venturi device during treatment. In addition, the Tg found for the sample of 8% MC was higher than the Tsurf measured before expansion. This difference may be connected to fact that the surface temperature measured was cooler than the temperature in the centre of the pellet.

3. 2. DIELECTRIC PROPERTIES DURING MICROWAVE EXPANSION

Dielectric properties of pellets during expansion were calculated from *in situ* measurement of cavity resonance parameters by the CPM according to the procedure described in the previous section and reported in Gutiérrez et al. (2017).

Figure 3 shows the dielectric constant and loss factor of the measured food pellets with approximate mositure content of 8%, 11% and 15%. The EI values have also been included in the figure to identify the different stages of the expansion process in each sample.

Because water content is related to dielectric properties (Meda et al., 2005; Venkatesh & Raghavan, 2004), the initial measured values were influenced by the moisture levels of the material in each sample. For the different moisture levels studied, the initial dielectric properties varied from 4.65-j0.5 (8% MC) to 7.35-j2.2 (15% MC).

As described in Gutiérrez et al. (2017), microwave irradiation triggered a progressive increase in the dielectric constant and loss factor with processing time due to the temperature increase (stage I), and a rapid drop in both values during expansion (stage II). After expansion, the dielectric properties, in particular the loss factor, decreased slightly when the pellet returned to a glassy state, but were dependent on the moisture retained in the matrix (Figure 3). As stated earlier, continual microwave irradiation would lead to additional heating that causes drying or even burning (stage IV), and dielectric properties could exhibit different characteristics.

During stage I, although the heating rate was identical for all the experiments, the rate of increase in the dielectric constant was faster in those samples with a higher moisture content, which confirms the findings reported in Lewicky (2004).

The first stages can be clearly appreciated in Figure 3 for samples of 8% and 11% MC. In this context, the sudden drop of the dielectric constant during stage II was a clear indication of an explosive expansion caused by high–pressure superheated steam, leading to high foaming in the pellet (EI ~8 for the 8% MC sample and EI ~7.5 for the 11% MC sample). By contrast, the 15% MC sample showed a progressive slowing of the rate before expansion. This could be attributed to a combination of effects due to an excessive softening caused by the lower Tg temperature of the pellet, which is unable to tolerate the steam pressure that is developed during microwave expansion, leading to reduced foaming (EI ~4.7). Also, the material reached the rubber-like state before the superheated steam had sufficient energy to induce the expansion, and the pores of the pellet were filled with unbounded water due to an excess of moisture content; therefore, there was a transient situation before expansion.

This effect also influenced the dielectric properties after expansion (stage III), with higher values for those samples with more water remaining inside the matrix: 6.17% for the 15% MC sample and 2.15% for the 8% MC sample (see Table I).

In all cases, the evolution of dielectric properties of a specific starch-based pellet formulation as a function of moisture content showed a direct dependence on Tg, since it has a direct influence on the rubbery state of the matrix and thus in the final pressure that the superheated steam needs to exceed before expansion can occur (Kusunose et al., 1999).

3. 3. DIELECTRIC PROPERTIES DURING PULSED MICROWAVE EXPANSION CYCLES

To analyze the influence of water activity on dielectric properties, and to further understand the expansion properties represented in Figure 3 (especially those corresponding to 15% MC), the samples were processed in short cycles prior to expansion, as described in section 2.2. Figure 4 shows the loss factor under pulsed heating cycles as a function of the processing time for each sample.

Each heating cycle progressively increased the temperature of the sample at a heating rate of 2ºC/s to reach approximately 110ºC, and therefore the loss factor showed a progressive increase to this temperature. After the heating cycle ceased, the venturi device was activated to remove the expelled water and the loss factor slowly decrease when the sample cooled to room temperature. The markers shown in Fig. 4 represent the moisture content calculated from the sample weight after the stabilization of the measured loss factor for each heating cycle.

The 15% MC sample experienced considerably higher drying than the other samples with this procedure (from 15.77% to 12.99%). This fast process of moisture reduction of the sample before expansion indicated that the water in this sample was unlikely associated with the pellet bulk, and it seems more probable that it was filling the porous of the matrix, as discussed in the previous section with respect to this moisture level.

The remaining samples experienced slight moisture losses and were allowed to expand for a few more cycles. The final moisture content before expansion was 7.57% and 9.77% for the samples with an initial moisture content of 7.88% and 10.34%, respectively.

Figure 5 shows the loss factor time evolution of the three samples during expansion in the last heating cycle. As shown, the pellet sample with an initial moisture content of 15.77% (now 12.99% MC) showed a behavior similar to that of the 11% MC sample represented in Figure 3, with an EI higher than 7, in contrast to the previous EI that was lower than 5. The reduction of moisture content caused an increase in the Tg of the pellet and therefore an increase in required temperature of the superheated steam to overcome the molecular bonding and expand the pellet. The progressive slowing of the dielectric properties rate before expansion shown in Figure 3 for this moisture content was also absent.

Conversely, the sample of 7.57% MC had an EI lower than that of the other samples, which was probably due to the reduced moisture available to be transformed into vapor during the heating cycle, limiting the amount/force (pressure) of the superheated steam before expansion.

3. 4. DIELECTRIC PROPERTIES DURING BURNING

If microwave heating continues after expansion, the temperature of the pellet will increase progressively until it reaches a point where the pellet may scorch and burn as a result of the low moisture content and the proximity to the carbonation point of starches (Moraru and Kokini, 2003).

Indeed, the risk of burning is a common problem of microwave heating, whereas conventional heating occurs at a much slower (and more controllable) rate (van der Sman and Bows, 2017). Consequently, the burning process can be viewed as a quality challenge for the microwave expansion process.

We therefore evaluated burning of the pellet formulations (stage IV) by extending the microwave heating for several seconds after expansion.

Figure 6 shows the dielectric loss factor of several 11% MC samples after extending the microwave application from 20 s to 100 s from when expansion commenced (time axis in Figure 6 from 85 to 165).

As described in the previous section, a high percentage of moisture was released during expansion (6–11% depending upon the initial moisture content). Although the loss factor quickly decreased during microwave expansion, the final value remained dependent on the final volume and moisture content still present in the sample. For 11% MC pellets, an average moisture content of 3.37% was measured after expansion (see Table I). Despite the continuous increase of pellet temperature, the loss factor decreased slightly, which could be attributed to drying of the remaining moisture in the samples (van der Sman and Bows, 2017). However, the decrease in the loss factor was followed by a plateau (and a slight change in the slope) if the heating continued for longer periods, which could be attributed to certain difficulties to maintain the moisture loss and to the beginning of burning. Finally, a sudden drop in the loss factor occurred when microwave energy was ceased and the venturi device was activated, which removed the released water and increased the cooling rate.

To verify the correlation between the burning process and the loss factor, the treated samples from the extended range (20–100 s in periods of 5 s) were sliced open and examined. Figure 7 represents the slices corresponding to 20 s periods. Samples corresponding to 115 s of heating showed the first signs of burning. As illustrated in Figure 7, burning was a gradual process starting from the center, which is consistent with the findings of Moraru and Kokini (2003) and the well-known inverse heating profile of microwaves. The moisture content of the material dropped below 2% for the 11% MC pellet, which does not fit with the hypothesis of Moraru and Kokini (2003), who proposed that the burning process starts when all the moisture content is eliminated from the pellet.

Burning of the samples appeared to correlate quite well with the change in the trend of loss factor after expansion. This trend, which was distinguished by a change in the slope after expansion, was better shown by the first derivative of the loss factor (right axis in Fig. 6). The data indicated that the first derivative values were close to zero after 115 s of microwave processing, which associated well with the first evidence of burning.

The same procedure of extending the processing time after expansion and calculating the derivative of the loss factor was applied to the other pellet samples with different moisture contents, and the results are represented in Figure 8. Similar to the findings of the experimental trial shown in Figure 6, the first signs of burning in the samples were observed when the derivative value approached zero. Positive derivative values indicated not only burning signals but also the complete destruction of the inner part of the pellet.

Those samples with higher moisture contents presented a higher burning resistance, even though they were the first to expand. By contrast, the samples with the lower moisture content started to burn approximately 10 s after reaching their full expansion. The burning temperature varied from 171ºC to 177ºC for samples of 15% MC and 8% MC, respectively.

Similar to that observed in the preceding experiments, the time evolution of dielectric properties during or after expansion was proven to be an effective method to monitor the different stages of the expansion process, in this case the initiation or beginning and duration of the burning process.

4. CONCLUSIONS

Here, we measured the dielectric properties evolution of potato starch-based snack food pellets to investigate the effect of moisture content during the different stages of microwave expansion, including the burning process. Dynamic dielectric measurements proved to be an excellent probe to monitor *in situ* the different stages during the process of microwave expansion. We confirmed that the pellet glass transition temperature was a determining parameter in the dielectric measurements and expansion kinetics because of its direct relationship with the moisture content, as it determines the rubbery state of the matrix and is the driving force that induces pellet expansion. Complementary dielectric measurements during microwave heating at short cycles prior to pellet expansion aided in further understanding the water activity in the pellets, particularly for samples with higher moisture content. These findings showed that for samples with high moisture content, the water was unlikely to be associated with the pellet bulk and more likely it was filling the porous of the pellet

In contrast to previous results reported in the literature, our experimental findings showed a maximum expansion bulk volume in pellets with a moisture content ~8%. Because of the high Tg value, there is an extensive build-up of vapor pressure at this moisture level, which occurs before the matrix enters the rubber-like state and expands. However, the high expansion temperature (~150ºC) required for this moisture content level is too close to the burning temperature of this potato starch based pellet, and therefore the risk of burning is high if microwave heating is not ceased quickly after expansion. In domestic microwave ovens, individual pellets within a larger serving size will have different times to full expansion due to non-uniformity of the electric field over heating time. Conversely, samples of 15% MC expanded at lower temperatures and the pellets were unable to withstand the steam pressure, causing reduced foaming.

From our comparative analysis, pellet samples of 10–11% MC produced the best results. This finding is consistent with that of Moraru and Kokini (2003); nevertheless, our interpretation differs from that of these authors. Considering the tradeoff between the foaming and expansion temperature, samples of 10–11% MC produced the optimum results due to the good EI and an expansion temperature that was sufficiently lower than the onset temperature of pellet scorching (for this particular potato-based pellet), which provides an operating window to maximize pellet expansion and minimize pellet scorching in domestic microwave ovens.

5. ACKNOWLEDGMENTS

The work presented in this paper was funded by PepsiCo R&D. The views expressed in this manuscript are those of the authors and do not necessarily reflect the position or policy of PepsiCo Inc.

6. REFERENCES

Bansal, N., Dhaliwal, A.S. & Mann, K.S. (2015). Dielectric properties of corn flour from 0.2 to 10 GHz. *Journal of Food Engineering*, 166, 255-262. http://dx.doi.org/10.1016/j.jfoodeng.2015.06.019

Boischot, C., Moraru, C.I. & Kokini, J.L. (2003). Factors That Influence the Microwave Expansion of Glassy Amylopectin Extrudates. *Cereal Chemistry*, 80(1), 56–61. http://dx.doi.org/10.1094/CCHEM.2003.80.1.56

Catalá-Civera, J.M., Canós, A.J., Plaza-González, P., Gutiérrez, J.D., García-Baños, B. & Peñaranda-Foix, F.L. (2015). Dynamic Measurement of Dielectric Properties of Materials at High Temperature During Microwave Heating in a Dual Mode Cylindrical Cavity. *IEEE Transactions on Microwave Theory and Techniques*, 63(9), 2905-2914. http://dx.doi.org/10.1109/TMTT.2015.2453263

Gutiérrez, J.D., Catalá-Civera, J.M., Bows, J. & Peñaranda-Foix, F.L. (2017). Dynamic measurement of dielectric properties of food snack pellets during microwave expansion. *Journal of Food Engineering*, 202, 1-8. http://dx.doi.org/10.1016/j.jfoodeng.2017.01.021

Kraus, S., Sólyom, K., Schuchmann, H.P. & Gaukel, V. (2013). Drying kinetics and expansion of non-predried extruded starch-based pellets during microwave vacuum processing. *Journal of Food Process Engineering*, 36, 763–773. http://dx.doi.org/10.1111/jfpe.12045

Kusunose, C., Fujii, T. & Matsumoto, H. (1999). Role of starch granules in controlling expansion of dough during Baking. *Cereal chemistry*, 76 (6), 920-924.

http://dx.doi.org/ 10.1094/CCHEM.1999.76.6.920

Lee, E.Y., Lim, K.I., Lim, J.-K. & Lim, S.-T. (2000). Effects of Gelatinization and Moisture Content of Extruded Starch Pellets on Morphology and Physical Properties of Microwave-Expanded Products. *Cereal Chemistry*, 77(6), 769–773. http://dx.doi.org/10.1094/CCHEM.2000.77.6.769

Lewicki, P.P. (2004). Water as the determinant of food engineering properties. A review. *Journal of Food Engineering*, 61(4), 483-495. http://dx.doi.org/10.1016/S0260-8774(03)00219-X

Ling, B., Guo, W., Hou, L. & Li, R. (2015). Dielectric Properties of Pistachio Kernels as Influenced by Frequency, Temperature, Moisture and Salt Content. *Food and Bioprocess Technology*, 8, 420-430. http://dx.doi.org /10.1007/s11947-014-1413-8

Meda, V., Orsat, V. & Raghavan, V. (2005). Microwave heating and the dielectric properties of foods. In H. Schubert and M. Regier (Eds.), *The Microwave Processing of Foods* (pp.61-75). University of Saskatchewan, Canada. http://dx.doi.org/10.1533/9781845690212.1.61

Moraru, C.I. & Kokini, J.L. (2003). Nucleation and Expansion During Extrusion and Microwave Heating of Cereal Foods. *Comprehensive Reviews in Food Science and Food Safety*, 2, 147–165. http://dx.doi.org/10.1111/j.1541-4337.2003.tb00020.x

Nath, A., Chattopadhyay, P.K. & Majumdar, G.C. (2007). High temperature short time air puffed ready-to-eat (RTE) potato snacks: Process parameter optimization. *Journal of Food Engineering*, 80, 770–780. http://dx.doi.org/10.1016/j.jfoodeng.2006.07.006

Nelson, S.O. & Datta, A.K. (2001). Dielectric Properties of Food Materials and Electric Field Interactions. In Ashim K Datta, Ramaswamy C. Anantheswaran (Eds.), *Handbook of Microwave Technology for food applications* (pp. 69-114).

Osman, M.G., Sahai, D.& Jackson, D.S. (2000). Oil absorption characteristics of a multigrain extrudate during frying: effect of extrusion temperature and screw speed. *Cereal Chemistry*, 77, 101-104. http://dx.doi.org/ 10.1094/CCHEM.2000.77.2.101

van der Sman, R.G.M. & Bows, J.R. (2017). Critical factors in microwave expansion of starchy snacks. *Journal of Food Engineering*, 211, 69-84. http://dx.doi.org/10.1016/j.jfoodeng.2017.05.001

van der Sman, R.G.M. & Broeze, J. (2013). Structuring of indirectly expanded snacks based on potato ingredients: a review. *Journal of Food Engineering*, 114 (4), 413-425.

http://dx. doi.org/10.1016/j.jfoodeng.2012.09.001

Venkatesh, M.S. & Raghavan, G.S.V. (2004). An Overview of Microwave Processing and Dielectric Properties of Agri-food Materials. *Biosystems Engineering*, 88(1), 1-18.

http://dx. doi.org/ 10.1016/j.biosystemseng.2004.01.007

Riaz, M.N. (2006). New technological solutions – Extrusion process. In *International Palm Oil Trade Fair and Seminar (POTS) Snack Foods and Palm Oil – Trends and Opportunities*, session 2, paper 6. Kuala Lumpur, Malaysia.

Sjöqvist M. & Gatenholm P. (2007). Effect of Water Content in Potato Amylopectin Starch on Microwave Foaming Process. *Journal of Polymers and the Environment*, 15(1), 43-50. http://dx.doi.org/10.1007/s10924-006-0039-y

Figure 1

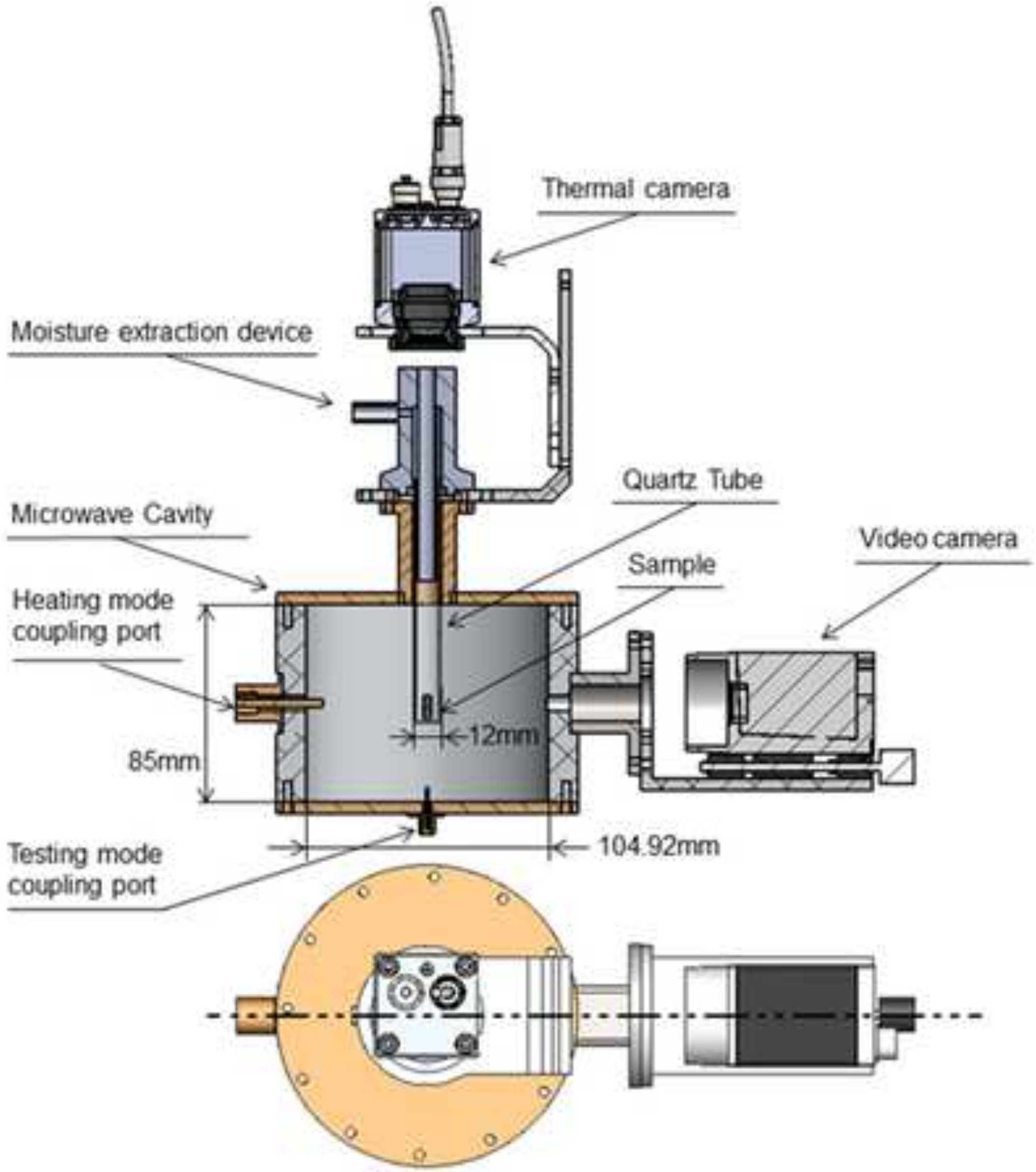

Fig. 1. Schematic view of the dual-mode cylindrical microwave cavity. Thermal camera located at the top access of the cavity. Video camera located at the lateral access of the cavity

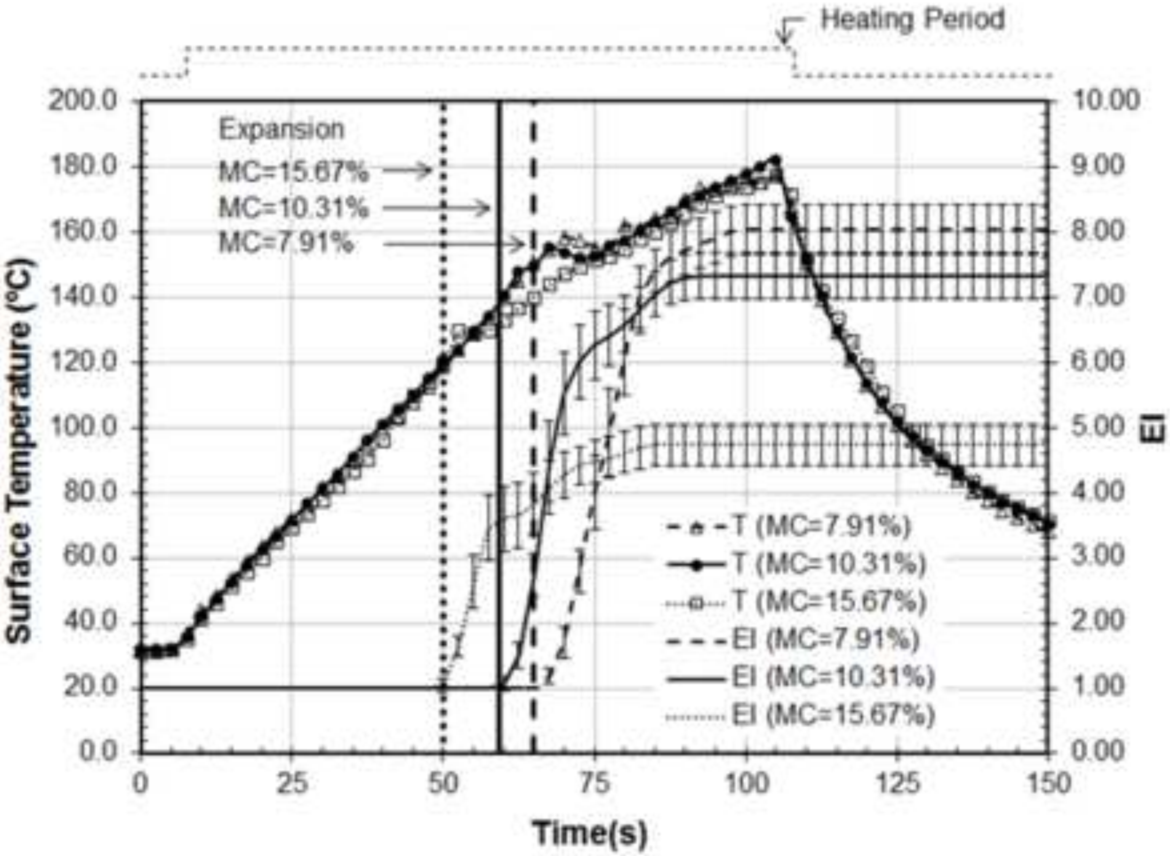

Fig. 2. Temperature and expansion index (EI) of three pellet samples with different moisture content. Error bars of ±0.3°C for the temperature measurements have not been added for clarity.

Figure 3

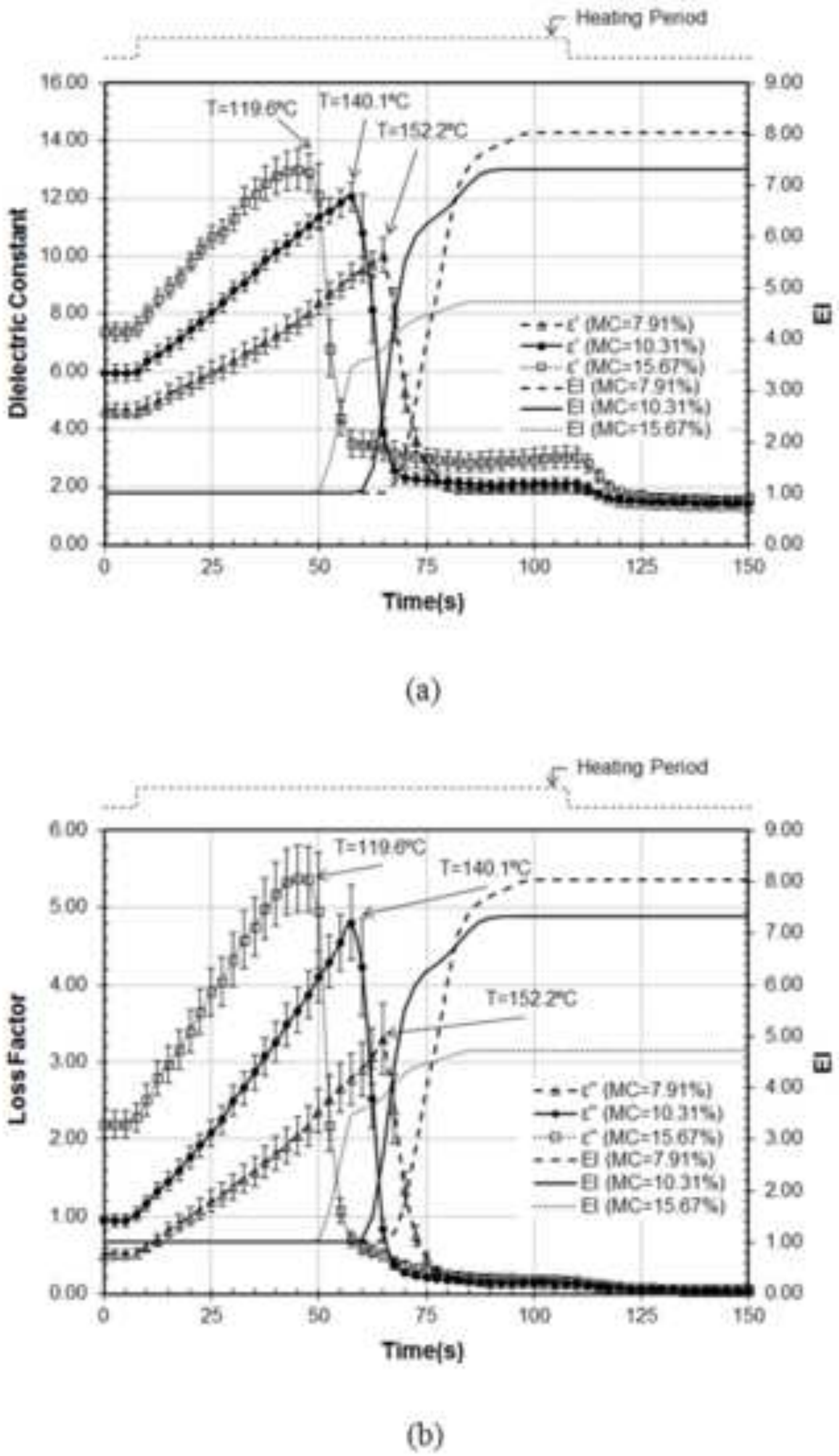

Fig. 3. Dielectric constant (a) and loss factor (b) of pellet samples at different moisture levels during microwave expansion.

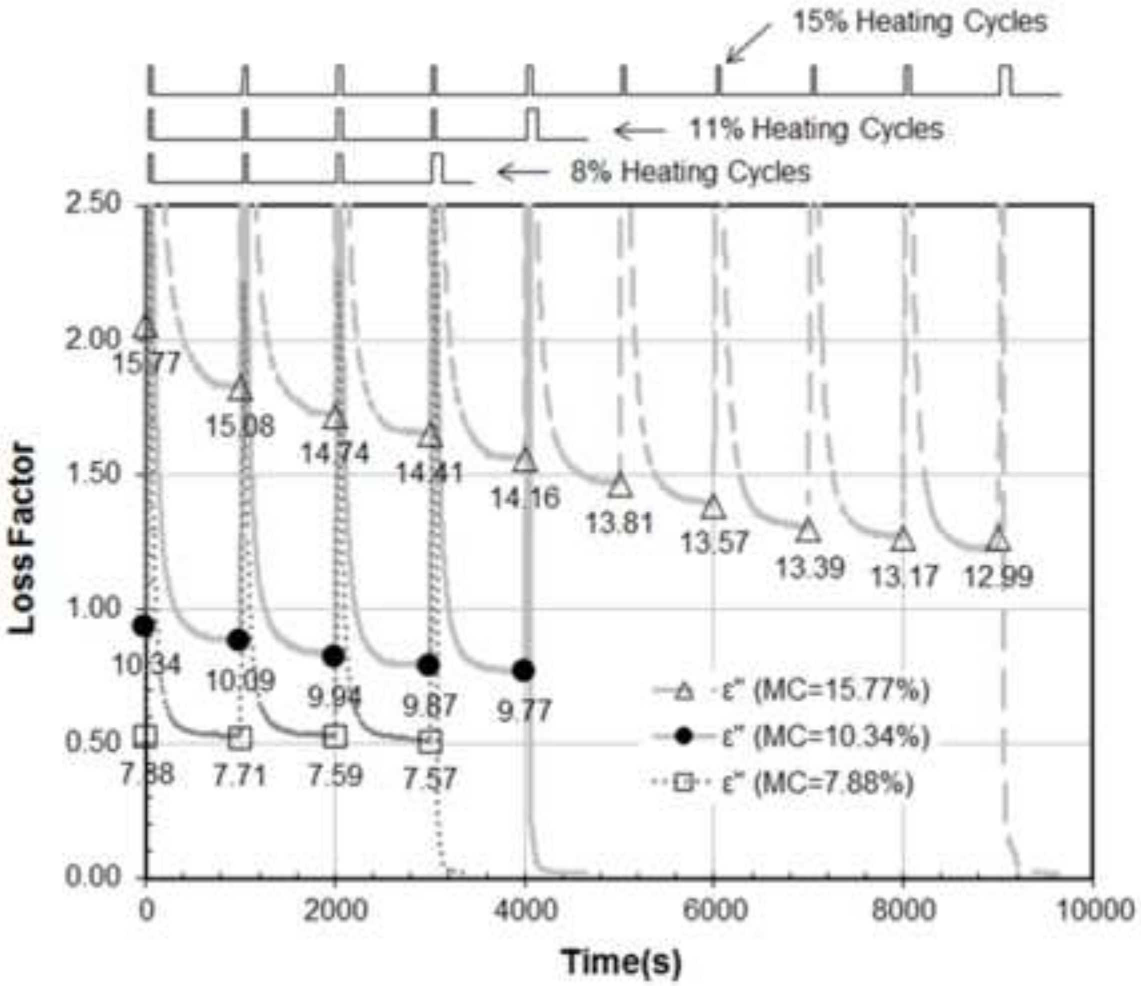

Fig. 4. Measured loss factor of pellets under pulsed heating cycles as a function of the processing time.

Figure 5

Click here to download high resolution image

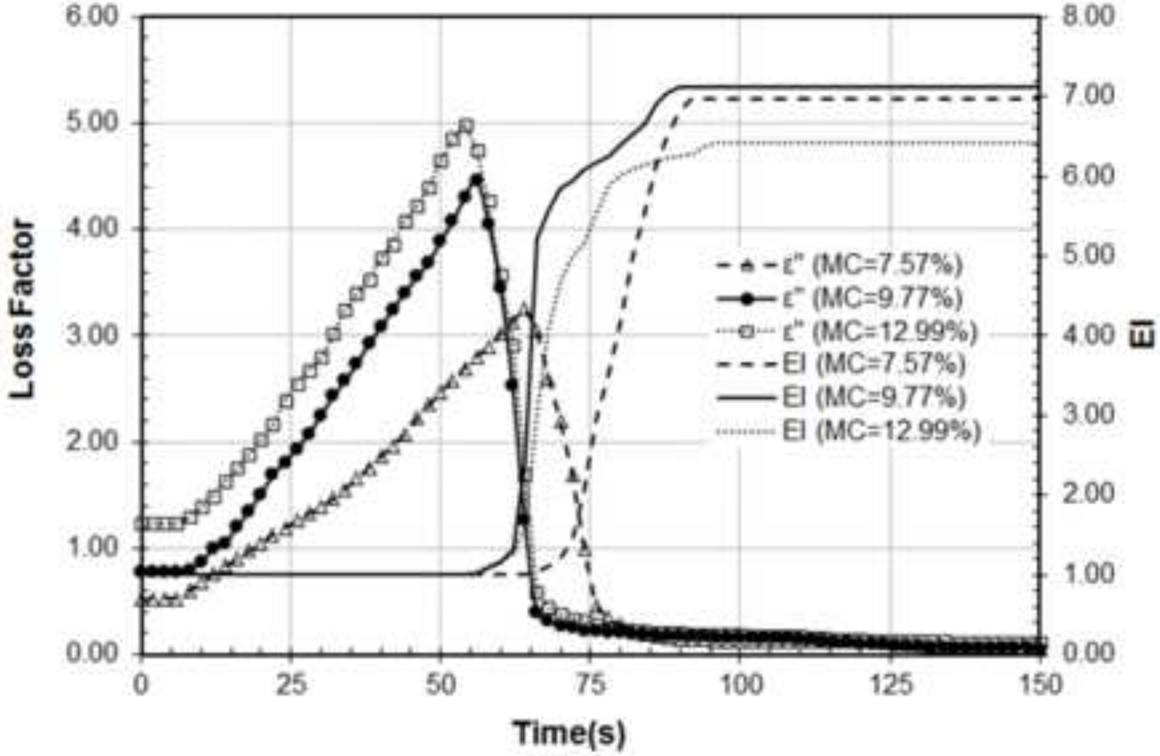

Fig. 5. Loss factor of pellet samples during microwave expansion at different moisture levels after pulsed microwave cycles.

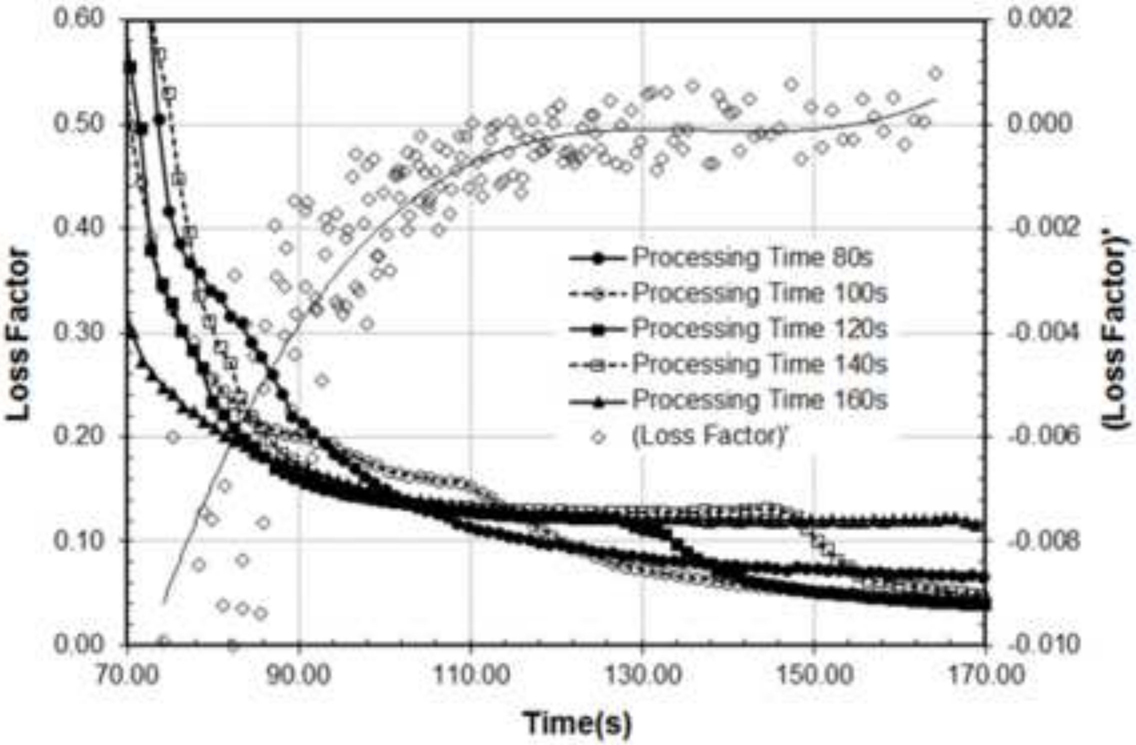

Fig 6. Loss factor of several 11% MC samples after extending the microwave application from 20 s to 80 s from expansion.

Fig. 7. Images of 11% MC pellets after extending the microwave application from 20 s to 100 s from expansion.

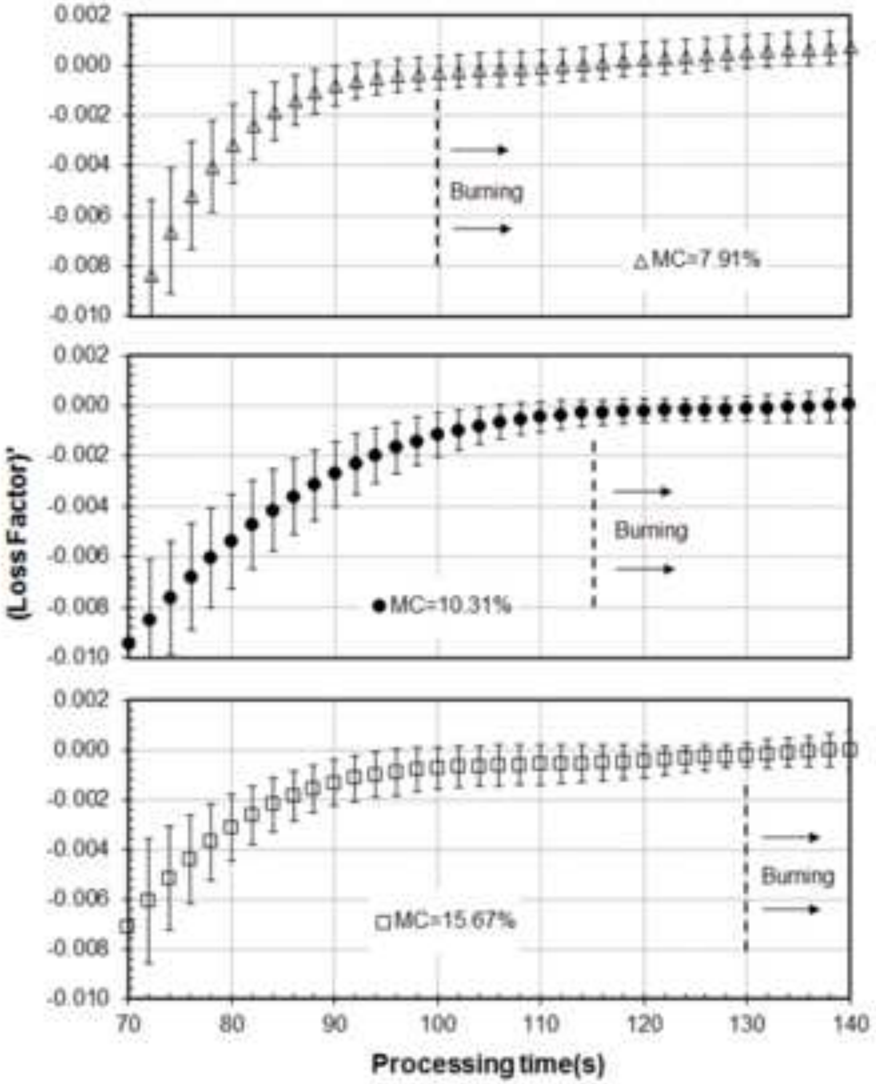

Figure 8. Loss factor first derivative for the different moisture content pellets as a function of the processing time extending the microwave application after expansion - note that the x-axis is different to that in Figure 6

Figure Captions

Fig. 1. Schematic view of the dual-mode cylindrical microwave cavity. Thermal camera located at the top access of the cavity. Video camera located at the lateral access of the cavity

Fig. 2. Temperature and expansion index (EI) of three pellet samples with different moisture content. Error bars of ±0.3ºC for the temperature measurements have not been added for clarity.

Fig. 3. Dielectric constant (a) and loss factor (b) of pellet samples at different moisture levels during microwave expansion.

Fig. 4. Measured loss factor of pellets under pulsed heating cycles as a function of the processing time.

Fig. 5. Loss factor of pellet samples during microwave expansion at different moisture levels after pulsed microwave cycles.

Fig 6. Loss factor of several 11% MC samples after extending the microwave application from 20 s to 80 s from expansion.

Fig. 7. Images of 11% MC pellets after extending the microwave application from 20 s to 100 s from expansion.

Figure 8. Loss factor first derivative for the different moisture content pellets as a function of the processing time extending the microwave application after expansion- note that the x-axis is different to that in Figure 6