A Calibration Approach for the Bulk Temperature Estimation from Container Surface Temperature during Microwave Heating Processes

Rafael Pérez-Campos*, José Fayos-Fernándeza, Juan Monzó-Cabreraa
aDepartamento de Tecnologías de la Información y las Comunicaciones, Universidad Politécnica de Cartagena, 30202, Cartagena, Spain

Abstract
Microwave dielectric heating has been demonstrated to have substantial advantages over traditional heating procedures. Nonetheless, the sample temperature must be continuously monitored during the microwave irradiation procedure. Despite the fact that various sensors exist to directly obtain the sample temperature, this is still an issue for microwave-heating processes. The sample temperature is typically measured with the use of a calibrated external sensor capable of monitoring the surface temperature of the sample container from a predetermined distance. Nonetheless, significant limits have been identified, implying that monitoring the container temperature is insufficiently accurate in many scenarios. In this paper, we present a case study of a calibration approach for estimating sample temperature from pyrometer records during microwave-heating processes in a multimode cavity. Various thermal properties have been determined to explain the errors that tend to take place when monitoring the temperature of the container surface instead of that of the sample. However, some discrepancies were found in calculating the bulk temperature. Furthermore, the estimation of the bulk temperature became more inaccurate at the beginning of the experiments, particularly at high power-to-mass ratio levels. Nonetheless, if low power-to-mass ratios are applied, bulk temperature at specific points can be properly estimated.

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Keywords: microwave heating; multimode cavity; pyrometer; temperature calibration; granular materials.

1 Introduction
The advantages of employing microwaves to heat materials are well known and have gained widespread acceptance in recent decades. Microwave dielectric heating has been found in several cases to significantly reduce processing times, enhance physical and/or mechanical properties, and increase product yields when compared to conventional heating processes [1, 2]. In microwave processing, energy is rapidly converted into heat inside the materials by electromagnetic radiation, whereas thermal energy is simply transported throughout the sample by an external heating source in conventional heating [3]. Although many microwave heating studies have been conducted in modified domestic microwave ovens, the present trend strongly favors the use of specialized instruments for chemical synthesis [4]. Today's commercially available microwave reactors incorporate, among other things, direct temperature control of the reaction mixture and software that allows temperature control via microwave power output adjustment. The main reason for that is the need to continuously monitor the reaction temperature during the microwave irradiation process. Unfortunately, traditional direct temperature sensors, such as thermometers or thermocouples, cannot be used because they will couple with the electromagnetic field. A more accurate method of monitoring temperature during experiments employing microwave energy is to use fiber-optic (FO) sensors, which are more accurate than infrared sensors and able to directly acquire the sample temperature. Unlike thermocouples, FO measuring instruments are immune to electromagnetic interference and do not require shielding. Nonetheless, FO probes offer significant drawbacks, including that they are highly fragile, have a high price, and also have a limited temperature measurement range (if compared to infrared sensors such as pyrometers).

The sample temperature is usually acquired using a calibrated external sensor integrated into the cavity, which is able to monitor the surface temperature of the sample container from a predefined distance [5]. It is expected that the temperature measured on the sample container's exterior will correspond to the temperature of the material within. However, utmost...
caution must be taken when relying on this fact. Given that the sample containers are commonly composed of a thermal insulator (e.g., borosilicate glass), a certain time is required for the temperature sensor to reflect the sample temperature [6, 7]. Hence, the reliable monitoring of reaction temperature becomes nontrivial in spite of being critical to the proper development of the microwave-assisted process [8].

Kappe studied the accurate measurement of reaction temperature in a single-mode microwave cavity [7]. The conclusion was that no differences between FO and infrared temperature sensors will be observed, with the latter being able to accurately reflect the internal reaction temperature provided that: the infrared thermometer is properly calibrated; the reaction mixture is both quite homogeneous and low microwave absorbing; and the reaction is not exothermic. García-Baños et al. developed a temperature calibration method to obtain the bulk temperature under microwave irradiation from the surface temperature measurements [9]. A linear relationship was found between bulk and infrared temperatures. Nonetheless, when the mixture becomes heterogeneous and difficult to stir, some disparities in the recorded FO and infrared temperatures may occur. In addition to that, some temperature gradients can be formed within the microwave-heated sample if efficient stirring is not provided during the microwave heating process as a result of heterogeneous electromagnetic field distribution [10]. Despite the strong interest in adequately monitoring the sample temperature during microwave processes, to the best of the authors’ knowledge, there is no contribution in the literature that estimates the thermal parameters affecting the pyrometer temperature records in a practical way in order to predict the sample temperature by means of that of the sample container in a multimode cavity.

In this paper, we provide a case study on a calibration approach describing the sample temperature estimation from the pyrometer records during microwave-heating processes in a multimode cavity. The sample temperature’s mean value is estimated as a function of the container’s surface temperature as detected by the pyrometer. Various thermal parameters have been considered to explain the errors that usually result from measuring the temperature of the container surface instead of that of the sample.

Materials and Methods
The materials under test (MUT) for the calibration tests were tap water and Peruvian quinoa (Chenopodium quinoa Willd) seeds, produced by Laboratorios Almond S.L. and commercially accessible at a well-known Spanish supermarket chain. The quinoa seeds had an apparent bulk density of 750 kg/m³, with seed sizes ranging from 1 to 2 mm. Its moisture content was about 10 %. In addition to their shape being quite homogeneous, their properties have been widely studied: physical [11], thermal [12], and dielectric [13]. Hence, the drawbacks of monitoring temperature during microwave-heating processes of heterogeneous mixtures or high-microwave-absorbing materials are avoided.

Finally, ground-tire rubber (GTR) was the MUT for the evaluation of the calibration approach. It was kindly provided by Synthelast (Spain). Its bulk density is about 0.4 g/cm³, with granule sizes ranging from 0.5 to 1 mm. Its main components are those described in [4]: natural rubber (36 %), carbon black (27 %), synthetic rubber (22 %), inorganic components (8.5 %), additives and oil (6.5 %). The main reason for employing it is that its heterogeneous particle size, its complex composition, and its dielectric properties vary over temperature (thermal runaway may occur) [4]. Thus, it can be considered the worst-case scenario to evaluate the calibration approach.

Microwave Processing Setup
Three metallic tubes (whose hollow diameters were 1.6 cm) were welded to the upper wall of the microwave cavity to act as by-passes for the connection of accessories and sensors situated inside the cavity. The lengths of the tubes (i.e., 14.5 cm) ensured a cut-off cylindrical waveguide, serving as microwave filters by preventing radiation leakage outward. A motorized PTFE stirrer was used to provide the MUT’s most uniform time-averaged temperature pattern. As depicted in Fig. 1, the stirrer shaft was passed through the central by-pass tube at a rotational speed of 5 rpm. An axial fan (Nidec Beta SL, D07R-12T2S4, Japan) with a maximum flowrate of 33.6 m³/hour was used to effectively extract the generated gases from the cavity through a drilled area (with holes of 2.5 mm in width) at the left side wall, ensuring adequate ventilation of the cavity. The drilled cavity wall’s flow rate rating was significantly decreased since the fan intake was attached to the outside of the wall.
A programmable power source (Dipolar AB, MagDrive 1000, Sweden) generated the microwave energy and supplied power to a 2.45 GHz, 1 kW-rated magnetron. A constant microwave power was supplied to the magnetron throughout all of the tests.

Temperature Monitoring

The MUT temperature was monitored using two FO sensors (OpSens, OTG-A, Canada) connected to a signal conditioner (OpSens, TempSens, Canada). According to the manufacturer’s specifications, the measurement accuracy for temperatures below 45 °C is 0.3 °C, and for temperatures above 45 °C, it is 0.8 °C. As shown in Fig. 2, the sensor tips were positioned in contact with the MUT in diametrically opposed positions on the inside walls of the quartz container. This arrangement prevented measurement points from floating out of control and minimized the chance of damaging the sensors because of the twisting of the FO.

When gases arise from the sample, the use of pyrometers or infrared cameras to measure the material’s surface temperature from above is unreliable, as detailed in [14]. The main reason for this is that gases (for example, water vapour) can cause both types of sensors to become blind. Hence, the pyrometer (CTLaser LT, Optris, Germany) was located behind the microwave oven. The pyrometer offers a measurement accuracy of 1 °C or 1% for temperatures below and above 100 °C, respectively. Its emissivity was set to 0.92 [15]. The pyrometer was pointing at the container through a by-pass tube with a diameter and a length of 1.6 cm and 4.5 cm, respectively. Thus, it is more difficult for gases emanating during the process to obstruct its sight line. The pyrex container temperature was also registered by two FO sensors.

The latter parameter was defined as a number comprised between 0 (the pyrometer is completely obstructed) and 1 (the pyrometer is registering the container temperature adequately) in order to rectify any type of error. As a result of the sample heating, a heat flux (Q_f) is generated from the container’s inner to its outer. The heat flux value can be mathematically determined by applying equation (2), as explained in [16].
Where $Q_T$ is the heat flux from the sample outwards through the container wall ($\frac{W}{m^2}$), $h_T$ is the convection factor ($\frac{W}{m^2 \cdot K}$), and $\Delta T_{out-env}$ is the temperature gradient between the container outside wall and the environmental temperature.

Some temperature differences are found between the container’s inner and outer temperatures, given the heat flux generated mainly as a consequence of the sample heating. This heat flux value can be determined by means of equation (3) [17].

$$Q_T = k_{pyrex} \frac{\Delta T_{in-out}}{x} = k_{pyrex} \frac{(T_{in} - T_{out})}{x} \cdot (3)$$

Where $k_{pyrex}$ is the container’s thermal conductivity ($k_{pyrex} = 1.15 \frac{W}{m \cdot K}$) [18], $\Delta T_{in-out}$ is the temperature gradient between the container’s inside ($T_{in}$) and outside ($T_{out}$) wall temperatures, and $x$ is the thickness of the container wall (3.1 mm). When heating water by applying electromagnetic energy, convection currents occur due to a difference in temperature (or density) between different regions within the same material, and therefore, the fluid is continuously moving. Energy is transferred from hotter areas to colder areas, and this process continues until total equilibrium is reached. Hence, convection currents are helpful to achieve a more homogenous temperature distribution.

However, convection current requires the presence of a fluid (liquid or gas). When working with granular material such as quinoa seeds, the solid’s internal particles are fixed and immobile, making it impossible to detect a flow by variations in temperature or density. Hence, convection currents cannot be formed and the distance from the FO sensors to the container inner wall ($x'$) needs to be known.

This distance is not easy to determine. Adhesive tape cannot be placed over the sensor tip, as it would not be consequently measuring the temperature of the MUT but that of the adhesive tape. In addition to that, the adhesive tape might be damaged as a consequence of the high temperatures, and therefore, the sensor tip will not be fixed. As a solution for that, the adhesive tape was placed above the sample, as shown in Fig. 3. Despite the fact that the FO sensors are well fixed, the exact location of the sensor tip is not yet determined.

From the MUT temperature measurement point to the outside, there are various layers of dielectrics (e.g., quinoa and pyrex) of different thermal conductivity. Thus, $x'$ can be determined by considering that the heat flux must be equal in quinoa seeds and in pyrex at the interface between the layers, as shown in equation (4) [17].

$$Q_T = k_{pyrex} \frac{\Delta T_{in-out}}{x} = k_{quinoa} \frac{\Delta T_{quinoa-in}}{x'} \cdot (4)$$

Where $k_{quinoa}$ is the thermal conductivity of quinoa ($\frac{W}{m \cdot K}$), $\Delta T_{quinoa-in}$ is the temperature gradient between the quinoa and the inside container wall temperatures, and $x'$ is the distance from the FO sensor to the container inside wall.

The thermal conductivity of quinoa ($k_{quinoa}$) was calculated by means of the relation described by equation (5), as indicated in [19]:

$$k_{quinoa} = \alpha \cdot \rho \cdot C_s \cdot (5)$$

Where $\alpha$ is the thermal diffusivity of quinoa seed ($8 \cdot 10^{-8} \frac{m^2}{s}$) [20], $\rho$ is its true density ($1058 \frac{kg}{m^3}$) [11], and $C_s$ is its specific heat capacity ($\frac{J}{kg \cdot K}$), which is strongly influenced by temperature. In this contribution, a linear relation between the specific heat capacity of quinoa and its temperature is used, as described in [12] and shown in equation (6):

$$C_s = 20.9 \cdot T + 1046.3 \cdot (6)$$

Where $T$ is the quinoa temperature (°C), 20.9 represents the variation of the specific heat capacity of the quinoa seeds with temperature ($\frac{J}{kg \cdot K^2}$), and 1046.3 denotes their specific heat capacity ($\frac{J}{kg \cdot K}$) at 0 °C.

Regarding the value of the GTR thermal conductivity, it is $0.15 \frac{W}{m \cdot K}$ (extracted from [21]).

* Rafael Pérez-Campos
E-mail address: rafael.perez@upct.es
Transmitted Power Regimes

In all calibration testing, the microwave power transmitted to the magnetron was 900 W. Table 1 describes the microwave testing parameters: material used, objective parameter (i.e., parameter value to determine), power level employed, temperature reached, and sample mass. Independently of the material employed, some water vapour emanated during the tests.

Table 1. Parameters for the calibration tests

<table>
<thead>
<tr>
<th>Material</th>
<th>Water</th>
<th>Quinoa</th>
<th>GTR</th>
</tr>
</thead>
<tbody>
<tr>
<td>Objective parameter $V_L$ and $x'$</td>
<td>3</td>
<td>3</td>
<td></td>
</tr>
<tr>
<td>Number of tests</td>
<td>3</td>
<td>3</td>
<td></td>
</tr>
<tr>
<td>Power (W)</td>
<td>900</td>
<td>900</td>
<td></td>
</tr>
<tr>
<td>Temperature reached (°C)</td>
<td>90</td>
<td>110</td>
<td></td>
</tr>
<tr>
<td>Mass (g)</td>
<td>1000</td>
<td>600</td>
<td>300</td>
</tr>
</tbody>
</table>

Table 2. Parameters for the evaluation tests

<table>
<thead>
<tr>
<th>Material</th>
<th>Water</th>
<th>Quinoa</th>
<th>GTR</th>
</tr>
</thead>
<tbody>
<tr>
<td>Number of tests</td>
<td>3</td>
<td>3</td>
<td>3</td>
</tr>
<tr>
<td>Power (W)</td>
<td>600</td>
<td>600</td>
<td>600</td>
</tr>
<tr>
<td>Temperature reached (°C)</td>
<td>87</td>
<td>110</td>
<td>94</td>
</tr>
<tr>
<td>Mass (g)</td>
<td>1000</td>
<td>600</td>
<td>300</td>
</tr>
</tbody>
</table>

Results and Discussion

Calibration Procedure

The data employed for the determination of the $V_L$ and the convection coefficient corresponds to the cooling period during the tests with water. The container wall outside temperature versus the pyrometer-registered temperature of water are shown in Fig. 4 (a) for a temperature range comprised between 27 and 90°C, as well as the data fit from equation (1). In Fig. 4 (b), one can see both the container wall’s inside and outside temperatures. The optimum value for the convection factor ($h_T$) was obtained by employing equations (2) and (3).

Table 3. RMSE and $R^2$ for the $V_L$ and the convection coefficient estimations

<table>
<thead>
<tr>
<th>Parameter</th>
<th>$V_L$</th>
<th>$h_T$</th>
</tr>
</thead>
<tbody>
<tr>
<td>Value</td>
<td>0.97</td>
<td>35</td>
</tr>
<tr>
<td>RMSE</td>
<td>0.55</td>
<td>0.29</td>
</tr>
<tr>
<td></td>
<td>0.40</td>
<td>0.37</td>
</tr>
<tr>
<td></td>
<td>0.48</td>
<td>0.45</td>
</tr>
<tr>
<td></td>
<td>0.9990</td>
<td>0.9995</td>
</tr>
<tr>
<td></td>
<td>0.9996</td>
<td>0.9993</td>
</tr>
<tr>
<td></td>
<td>0.9993</td>
<td>0.9991</td>
</tr>
</tbody>
</table>

* Rafael Pérez-Campos
E-mail address: rafael.perez@upct.es
The next step consists of the determination of the \( x' \) value. For that purpose, the data corresponding to the cooling period during the tests with quinoa seeds was taken into consideration. Fig. 5 shows the container’s inside wall temperature dependence on the bulk temperature of quinoa. The data fitting obtained using equation (4) is also shown.

![Graph showing quinoa bulk temperature versus container inside wall temperature](image)

Despite the fact that some differences between the experimental data from different tests are observed (mainly due to a not completely homogenous temperature distribution), the data fitting shows a good agreement for the work temperature range. Table 4 describes the value for \( x' \) employed for the experimental data fitting, the RMSE, and the \( R^2 \) values obtained.

<table>
<thead>
<tr>
<th>( x' ) (mm)</th>
<th>( R^2 )</th>
<th>RMSE</th>
</tr>
</thead>
<tbody>
<tr>
<td>1.0</td>
<td>0.9958</td>
<td>0.65</td>
</tr>
<tr>
<td>1.06 (test 1)</td>
<td>0.9937</td>
<td>1.37</td>
</tr>
<tr>
<td>0.9979 (test 3)</td>
<td>1.06 (test 1)</td>
<td>1.06</td>
</tr>
</tbody>
</table>

Table 4. RMSE and \( R^2 \) values for the \( x' \) estimation

**Evaluation**

Three tests employing water were conducted to evaluate the optimum values determined for \( V_L \) and the convection coefficient. The water temperature monitored by the FO sensors over time is shown in Fig. 6. The temperature corrections obtained after applying equations (1) and (2) are also shown. The 900 W-powered test length is shorter than expected (1200 s), as steam hindered the pyrometer. Small differences are observed between the experimental data and the values after the correction. As a general trend, estimated values tend to be a bit lower than experimental ones. Despite the fact that an anomalous behaviour is found for the test transmitting 300 W during heating (i.e., in the initial stage, the temperature increment velocity is lower than that at the heating final stage), the pyrometer seems to be useful for the temperature estimation.

![Graph showing water temperature over time for evaluation tests at different power levels](image)

* Rafael Pérez-Campos
E-mail address: rafael.perez@upct.es
The RMSE and $R^2$ values for the temperature corrections during the heating period are shown in Table 5.

<table>
<thead>
<tr>
<th>Test</th>
<th>RMSE</th>
<th>$R^2$</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>1.25</td>
<td>0.9919</td>
</tr>
<tr>
<td>2</td>
<td>2.01</td>
<td>0.9869</td>
</tr>
<tr>
<td>3</td>
<td>1.01</td>
<td>0.9976</td>
</tr>
</tbody>
</table>

Table 5. RMSE and $R^2$ values for the temperature corrections using water

For a better understanding of the error committed when estimating the temperature, it is shown (in absolute values, that is, in degrees) in Fig. 7, in addition to both the actual and the estimated heat fluxes. The error strongly increased at the beginning of the tests, as can be easily observed in the 900W-rated test. Regarding the heat fluxes, the estimated values are lower than the actual ones. After that moment, the heat flux continues growing until the water begins to evaporate, that is, until the pyrometer temperature reaches near 70°C. Given the fact that heat is lost during evaporation, as indicated in [22], the actual heat flux through the sample container is decreased, and the estimated one is consequently approaching it.

There are two main reasons to explain the differences between the estimated and the actual heat flux. First, the fact that the pyrex offers a low thermal conductivity can delay the measurement of thermal effects in the sample. The other significant limitation is due to the air inside the cavity, which remains cool (at room temperature), thereby causing a decrease in the surface temperature of the sample container and resulting in temperature gradients during microwave heating, as explained in [9]. As a result, the pyrometer's measurement of the surface temperature of the pyrex container cannot be considered representative of the sample temperature, at least, at the beginning of the heating process. Following this point, the error barely increases or remains constant, as shown in the 900W-rated test. A different situation is found when evaporation starts, as both estimated and actual heat fluxes offer similar values, and therefore, the temperature error is close to zero, as previously shown in Fig. 7.
Given the varied fluctuations in the actual heat flux (and consequently, in the error), this temperature calibration only works effectively for single temperature points once evaporation starts, i.e., at a pyrometer temperature of 80 °C. However, it may also be viable for certain temperature ranges (i.e., for the pyrometer temperatures ranging from 35 to 65 °C), provided that the increase in the actual heat flux remains low enough. This consequently helps minimize the error caused by the delay due to the low thermal conductivity of the sample container.

Fig. 8 shows the bulk temperature of the quinoa seeds, as well as both the pyrometer records and its temperature correction. In this case, temperature values after the correction were much lower than those of the quinoa. It must be noted that the estimated temperature was very near the quinoa temperature during the final stage of the tests, with the exception of the 900W-rated test. It seems that the use of higher power levels could potentially result in the generation of hotspots, as detailed in [23], thereby causing the emanation of water vapour in spite of the fact that the mean temperature values of the quinoa seeds were similar (100-110°C) to those seen in the other tests.

Fig. 8. Quinoa temperature during the evaluation tests: (a) 300 W; (b) 600 W; (c) 900 W

Despite the fact that both the thermal conductivity of quinoa and the distance from the temperature measurement point to the container inner wall (x') were considered, the differences between the quinoa temperature values and those estimated are quite higher than those observed when employing water. Thus, the RMSE and $R^2$ values are quite poor for the temperature corrections during the heating period before the pyrometer was obstructed by the gases, as observed in Table 6.

Table 6. RMSE and $R^2$ values for the temperature corrections using quinoa

<table>
<thead>
<tr>
<th>Test</th>
<th>RMSE</th>
<th>$R^2$</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>9.06</td>
<td>0.8695</td>
</tr>
<tr>
<td>2</td>
<td>20.73</td>
<td>0.2149</td>
</tr>
<tr>
<td>3</td>
<td>18.15</td>
<td>0.3349</td>
</tr>
</tbody>
</table>

The error committed due to the temperature estimation using quinoa seeds, the actual heat flux, and the estimated heat flux can be observed in Fig. 9. The error increment was significant at the beginning of the test, especially in the tests transmitting higher power levels (i.e., 600 W and 900 W). Subsequently, the error continued to increase, although at a lower rate, corresponding to the lower actual heat flux rate. It is noteworthy that a substantial increase in the actual heat flux (and consequently, in error) occurred at a pyrometer temperature of approximately 50°C, likely attributed to the increase in permittivity value of quinoa seeds with temperature [13]. Thus, the estimated heat flux values are much lower than the actual ones. The moisture content of quinoa seeds starts to evaporate at around 60 °C according to the pyrometer measurements. This is because of their hygroscopic equilibrium, which states that moisture content decreases with increasing temperatures [24].
can be observed in the heat flux behaviour, which reaches its highest value at this temperature. From that moment on, the values of the estimated and actual heat fluxes are consequently more similar.

![Graph A]

![Graph B]

![Graph C]

**Fig. 9.** Temperature estimation error and heat flux versus pyrometer temperature for the evaluation tests with quinoa seeds at: (a) 300 W; (b) 600 W; (c) 900 W

The delay in the bulk temperature measurement leads to a significant error when estimating the temperature, as explained before. However, this error was rather low for the 300W test. Despite the fact that the pyrometer’s records are not representative of the sample temperature at the beginning of the heating process, smaller temperature errors were observed once evaporation started. Nevertheless, the appearance of water vapour can partially blind the pyrometer, as demonstrated in [25]. This fact actually occurred for the test with the highest power level. In summary, for quinoa seeds, this calibration in temperature can be useful for single temperature points, provided that the increase in the heat flux through the pyrex is sufficiently low (i.e., low power levels are utilized). The GTR temperature, the pyrometer records, and the temperature correction can be seen in Fig. 10. One can observe that, after the correction, the estimated temperature values are still lower than those of the GTR. Nonetheless, these values were near the bulk ones at the final stage of the tests, for the test employing the lowest power level.

Regarding the 600W and 900W-rated tests, a larger GTR temperature increase as well as a decrease in the pyrometer temperature records are observed at the end. It seems that some hotspots were generated as a consequence of the heterogeneous temperature distribution [26], thereby emanating some gases that hindered the pyrometer [14].

* Rafael Pérez-Campos
E-mail address: rafael.perez@upct.es
There is a significant difference between the GTR and the estimated temperature values. Furthermore, this difference seems to be higher with higher power levels, as indicated by the RMSE and $R^2$ values shown in Table 7.

<table>
<thead>
<tr>
<th>Test</th>
<th>RMSE</th>
<th>$R^2$</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>6.35</td>
<td>0.9270</td>
</tr>
<tr>
<td>2</td>
<td>8.31</td>
<td>0.8553</td>
</tr>
<tr>
<td>3</td>
<td>14.87</td>
<td>0.5415</td>
</tr>
</tbody>
</table>

Table 7. Evaluation of the temperature corrections with GTR

The difference in value between the GTR and the estimated temperatures (i.e., error) when using the GTR, as well as the heat flux and the estimated one, are depicted in Fig. 11. As seen before with quinoa seeds, the error increments significantly at the beginning of the tests, and the actual heat flux values are significantly higher than the estimated ones. From the tests delivering 600W and 900W, a strong increase in the temperature error can be observed at the end of the experiments. Allegedly, the fact that some hotspots in the GTR sample appear can lead to thermal runaway and the appearance of gases, as shown in [26]. Thus, the GTR temperature strongly increases [4] while the pyrometer readings start to degrade [14].

Fig. 10. GTR temperature during the evaluation tests: (a) 300 W; (b) 600 W; (c) 900 W

Fig. 11. Temperature estimation error and heat flux versus pyrometer temperature for the evaluation tests with quinoa seeds at: (a) 300 W; (b) 600 W; (c) 900 W
The error when estimating the temperature, mainly due to the delay in the bulk temperature measurement, was higher as higher power levels were employed. If some chemical processes happen, the temperature estimation is seriously affected given the fact that the dielectric properties might strongly vary [9], leading to significant variations in the heating rate, as observed at the end of the 900W-rated test using GTR. Although similar GTR temperatures were reached, the temperature estimation was not affected during the 300W test. A possible reason is that a more homogeneous temperature distribution is achieved when using lower power levels, thereby avoiding hot spots [26].

Regarding the use of this sort of temperature calibration for the microwave heating of GTR, it can prove valuable for single temperature points or even for specific temperature ranges if the rate of the heat flux through the pyrex is sufficiently low, implying that low power levels should be employed.

Conclusions
This study introduces a calibration approach for estimating the bulk temperature of a sample within a container during microwave processing in a multimode oven. During the heating process, the payload was continuously agitated with a PTFE stirrer, its temperature was measured with two FO sensors, and that of the container was measured with another two FO sensors aligned with a pyrometer. Thus, the observed temperature deviations were analysed and mathematically characterized. The calibration performs effectively in liquids (e.g., water), in which convection currents are formed. However, a different scenario was observed with granular materials, as the estimated temperature may significantly differ from the actual one.

In the initial stage of the experiments, the estimation of the bulk temperature became inaccurate, especially for high relative power levels, due to the delay time for the sample temperature increment to be reflected by the container surface and consequently acquired by the pyrometer. Nonetheless, the bulk temperature estimation becomes appropriate for single bulk temperatures, or even for a certain bulk temperature range, provided that the heat flux increment with respect to the pyrometer temperature is linear. Some water vapour was emanated during the experiments. Although the steam was extracted from the cavity, it hindered the pyrometer’s vision for the test with the highest power level, as easily observed in the evaluation tests with quinoa seeds. The more power is transmitted, the more cold and hot spots are generated. As a result, the amount of water vapour will increase, even obstructing the pyrometer readings if the gas extraction level is not high enough. Thus, in order to achieve better temperature uniformity and avoid the pyrometer obstruction, it seems advisable to employ low relative power levels.

It must be emphasized that the discussion on appropriate temperature monitoring herein has been restricted to determining the bulk temperature. Some complications may arise if the sample is strongly microwave-absorbing [7], if its complex permittivity values vary significantly with increasing temperatures (e.g., ground-tire rubber [4]), or when the chemical reactions occurring during the microwave processing are endothermic, thus requiring an extensive calibration procedure [27]. Special attention should be paid if some chemical processes take place during the microwave-heating process, as observed during the experiments using GTR. Chemical changes in the material may imply meaningful variation in its temperature, and therefore the estimation error might be inaccurate. Furthermore, some gases may emanate and partially obstruct the pyrometer. As a result, the pyrometer temperature would be decreasing whereas the bulk temperature would be rapidly increasing. If the application of this calibration approach is intended in the presence of thermal runaway phenomena, certain considerations need to be taken into account, as stated in [28]. A potential solution could involve utilizing a method of microwave power control based on a single temperature threshold, as described in [23].

This research will contribute to a better understanding of bulk temperature estimation during microwave treatment of granular material. Furthermore, the calibration approach presented in this paper can help determine some thermal parameter values affecting the heating performance, thereby enhancing further simulation of dielectric heating.

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Ethics and Consent
This article does not contain any studies with human participants or animals performed by any of the authors.

Authors’ Contributions
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**Conflicts of Interest**
The authors declare no conflict of interest.

**Data Accessibility**
Data will be made available on request.

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* Rafael Pérez-Campos
E-mail address: rafael.perez@upct.es
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* Rafael Pérez-Campos
E-mail address: rafael.perez@upct.es