ECO-EFFICIENT DRYING OF FRESHLY PRESSED CERAMIC TILES THROUGH MICROWAVE TECHNOLOGY

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1. ABSTRACT

In the production of ceramic tiles, between 8 and 10% of the thermal energy consumed is used in drying the freshly pressed ceramic tiles. The pieces obtained by semi-dry uniaxal pressing contain between 5 and 7% water by weight, which must be eliminated before the glazing and decorating stage. Currently, vertical or horizontal dryers with gas burners are used for drying by air convection. Depending on tile composition, size, thickness and above all, on the type of dryer used, drying times can vary widely from 25 to 120 minutes.

Despite the fact that microwave technology has been identified as one of the most promising technologies in this field, it has not been developed for this type of industrial application. One of the reasons for its limited experimentation and implementation is that conventional microwave technology generates overheating points and high vapour pressure in the pores, which can cause fractures. This study presents the development of a fast but gentle drying process using microwave

technology, which allows freshly pressed ceramic pieces to be dried, avoiding overheating points and overpressure inside the pieces.

The drying tests were carried out in a microwave facility, set up such that the exposure of the piece to the microwave electric field had a homogeneous distribution in the longitudinal direction and a gradient in the transverse direction. In particular, a continuous microwave oven with a conveyor belt was used, built by CEINNMAT with a frequency of 2.45 GHz and an applicator of 2 metres in length and filters to avoid radiation leaks. In order to monitor the surface temperature of the pieces during the drying process, pyrometric sensors were installed. The study was supplemented by measurements of the dielectric properties of typical ceramic materials in tile manufacture, measurements of temperature distribution through an infrared camera when the material exited the drying treatments and evaluation of various ceramic properties of the dried pieces.

2. INTRODUCTION

Despite the fact that microwave technology has been identified as one of the most promising technologies in drying ceramics [1-4], it has not been developed for industrial applications in drying ceramic tiles [5]. This has been identified in relation to pore pressures due to the phase change that produces fractures in conventional microwave systems [3, 6-8].

On comparing microwave technology with conventional hot air drying, each system presents a series of advantages and disadvantages. The conventional drying system uses natural gas with a much more economical price than electricity and a large production capacity, but with great heat losses due to thermal conduction and low efficiency, above all in open systems and at low temperatures. The microwave drying system is very efficient in thermal conversion and has minimal losses due to conduction, as the oven does not heat up, but electric energy has an elevated cost compared to gas.

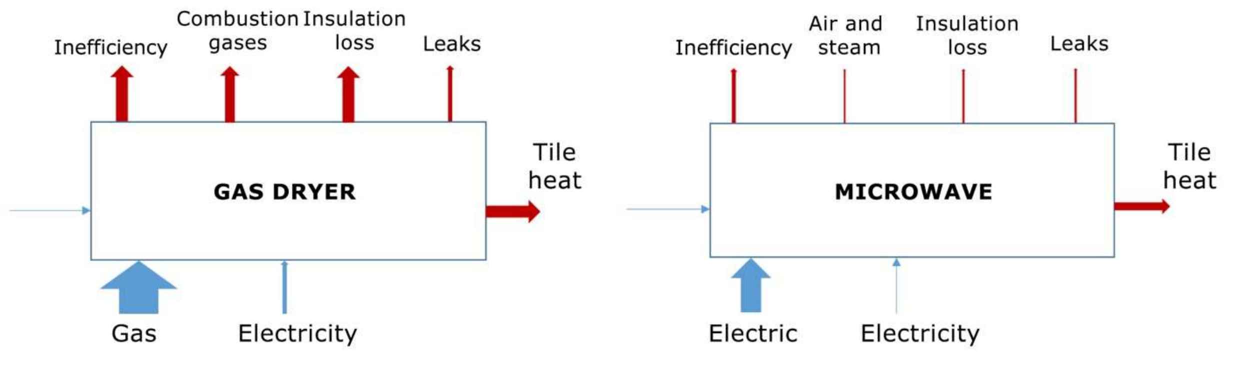

***Figure 1**. Sankey diagram of energy flow in a gas dryer compared to a microwave dryer in the ceramic tile drying process.*

The thermal flow in a gas dryer is characterised by a combustion system that generates a stream of hot air that dries the tiles by convection from the outside inwards into the piece. The hot air generated, in addition to heating the pieces, also heats, in an undesirable manner, the whole dryer structure. The water vapour-laden combustion gases coming from the pieces must be extracted through the chimney at high temperature in order to avoid condensation. The low efficiency of gas combustion, together with heat losses produced by chimney extraction, leaks, insulation losses and residual heat in the pieces result in low energy efficiency of the system.

In the microwave system, heat is produced by the transformation of microwave energy in the tile material, so that heat losses are very low and special insulations are not required (cold oven). The system's efficiency is mainly due to the conversion of electricity into microwaves (greater than 70%), microwave energy not exploited by the material and the residual heat of the pieces (figure 1).

In a microwave system, heat is generated inside the piece due mainly to dipole rotation of the water molecules and to a lesser extent to ionic movement within the alternate electric field. The polar water molecule (H_2O) is very sensitive to the orientation of the electric field, generating heat and water evaporation. The water vapour generated must be extracted from the facility in order to avoid condensation and, on the other hand, so that the microwave power is focused on the piece being dried and is not lost in the water vapour-laden atmosphere.

One aspect to bear in mind using this drying technique is that a large amount of power can be applied instantly to the whole piece. If the power is very high, the drying kinetics allowed by the piece will be exceeded, so that vapour may be generated inside the piece which surpasses the permissible pore pressure and breaks the processed pieces in a catastrophic manner. Another point to bear in mind when using microwave equipment is the formation of overheated areas. It is very important to design a facility or microwave applicator for drying ceramic materials that maintains a high level of field uniformity in order to avoid the so-called hot spots during exposure.

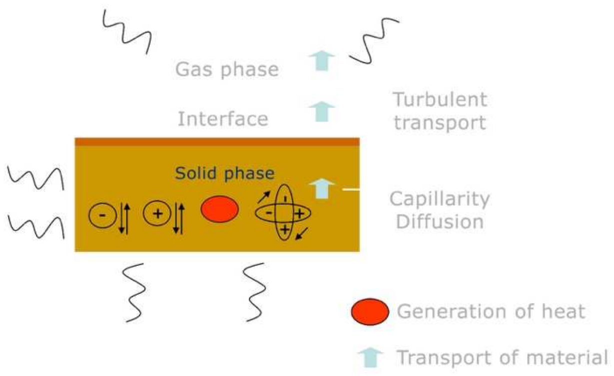

Figure 2. *Diagram of microwave drying.*

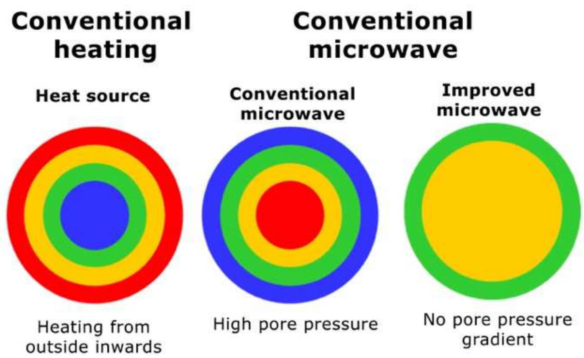

Figure 3. *Temperature gradient in a solid, dried using various techniques.*

When the moist tile is exposed to electromagnetic radiation by the microwaves, it heats up by converting the microwave energy into heat. The heating generated by the oscillating electric field in microwave frequency is related to permittivity (ε*), a complex variable that is defined by the following equation:

$$\varepsilon_{*} = \varepsilon' - j \cdot \varepsilon'' \qquad \textbf{Equation 1}$$

where the real part (ε') is called the dielectric constant and the imaginary component (ε'') is called the loss factor.

The dielectric constant (ε') reflects the ability of the material to store the energy when the material is subjected to an electric field. This accumulation of energy is produced fundamentally by the displacement of positive and negative charges from their equilibrium positions, under the effect of the applied electric field and against atomic and molecular attraction. The loss factor (ε'') is related to this phenomenon of energy absorption and dissipation in other forms of energy such as thermal energy.

The relation between the loss factor and the dielectric constant is known as tan delta and is related to the dissipative and capacitative behaviour of the materials.

In the microwave electric field, volumetric conversion of the heat produced by the material can be expressed in accordance with the following well-known expression:

$$Q_{GEN} = 2\pi f\, \varepsilon_0\, \varepsilon'' |E_{eff}|^2 \qquad \textbf{Equation 2}$$

where Q_{GEN} refers to the volumetric heat generated by microwave energy (W/m3); f, is the operating frequency (Hz); E_{eff}, is the intensity of the effective electric field (V/m); ε_0, is the permittivity in the vacuum, and ε'' is the loss factor of the irradiated material. As deduced from equation 2, for a working frequency, heat generation is determined by the loss factor (ε'').

Due to the importance of permittivity in the microwave drying of ceramic tiles, in this study test pieces were formed with different ceramic materials (different compositions) under different processing conditions (moisture, porosity...) to observe how this parameter varied.

3. OBJECTIVE

The main objective of this study was to evaluate the feasibility of microwave drying of bodies used for the manufacture of ceramic tiles, based on the determination of the dielectric properties of various ceramic materials.

4. MATERIALS AND EQUIPMENT

4.1. PERMITTIVITY MEASUREMENT

For measuring permittivity, ceramic test pieces with a diameter of 75 mm and thickness greater than 20 mm were formed in a universal testing machine, using four different ceramic compositions and various moisture contents. Two different porcelain tile compositions commonly used in the ceramic sector were used, in addition to a porcelain tile composition coloured with a black pigment and a porous white wall tile composition. The freshly pressed test pieces were immediately placed into a bag to maintain the moisture content until the permittivity testing was carried out.

The permittivity measurements were carried out in the model equipment: "Microwave Dielectric Measurement KIT, ITACA, µwaveanalizer", shown in figure 4. The sensor consisted of an open coaxial resonator, which emitted a low-intensity microwave signal at frequencies close to 2.45 GHz. Upon interacting with the sample, it reflected part of the microwave signal back to the sensor and absorbed the rest. The reflected signal determined the resonance frequency and the quality factor of the coaxial resonator. The dielectric properties of the sample were obtained based on the measurement of the resonance frequency and the quality factor [9].

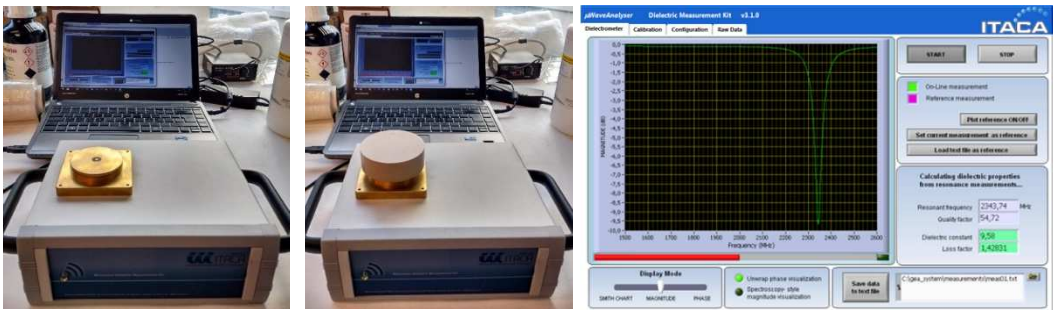

Figure 4. *Equipment for measuring dielectric permittivity.*

4.2. MICROWAVE TUNNEL OVEN

For the drying tests a microwave tunnel oven with a conveyor belt was used, model CEINNMAT KOTO W3/60 version KER (figure 5) adapted to the processing requirements with 8 radiators. The tests were carried out with a configuration of 4 radiators and 4 magnetrons at 2.45 GHz of 3000 W, each with a homogeneous electric field distribution system on the horizontal and vertical plane adjusted to the dielectric properties. The temperature measurements of the continuously processed product were obtained with the incorporation of calibrated pyrometers in the temperature intervals to be measured.

Figure 5. *View of the KOTO-W3/60microwave module installed in-plant, indicating the main elements in the 100% microwave version.*

In laboratory pieces, a conventional 700W microwave system with a rotating plate was used.

4.3. THERMOGRAPHIC CAMERA

Thermographic measurements of the pieces were taken right at the dryer exit, using a FLIR-T425 thermographic camera like the one shown in **figure 6**. In order to take the measurements, emissivity was adjusted to a value of 0.92 and images were captured, for various drying times, of pieces processed with a conventional dryer and of pieces dried with the microwave drying equipment.

Figure 6. *FLIR-T425 thermographic camera.*

5. RESULTS AND DISCUSSION

5.1. DIELECTRIC PROPERTIES

5.1.1. INFLUENCE OF POROSITY

The values of the dielectric constant, loss factor and loss tangent obtained in two tests, in which composition, thickness (25 mm) and moisture content were kept constant, but pressing pressure and therefore bulk density and total porosity of the formed piece were modified considerably, are shown in **table 1**. It was observed that even with this change in porosity, the loss tangent was very similar in both cases with values slightly higher in the test piece with greater density. The permittivity values obtained for the previous pieces, once they had been dried in an oven at 110 ºC to constant weight, are shown in **table 2**. In this case it was observed that the loss tangent of the more porous piece was significantly higher than that of the denser piece and the values obtained were lower than when they were moist.

Composition	Pressing pressure (kg/cm^2)	Moisture on a dry basis (%)	ε'	ε''	tg(δ)
Porcelain tile 1	200	6.0	7.24	2.231	0.308
Porcelain tile 1	400	6.0	7.56	2.412	0.319

Table 1. *Influence of porosity on permittivity with moist test pieces.*

Composition	Pressing pressure (kg/cm^2)	Moisture on a dry basis (%)	ε'	ε''	tg(δ)
Porcelain tile 1	200	0.3	3.73	0.518	0.14
Porcelain tile 1	400	0.3	4.22	0.112	0.03

Table 2*. Influence of porosity on permittivity with dry test pieces.*

5.1.2. INFLUENCE OF COMPOSITION

In table 3 and table 4, the results are shown for the various compositions processed under the same pressing conditions and constant thickness of 25 mm. As can be seen, the moisture values for the porcelain tile 1 composition were greater than the rest, but upon being dried all compositions had the same loss tangent with insignificant differences. The composition with and without pigment exhibited the same results.

Composition	Pressing pressure (kg/cm^2)	Moisture on a dry basis (%)	ε'	ε''	tg(δ)
Porcelain tile 1	400	6.0	7.56	2.412	0.319
Porcelain tile 2	400	6.0	7.35	1.860	0.253
Porcelain tile 2 + black pigment	400	6.0	7.86	1.986	0.253
White porous wall tile	400	6.0	8.14	2.266	0.278

Table 3. *Influence of composition on permittivity with moist test pieces.*

Composition	Pressing pressure (kg/cm²)	Moisture on a dry basis (%)	ε'	ε''	tg(δ)
Porcelain tile 1	400	0.3	4.22	0.122	0.03
Porcelain tile 2	400	0.3	3.80	0.081	0.02
Porcelain tile 2 + black pigment	400	0.3	4.07	0.106	0.03
White porous wall tile	400	0.3	4.30	0.130	0.03

Table 4. *Influence of composition on permittivity with dry test pieces.*

5.1.3. INFLUENCE OF TEST PIECE MOISTURE CONTENT

The results obtained by varying moisture content, pressing pressure and thickness (25 mm) for the same composition are shown in table 5. These results are plotted in figure 7 and figure 8. It can be observed that, on increasing test piece moisture content, both the dielectric constant and the loss factor increased. In regard to the loss tangent, it can be noted that it increased when sample water content was increased, but it stabilised upon reaching values of 4% moisture content on a dry basis. This fact indicates that in the initial drying phase the same irradiation power applied will generate the same driving force in the sample until this level of moisture content is reached. Based on this point, if it is sought to continue generating the same amount of heat, due to the fact that the permittivity is lower and the material begins to be more "transparent" to microwave radiation, greater power should be applied.

It may be noted that, from the point of view of drying optimisation, it is possible, as reported in the literature, that at the beginning of drying the diffusion of water from inside the ceramic piece to the exterior is sufficiently fast that a high irradiation power is permitted, but when the diffusion stage slows on heating the material with microwaves from the interior, water may be vaporised that makes the material explode or crack.

Composition	Pressing pressure (kg/cm²)	Moisture on a dry basis (%)	ε'	ε''	tg(δ)
White porous wall tile	400	6.0	8.11	2.278	0.28
White porous wall tile	400	4.0	6.52	1.800	0.28
White porous wall tile	400	2.0	4.97	1.004	0.20

White porous wall tile	400	0.3	4.31	0.128	0.03

Table 5. *Influence of test piece moisture content* ***on permittivity.***

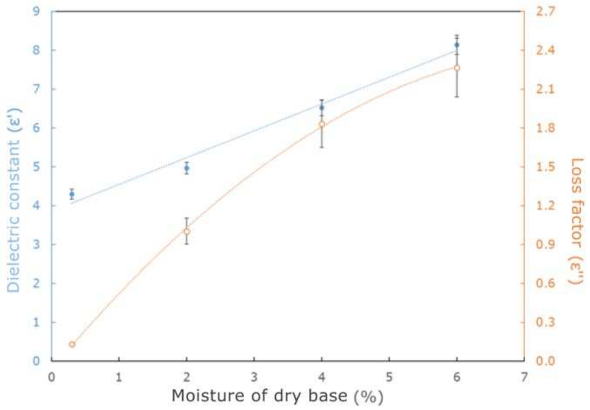

Figure 7. *Variation of the dielectric constant*

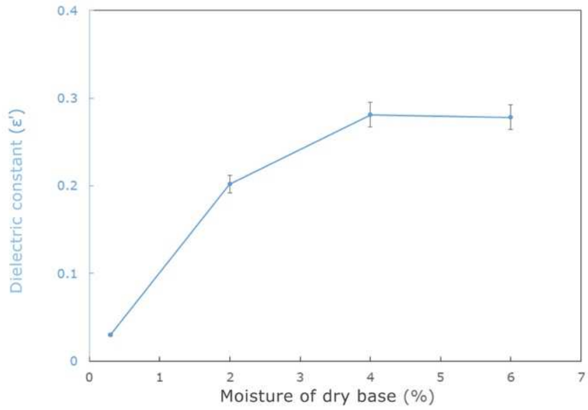

Figure 8. *Variation of the loss tangent with moisture content.*

5.2. DRYING TESTS OF GREEN CERAMIC PIECES

Conventional system

In a closed conventional microwave system, all the tested pieces reached the tile breaking point when the outside of the tile was over 60 °C. The irradiation with intermittencies of 20 s every 40 s also showed expansion fractures from internal vapour.

KOTO W3/60 conventional tunnel system

A first heating adjustment up to 100 ºC was made with increases in power until fracture, yielding power densities close to 0.3 W/cm^2. The temperature cycle recorded by the optical pyrometer and the moisture content of a porous white wall tile throughout a drying cycle in which the equipment was working continuously with irradiation of 750 W per magnetron are shown in **figure 9**. It can be observed how the initial moisture loss is quick without the piece excessively increasing its surface temperature.

Upon reaching 100 ºC the piece already had a low moisture content and, therefore, its loss factor was also low. The power supplied was lower in order to maintain the evaporation conditions without fractures.

In the tests carried out, it was observed that depending on the charge introduced in the drying equipment, the power supplied must be varied to obtain the same drying cycle, as the field density was spread over more material. It was also verified that there was an initial maximum applicable power and that when this was exceeded, the pieces exploded instantly.

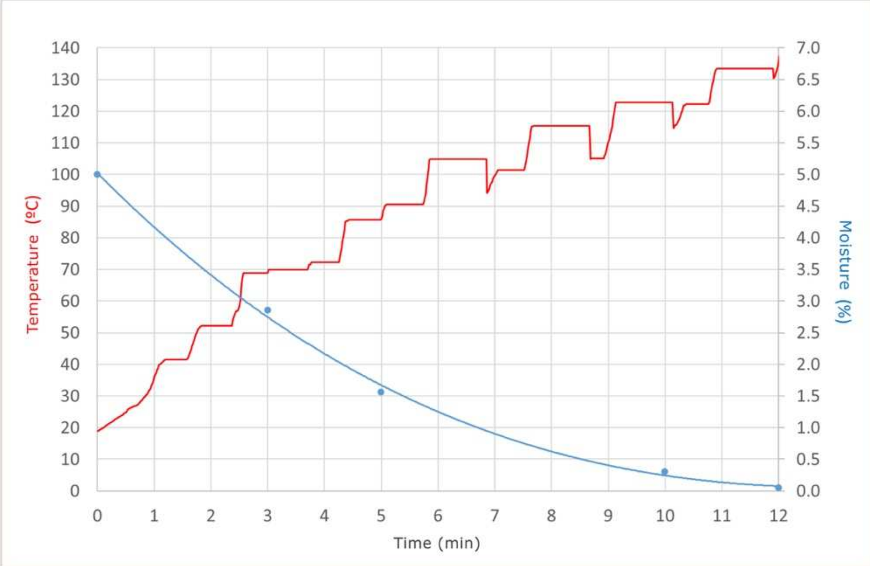

Figure 9. *Drying cycle of a green ceramic piece in the microwave oven.*

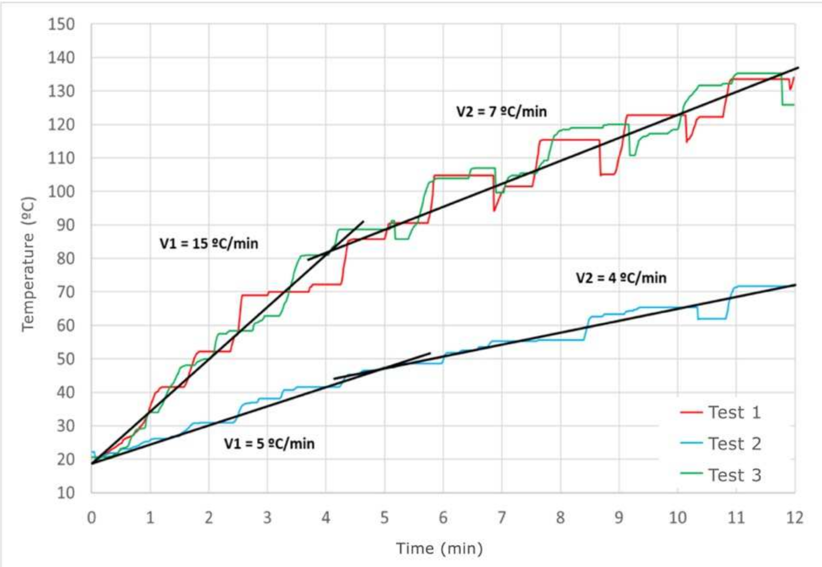

Figure 10. *Temperature recorded in drying green ceramic pieces in the microwave oven.*

Three different tests are shown in **figure 10**. In test 1, a ceramic piece of 25 cm x 50 cm was dried with a power of 4x750 W. An initial ramp of 15ºC/min was observed until minute 4 and a second one of 7ºC/min until minute 12, when the piece was totally dry. In test 2, the processing conditions were maintained but instead of charging a single piece, 4 pieces were charged. It was observed that the heating rates decreased and, upon finishing the 12-minute cycle, the pieces had not totally dried. In test 3, the power was increased to 4x1500 W and 4 fresh pieces were charged. It was observed in the test that there were heating ramps similar to those in test 1 and by the end of the cycle, 4 pieces had been obtained that were totally dry and in optimum conditions.

5.3. EVALUATION OF TEMPERATURE UNIFORMITY IN THE PIECES ON EXITING THE DRYING PROCESS

Thermographs of a piece measuring 25 cm x 50 cm dried using a vertical gas dryer and using the microwave dryer at 10 seconds after exiting the equipment and at 60 s are shown in **figure 11**. It can be observed that the temperature gradient in both cases was very uniform and that upon cooling, different cooling patterns were obtained depending on the heating system, a greater surface thermal homogeneity resulting with the microwave, so that it is expected not to produce problems during the glazing process.

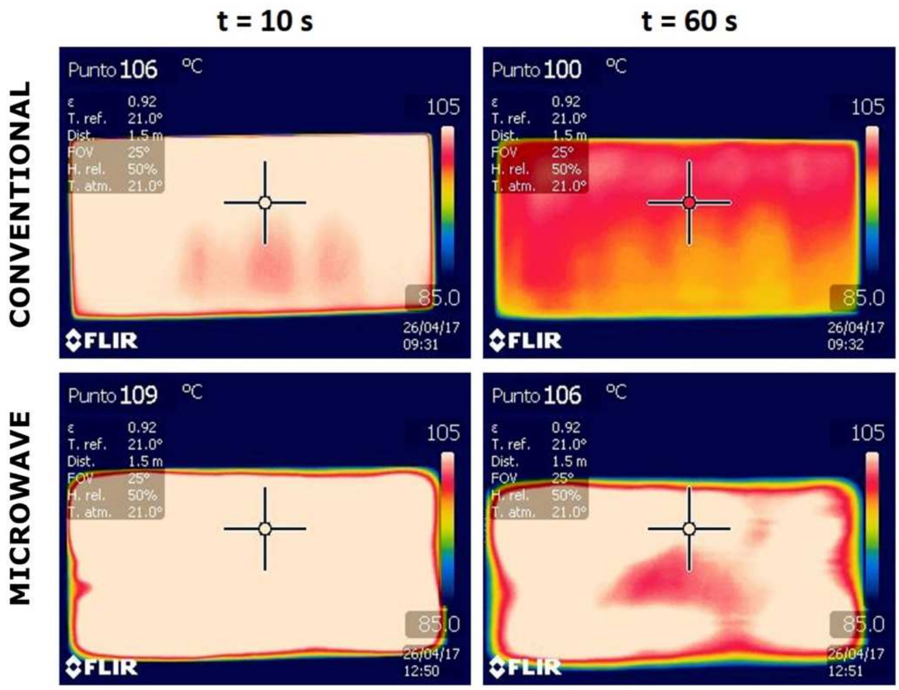

Figure 11. *Thermographs of a piece dried using a conventional dryer and another dried using the microwave equipment.*

6. CONCLUSIONS

The following conclusions may be drawn from this study:

- The dielectric properties of green ceramic tiles depend mostly on their water content, the formulation of the raw material exhibiting less influence.
- The drying of green tiles is a critical process in which fractures due to overpressure from water vapour are easily produced under conditions of homogeneous or intense irradiation. A drying system with electric field gradient was developed, which was optimised so that the rate of vapour release generated exceeded the critical pressure from pore overpressure and breakage of the piece.
- The new microwave irradiation design, together with an adequate radiation profile, allows high treatment rates and high tile exiting temperatures when required.

7. REFERENCES

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Modificar en las figuras 7 y 8

Moisture of dry base debería ser Moisture on a dry basis