INFLUENCE OF TEMPERATURE AND ULTRASOUND ON DRYING KINETICS AND ANTIOXIDANT PROPERTIES OF RED PEPPER

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ABSTRACT

Red pepper samples (1 m/s) were dried at different temperatures (30, 50, 70°C) without and with (20.5 kW/m³; 21.7 kHz) ultrasound application. The antioxidant capacity (AC), the total phenolic content (TPC) and the ascorbic acid (AA) content of fresh and dried red pepper samples were used as indicators of the quality of the dried products. Ultrasound application significantly improved the kinetics in every case, influencing not only the effective diffusivity but also the mass transport coefficient thus implying a reduction in energy needs. Drying significantly reduced AC, TPC and AA, this reduction being significantly smaller at 70°C due to the shorter drying time. Compared with conventional drying, ultrasound application reduced the loss of antioxidant properties at 50 °C but produced greater degradation at 70 °C, which points towards an optimal drying temperature when using ultrasound.

Keywords: dehydration, effective diffusivity, drying time, modelling, quality.
1. INTRODUCTION

Red pepper (*Capsicum annuum*), one of the most important crops in the world with about 2 million cultivated hectares [1], is appreciated for its organoleptic and nutraceutical quality and is consumed fresh, dried or preserved. When dried, it could be found in different forms, such as whole, in chips or powdered. Traditionally, red pepper has been dried by the own farmers using solar energy. Nowadays, drying is carried out at industrial scale, mainly by convective hot air drying. This permits the standardization of the production and avoids problems, such as the contamination with particles (dust) or insects.

The external layer of the peppers, the epicarp, has waterproof characteristics by means of which the natural dehydration of the fruits is limited. This makes the drying process highly time and energy consuming. In this sense, the conventional way to reduce the drying time is via the use of high drying temperatures. However, this fact can influence the quality of the dried product, affecting characteristics such as color, texture, ascorbic acid, total carotenoids, antioxidant properties, shrinkage or rehydration capacity [2, 3, 4]. Moreover, the low efficiency of convective drying and the relatively high operating cost make this operation prone to intensification. For this reason, different strategies have been considered with the aim of improving both the drying process kinetics and the quality of the obtained product. One of these strategies consists of the pretreatment of the pepper before drying with techniques like blanching or immersion in solutions of potassium or sodium metabisulfite, citric acid, calcium chloride, ascorbic acid or different osmotic solutions [5, 6]. Another strategy followed consists of the combination of hot air drying with other sources of thermal energy, such as microwave or infrared [4, 7].

High intensity ultrasound has been found to be an efficient way to intensify the convective drying of different food products [8, 9]. The effects produced by ultrasound
can reduce not only the internal (sponge effect, microchannel creation...) but also the external (microstreaming at interfaces...) mass transport resistance [10, 11]. Moreover, the mechanical stress produced by ultrasound could produce some microcracks at the epicarp level, facilitating the transport of water from the inner part of the pepper to the air-product interface. All these effects could allow the drying time to be shortened and the drying temperatures reduced [9, 10, 11]. On the other hand, the ultrasound effects can also influence the final quality of the obtained product [12]. As a general rule, it can be stated that the application of ultrasound at moderate temperatures not only produces no negative effects in product quality but can also contribute to an improvement in product quality [13, 14]. However, this fact is greatly dependent on the product considered [10].

Therefore, the aim of the study was to analyze the influence of ultrasound application on the hot air drying kinetics and antioxidant properties of red pepper.

2. MATERIALS AND METHODS

2.1. Raw material

Red peppers (Capsicum annuum var. Lamuyo) were purchased in a local market (Valencia, Spain). The fruits chosen were fresh in appearance and similar in size and color. Then, they were transported to the laboratory and maintained at room temperature (20±2 ºC) until processing (less than 24 h). After that, slab samples (30 x 30 x 5 mm) were obtained with the help of a sharp knife. The initial moisture content was measured by placing ground samples in a vacuum oven at 70°C and 200 mmHg until constant weight (24 h approx.) [15].
2.2. Drying experiments

The drying experiments were carried out in an ultrasonic assisted dryer described in detail by Riera et al. [16]. The system is provided with a vibrating cylindrical drying chamber driven by a piezoelectric transducer (21.8 kHz). An impedance matching unit permits the impedance output of the generator to be tuned to the transducer resonance frequency providing the system with a better electrical yield. A high intensity ultrasonic field (up to 154.3 dB) is produced inside the drying chamber while the drying air goes through it. The samples were randomly placed using a customized sample holder that allows a homogeneous air flow and ultrasonic treatment.

The experiments were conducted at 1 m/s and three different temperatures, 30, 50 and 70 °C, with the application of ultrasound (US; 20.5 kW/m³; 21.7 kHz) or not (AIR). All of these conditions were carried out in triplicate at least.

2.3. Modelling of drying kinetics

The modelling of the experimental data of drying kinetics was carried out to quantify both the influence of the drying temperature and the application of ultrasound on the drying rate. For this purpose, it was assumed that the samples exhibited infinite slab behavior and, therefore, the flux of the moisture during drying only takes place in one direction. Considering the effective moisture diffusivity as constant and the solid as isotropic, diffusion models based on Fick’s second law were used to mathematically describe the drying kinetics according to Equation 1.

\[
\frac{\partial W_p(x, t)}{\partial t} = D_e \frac{\partial^2 W_p(x, t)}{\partial x^2}
\]  

Where \( W_p \) is the local moisture content (kg water/kg dry matter, d.m.); \( D_e \) is the effective moisture diffusivity (m²/s); \( t \) is the time (s); and \( x \) is the direction of the water transport (m). Equation 1 was solved by assuming that the moisture content of the
samples was uniform at the beginning of the drying process. For modelling purposes, shrinkage was not taken into account and the epicarp of red pepper was considered as a waterproof layer. Then, the moisture transport takes place from this waterproof layer to the opposite side of the sample. This behavior will be similar to a symmetrical moisture transport in a slab twice as thick.

Due to the experimental low air velocity used in this study, the external mass transport resistance cannot be considered negligible when compared with the internal. For this reason, the external resistance was introduced into the model through the boundary condition shown in Equation 2.

\[-D_e \rho_{ss} \frac{\partial W_p(L, t)}{\partial t} = k (a_w(L, t) - \varphi_{air})\]  

(2)

Where \(\rho_{ss}\) is the density of the dry solid (kg d.m./m\(^3\)), \(L\) is the thickness of the pepper samples, \(k\) is the mass transfer coefficient (kg water/m\(^2\)s), \(a_w\) is the water activity and \(\varphi_{air}\) is the relative humidity of the drying air. This expression reflects how the moisture flows to the solid surface by diffusion and then moves by convection to the drying air.

The model was solved by means of an implicit finite difference method using the Matlab 2011B® (The Mathworks, Inc, Natick, USA) software that allows the evolution of the average sample moisture content to be estimated. The fitting of this model was carried out by the simultaneous identification of both kinetic parameters, effective moisture diffusivity \(D_e\) and mass transfer coefficient \(k\), which minimize the distance between the experimental and calculated average moisture contents. In this case, the optimization was carried out by means of the SIMPLEX method available in Matlab (fmin search function).

The fitting of the model was evaluated considering the percentage of explained variance (% VAR, Equation 3) and the mean relative error (MRE, Equation 4).
\[
\%\text{VAR} = \left[ 1 - \frac{S_{xy}^2}{S_y^2} \right] \cdot 100
\]  
\[
\%\text{MRE} = \frac{100}{N} \left[ \sum_{i=1}^{N} \left| \frac{W_{\text{exp}} - W_{\text{cal}}}{W_{\text{exp}}} \right| \right]
\]

Where \( S_y^2 \) and \( S_{xy}^2 \) are the variance of the experimental and the calculated data and \( W_{\text{exp}} \) and \( W_{\text{cal}} \) are the experimental and calculated moisture content of pepper, respectively.

2.4. Antioxidant properties of dried red pepper

The antioxidant properties were measured in extracts obtained from fresh and dried samples of red pepper. For that purpose, approximately 9 g of fresh sample or 3 g of dried were ground in a domestic mixer (D56, Moulinex). Extractions were carried out in a volumetric flask using ethanol 96\% as solvent and a red pepper weight/solvent volume ratio of 1 g/20 mL. The process took place at room temperature (20±1 °C) for 30 min with an ultrasonic pre-treatment for 1 min in an ultrasonic bath (Ultrasonic Cleaner USC-T, VMR). After that, the extracts were filtered using a quantitative filter paper, protected from light and stored at 4 ± 1°C.

2.4.1. Determination of the antioxidant capacity (AC)

The antioxidant capacity (AC) of red pepper was evaluated by using the Ferric-Reducing Ability Power (FRAP) method, following the procedure described by Pulido et al. [17] with some modifications. The FRAP reagent was prepared by adding 2.5 mL of 10 mM TPTZ (Fluka, Steinheim, Germany) in a 40 mM HCl (Panreac, Barcelona, Spain) solution plus 2.5 mL of 20 mM FeCl₃·6H₂O (Panreac, Barcelona, Spain) and 2.5 mL of 0.3 M acetate buffer (Panreac, Barcelona, Spain), pH 3.6. Then, 1 mL of fresh pepper extract or 2 mL of dried pepper extract were diluted until 10 mL with ethanol.
(96%). For testing purposes, 30 µL of distilled water and 900 µL of FRAP reagent were added to 30 µL of sample, then kept at 37°C for 30 min. The absorbance was read at 595 nm using a spectrophotometer (Helios Gamma, Thermo Spectronic, Cambridge, UK). The antioxidant capacity was evaluated by means of a calibration curve, previously determined using ethanol solutions of known Trolox (Sigma-Aldrich, Madrid, Spain) concentrations and expressed as mg Trolox per g of dry mass of red pepper. The measurements were taken in triplicate.

2.4.2. Determination of the total phenolic content (TPC)

The total phenolic content (TPC) of the dried samples was determined by the Folin–Ciocalteau method [18]. For that purpose, 1.25 mL of fresh pepper extract, or 2.5 mL dried pepper extract, were diluted until 5 mL with ethanol (96%). Then, 100 µL of this dilution was mixed with 200 µL of Folin–Ciocalteau’s phenol reagent (Sigma-Aldrich, Madrid, Spain) and 2 mL of distilled water. After 3 min at 25 °C, 1 mL of Na₂CO₃ (Panreac, Barcelona, Spain) solution (Na₂CO₃–water 20:80, p/v) was added to the mixture. The mix was kept in the dark at room temperature (20±1 ºC) for 1 h. Finally, the absorbance was read at 765 nm using a spectrophotometer (Helios Gamma, Thermo Spectronic, Cambridge, UK). The measurements were taken in triplicate. The standard curve was prepared with Gallic acid hydrate (Sigma-Aldrich, Madrid, Spain) at several known concentrations. The results were expressed as mg of Gallic acid (GAE) per g of dry mass of red pepper.

2.4.3. Determination of the ascorbic acid content (AA)

The ascorbic acid content (AA) was determined following the method proposed by Jagota and Dani [19]. For that purpose, 0.5 mL of sample extract was mixed with 0.5 mL of a trichloroacetic acid solution (7.5%). After 5 min at 4 °C, the mix was filtered. Then, 0.2 mL of extract, 2 mL of distilled water and 0.2 mL of diluted solution (1:10 v/v)
of Folin-Ciocalteau reagent were mixed and maintained for 10 min at room temperature. After that, absorbance was measured at 760 nm in a spectrophotometer (Helios Gamma, Thermo Spectronic, Cambridge, UK). A calibration curve was previously prepared with ethanol solutions of known ascorbic acid concentrations. The results were expressed as mg of ascorbic acid per g of red pepper dry matter. The determinations were carried out in triplicate.

The percentage of degradation of AC, TPC and AA at the end of drying was calculated by using Equation 5.

\[
\% \text{ Degradation} = \frac{C_o - C_f}{C_o} \times 100
\]

where \(C_o\) is the AC, TPC and AA of fresh samples and \(C_f\) is the AC, TPC and AA measurements in dried samples.

3. RESULTS AND DISCUSSION

3.1. Experimental drying kinetics

The initial moisture content of the red pepper was 11.6 kg water/kg dry matter, this value being similar to those found by Di Scala and Crapiste [2].

As expected, temperature significantly influenced the drying rate (Figure 1). The influence of the temperature on the drying rate was dependent on the product considered [20, 21]. In fact, the influence of the drying temperature on the drying rate of red pepper was very important. Thus, the drying time needed to achieve a moisture content of 0.2 kg of water/kg of dry matter by means of experiments carried at 70 °C was just 11 % of the time needed for experiments carried out at 30 °C (Table 1). This showed how the drying of red pepper is dependent on the air temperature considered.
This numerically illustrates the use of high temperatures for the conventional drying of red pepper on an industrial scale.

The application of ultrasound significantly increased the drying rate for every temperature tested (Figure 1); the lower the temperature tested, the shorter the drying time. Thus, when the drying was performed at 70 °C, the application of ultrasound reduced the drying time needed to achieve a moisture content of 0.2 kg water/kg dry matter by 32 % (Table 1). This reduction increased up to 62 % in experiments carried out at 50°C. In fact, the times needed in US-50 and AIR-70 experiments were not significantly different (p<0.05). This means that applying ultrasound led to an intensification of the drying process of red pepper that was equivalent to a rise in the drying temperature of 20 ºC.

3.2. Modelling

The modelling of experimental data permitted the quantification of the influence of the variables considered in the drying rate. In this case, the diffusion model considered which includes the external resistance to mass transfer, adequately fitted the experimental data of moisture content evolution. The percentage of variance explained (% VAR) by the model was higher than 99.8% for all experimental runs carried out (at least three replicates for each drying condition tested). As regards the mean relative error (MRE), the figures calculated were lower than 14 %, showing the quality of the obtained fit. Moreover, the trend of both the experimental moisture content and that calculated using the model was quite similar (Figure 2). No differences were found between the experiments carried out with or without ultrasound applications.

As far as the effective diffusivity ($D_e$) is concerned, the identified figure increased as the drying temperature rose (Table 2). For example, the mean value identified in AIR-70 experiments was approximately 250% greater than that identified in AIR-30 ones.
These results quantify the great influence of temperature on the drying rate of red pepper.

The identified $D_e$ increased when ultrasound was applied, this increase being significantly higher at the lowest temperatures considered. Thus, while the increase in $D_e$ observed at 70ºC was 51.5%, at 30 ºC it was 110.7 %. This has been previously observed in other products [13]. Thus, the effects of ultrasound on the drying rate were greater at lower temperatures, when the energy provided by the drying air was low. At higher temperatures, however, the greater level of energy provided by the drying air reduced the relative significance of the ultrasound effects. In any case, the results obtained showed that ultrasound can be used to intensify the process by reducing the internal mass transfer resistance, mainly at low drying temperatures, when the drying rate is slower.

Regarding the influence of drying temperature on the identified mass transfer coefficient ($k$), it was similar to the case of $D_e$; the higher the drying temperature, the higher the identified $k$ value (Table 2). Thus, in the AIR-70 experiments, the $k$ figure was 60% higher than that identified in AIR-30. As for the application of ultrasound, it also significantly increased the mass transfer coefficient identified at all temperatures tested. This increase was greater the lower the temperature considered. As an example, the application of ultrasound increased the value of $k$ by 73 % at 30 ºC, whereas this was only 27 % at 70 ºC. The effects of ultrasound on the external mass transport resistance are probably related with different mechanisms, such as oscillating velocities, pressure variation or interfacial instabilities [22], which produce the so-called "sonic wind". The sonic turbulence generates an intense microstirring, which contributes to a reduction of the boundary layer of diffusion and, therefore, leads to the decrease in the external resistance to moisture transport.
Also as a reference, as in the case of $D_e$, the average $k$ value identified in US-50 was not significantly different from that obtained in AIR-70. This means that the external resistance to mass transfer was similar with ultrasound application at 50°C to without ultrasound application at 70°C. Therefore, ultrasound intensifies the process attaining the same processing time with a reduction in the drying temperature. That could mean that the low electrical energy applied to the ultrasonic transducer can be largely compensated for by the reduction in both the drying time and/or drying temperature, helping to save energy. This fact must be validated by taking experimental measurements of the total energy consumption of the system.

### 3.3. Antioxidant properties

In order to address the influence of both drying temperature and the application of ultrasound during drying on the antioxidant properties of red pepper, the antioxidant activity (AC), the total phenolic content (TPC) and ascorbic acid content (AA) were measured in samples dried under the different conditions.

#### 3.3.1. Antioxidant capacity (AC)

Drying significantly reduced the antioxidant capacity of red pepper. As can be observed in Figure 3A, the percentage of degradation of the AC was in the range of 89 to 97%. The comparison with the results reported by other authors is complex for several reasons. Thus, only the antioxidant capacity of the dried samples obtained is commonly provided, but not that of the fresh samples. Moreover, the analytical methods used to determine the antioxidant capacity are different [23, 24] making it difficult to assess the influence of the drying process. In any case, the percentage of degradation of the AC was slightly greater than that reported by Zhou et al. [4] also using the FRAP method, 65%. The data for red pepper was of much greater significance than that observed in other food products, such as apple [25]. This fact can
be related with the longer time needed for the purposes of drying red pepper. Thus, the prolonged exposure of samples to the drying air can facilitate oxidation [26].

The drying temperature affected the degree of degradation of AC. Thus, the value obtained in AIR-30 experiments, 96.3±0.2 %, was higher than that obtained in AIR-70 °C, 89±0.1%. The relationship between drying temperature and drying time may be what mainly explains these results. The long drying times at low drying temperatures may promote a decrease in antioxidant capacity [27]. On the contrary, at high temperatures, not only do degradation reactions take place but Maillard-derived compounds with antioxidant activity are also generated and accumulated, which could also enhance the antioxidant properties [28]. Thus, despite 30 °C being a moderate drying temperature, the longer exposure of samples to the drying air can facilitate degradation reactions. On the contrary, at 70 °C, the faster drying can partially compensate the damage produced by high temperatures. The highest percentage of AC degradation was obtained at 50 °C, probably due to the combination of a relatively long process with an intermediate temperature.

As regards the application of ultrasound, the AC degradation observed in US experiments at 30 °C (US-30) was not significantly different from that obtained in the AIR experiments. In this case, the drying time reduction produced by ultrasound might not be sufficient to affect the percentage of AC degradation. In US-70 experiments, the percentage of AC degradation was lower than that obtained at 30 °C (both US and AIR experiments) but slightly higher than that obtained in AIR-70 experiments. In previous research, ultrasound was also found to have a negative effect on AC when applied during high temperature drying [13]. This can be attributed to some cell disruption caused by the mechanical stress produced by the acoustic waves. In the case of US-50, the AC degradation was significantly lower than in AIR-50 experiments. In this case, the shortening of the drying process produced by ultrasound could be enough to compensate the possible negative effects linked to the acoustic energy.
3.3.2. Total phenolic content (TPC)

The influence of drying on the TPC of red pepper was quite similar to the case of AC. As a general rule, it can be concluded that drying significantly reduced the phenolic content of samples. This decrease has been previously reported [4] and can be attributed to irreversible chemical changes during drying. The degradation was more important at temperatures of 30 and 50 ºC (Figure 3B). At 70 ºC, although important, the percentage of degradation was significantly lower. The lower impact of AIR-70 experiments on TPC could not only be related with the shortest drying process, as in the case of AC, but also with the possible appearance of new phenolic compounds due to the treatment at this temperature [4]. In fact, it has been reported that some phenolic acids can be generated because of the structural transformation of polyphenols due to the high temperatures used during drying processes [29].

Compared with AIR experiments, the application of ultrasound did not affect the TPC degradation at 30 ºC. In the case of US-70 experiments, the degradation was greater than that obtained in AIR-70 experiments but not significantly different from that obtained in AIR-30 or US-30. Do Nascimento et al. [13] and Rodriguez et al. [30] found similar results for the ultrasonically assisted drying of passion fruit peel and apple, respectively. The combination of high drying temperatures and the mechanical stress induced by ultrasound can generate greater cellular damage, which may affect the polyphenol content. On the contrary, at 50 ºC, there was less TPC degradation in US experiments than in AIR experiments. As in the case of AC degradation, the shortening of the drying time produced by the application of ultrasound at this intermediate temperature can reduce the negative effects of both drying and ultrasound itself on the phenolic content.
3.3.3. Ascorbic acid content (AA)

The initial ascorbic acid content of fresh samples was 2.7±0.4 mg/100g of fresh sample. This value was similar to that found by Vega-Galvez et al. [31] (1.60±0.26 mg/100g of sample) also for *Capsicum annuum* var. Lamuyo, but lower than that obtained by Vega-Galvez et al. [3] for *Capsicum annuum*, L. var. Hugarian (188.2±0.4 mg/100g of sample). As in the case of other antioxidant properties tested, the AA content was greatly affected by the drying process, the percentage of degradation ranging from 90 to 97%. Di Scala and Capriste [2] reported a degradation of ascorbic acid in the range of 82-88 % during the air drying of red pepper at 50, 60 and 70 ºC and Vega-Galvez et al. [3] found a maximum loss of 98.2% in samples dried at 90 ºC. On the contrary, Carrillo-Montes et al. [24] obtained an ascorbic acid retention of 75.5% after the intermittent drying (60ºC) of red pepper pre-treated in a CaCl₂ solution. Therefore, the pre-treatment of samples can contribute to a reduction in the degradation of this vitamin during drying [31]. In fact, the blanching of samples prior to drying can significantly reduce the activity of the polyphenol oxidase [26].

The degradation observed in AIR-50 experiments was slightly lower than that observed in the AIR-30 ones, but this difference was not significant (p<0.05). For experiments carried out at 70 ºC, the percentage of degradation was significantly lower (Figure 3C). Due to the fact that ascorbic acid is a compound which is highly prone to oxidation, the shortening of the time during which samples were exposed to drying air can be one of the main reasons for the better preservation at a higher drying temperature.

The percentage of degradation of AA content was similar in all the experiments carried out with ultrasound application, regardless of the drying temperature, and similar to the values obtained in AIR-30 experiments. In this case, the negative effects of ultrasound on ascorbic acid content when the drying temperature increased were not masked by the shortening of the drying process.
Consequently, these results showed that ultrasound application during the drying of red pepper had no significant effect on the antioxidant properties studied when the drying took place at the lowest temperature tested, 30 °C; had a positive influence when drying took place at the intermediate temperature, 50 °C and a negative effect at the highest drying temperature, 70 °C. Due to the fact that the AC, TPC and AA degradation values measured in experiments carried out at 30 and 50 °C were lower than those performed at 70 °C, the ultrasonically assisted drying of red pepper can only be justified by a reduction in energy consumption linked to a decrease in the drying time or drying temperature. This last fact must be studied in depth.

**CONCLUSIONS**

The red pepper drying rate was highly dependent on the drying temperature. The application of ultrasound significantly accelerated the red pepper drying process. However, the use of high drying temperatures reduced the loss in the antioxidant properties of red pepper. The application of ultrasound only significantly reduced the degradation at intermediate temperatures, but increased them at the highest. Therefore, ultrasonically assisted red pepper drying can only be considered in terms of reducing the drying temperature in order to save energy. Moreover, further experimentation is needed for the purposes of elucidating the time/temperature effects.

**ACKNOWLEDGEMENTS**

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FIGURE CAPTIONS

Figure 1. Experimental drying kinetics of red pepper ($W/W_0$, average moisture content/initial average moisture content) at different temperatures (30, 50, 70 °C) without (AIR) and with (US) ultrasound (20.5 kW/m$^3$).

Figure 2. Experimental dimensionless moisture content (moisture content/initial moisture content, $W/W_0$) versus calculated for the drying of red pepper at different temperatures (30, 50, 70 °C) with (US) ultrasound (20.5 kW/m$^3$) application.

Figure 3. Degradation of the antioxidant capacity (AC) (A), total phenolic content (TPC) (B), and ascorbic acid content (AA) (C) of red pepper after drying at different temperatures (30, 50, 70 °C) without (AIR) and with (US) ultrasound (20.5 kW/m$^3$) application. Mean values and Least Significant Difference intervals (p<0.05) are shown.
Table 1. Drying of red pepper (1 m/s) at different temperatures, without and with ultrasound (20.5 kW/m³; 21.7 kHz) application. Time needed to achieve a final moisture content of 0.2 kg water/kg of dry matter.

Table 2. Effective diffusivity ($D_e$) and mass transfer coefficient ($k$) identified for the drying of red pepper at different temperatures, without and with ultrasound (20.5 kW/m³; 21.7 kHz). Mean values and standard deviation. Identical letters in the same column indicate homogeneous groups obtained from LSD intervals (p<0.05).
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Table 2. Effective diffusivity (D_e) and mass transfer coefficient (k) identified for the drying of red pepper at different temperatures, without and with ultrasound (20.5 kW/m³; 21.7 kHz). Mean values and standard deviation. Identical letters in the same column indicate homogeneous groups obtained from LSD intervals (p<0.05).

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