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Ultrasound assisted low-temperature drying of kiwifruit: Effects on drying kinetics, bioactive compounds and antioxidant activity

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Abstract

BACKGROUND: Low-temperature drying is considered to be a promising technique for food processing. It preserves thermolabile compounds and might be intensified by acoustic assistance. The effect of acoustic assistance (20.5 kW m^{-3}) during low-temperature drying of kiwifruit (at 5, 10 and 15 °C, and 1 m s^{-1}) on drying kinetics, bioactive compounds (such as ascorbic acid, vitamin E, and total polyphenols), and antioxidant activity was studied.

RESULTS: Drying time was shortened by 55–65% when using power ultrasound. A diffusion model was used to evaluate the drying kinetics. The effective diffusion coefficient increased by 154 ± 30% and the external mass transfer coefficient increased by 158 ± 66% when ultrasound was applied during drying, compared with drying without ultrasound application. With regard to bioactive compounds and antioxidant activity, although samples dried at 15 °C presented significantly higher ($P < 0.05$) losses (39–54% and 57–69%, respectively) than samples dried at 5 °C (14–43% and 23–50%, respectively) when ultrasound was not applied, the application of ultrasound during drying at 15 °C significantly reduced ($P < 0.05$) those losses in all quality parameters (15 – 47% and 47 – 58%, respectively).

CONCLUSION: Overall, low-temperature drying of kiwifruit was enhanced by acoustic assistance preserving bioactive compounds and antioxidant activity, especially at 15 °C.

Keywords: kiwifruit; low-temperature drying; power ultrasound; bioactive compounds

INTRODUCTION

Kiwifruit crops and consumption have increased during recent decades, this fruit being appreciated by consumers in Western countries as an exotic food with health benefits mainly related to its antioxidant content.[1] According to Du *et al.*[2] kiwifruit is characterized by its high ascorbic acid and vitamin E content and other useful compounds such as carotenoids, chlorophylls, flavonoids, and minerals. The kiwi, like many other fruits, is highly perishable, so the development of optimal methods for its conservation is interesting, taking into account the fact that consumers demand minimally processed food products, with similar or equivalent nutritional and sensorial attributes to the fresh product and in compliance with food safety requirements.[3]

Convective drying is one of the most commonly used techniques for food preservation in industry. Drying improves food stability by reducing water activity, but it also promotes color and texture changes, shrinkage, and losses of different nutritional biocompounds.[4] The extent of these changes, especially the losses of thermolabile biocompounds, is usually higher as both the drying temperature and the drying time increase.[5]

Low-temperature drying has thus been considered a promising technique for food preservation. Working at atmospheric pressure and using air at a temperature below standard room conditions and close to the water freezing point, and with low relative humidity (below 30%), it has been found to preserve thermolabile compounds.[6] For instance, according to Santacatalina *et al.*[4] and Rodríguez *et al.*,[7] the losses of some biocompounds (total polyphenols and flavonoids) in Granny Smith apples during convective drying were 25% at 0 °C, 28% at 10 °C, but 39% at 30 °C. Using this technique, a previous freezing process that would have had to be conducted during freeze-drying is not required[4]. Any extra quality loss caused by the ice crystal formation during freezing, as well as the high cost of freezing and vacuum, is therefore avoided.

However, by decreasing the air temperature, the mass transfer rate during drying also decreases, thus making low-temperature drying a time-consuming operation compared with conventional convective drying at high temperatures. Low-temperature drying is prone to be intensified by using complementary techniques to

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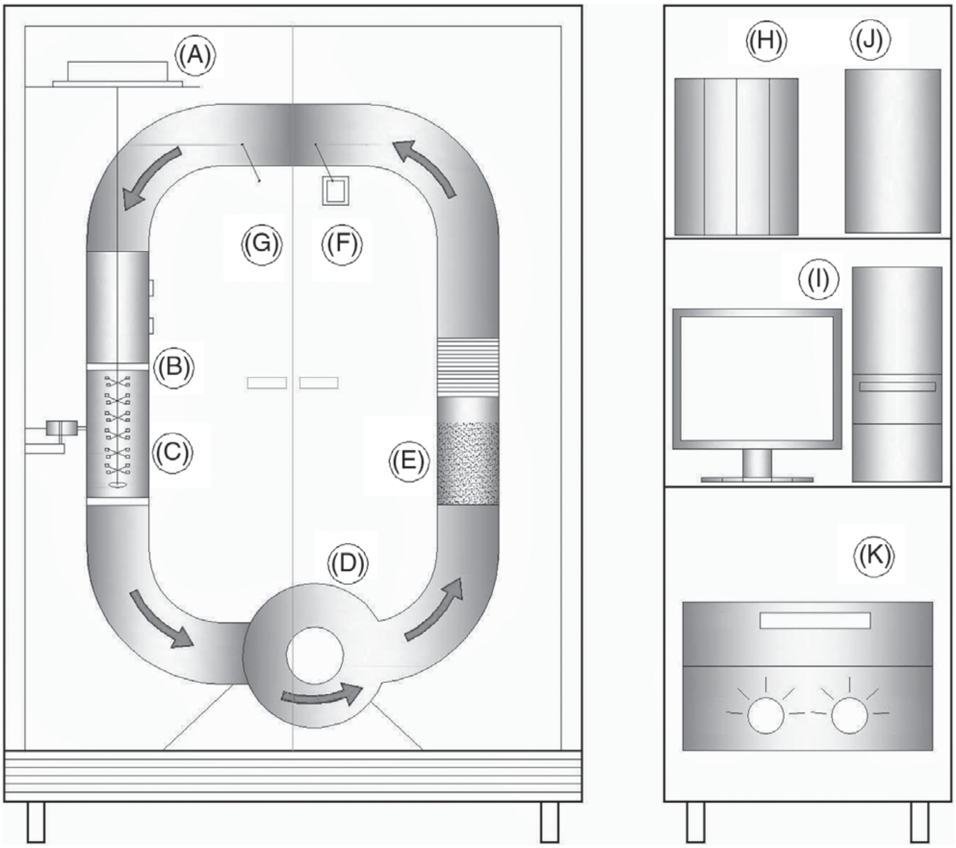

Figure 1. Schematic layout of the drying system. Arrows indicate the air blowing direction. A: Electronic scale; B: Drying chamber; C: Cylindrical radiator; D: Fan; E: Desiccant material; F: Humidity and temperature sensor; G: Flow sensor; H: Proportional-integral-derivative controller; I: Computer; J: Power ultrasonic transducer; K: Dynamic resonance controller and power amplifier.

enhance the water removal.[8] One of these techniques is the use of power ultrasound, which has been applied during the convective drying of food products, proving its efficiency in shortening the drying time.[9,10] Moreover, according to García-Pérez *et al.*[10] the development of a new family of power generators with extensive radiating surfaces has significantly contributed to the implementation at industrial scale of several applications in sectors such as the food industry, environment, and manufacturing. But, changes in biocompound content and antioxidant activity in food products during low-temperature drying, with and without ultrasound application, have barely been studied in the literature.

The aim of this study was therefore to analyze the influence of the drying temperature and acoustic assistance on the low-temperature drying kinetics, the ascorbic acid and vitamin E content, the total polyphenol content, and antioxidant activity of kiwifruit.

MATERIALS AND METHODS

Sample preparation

Kiwifruit (*Actinidia deliciosa* cultivar Hayward) were purchased in a local market in Spain. To ensure homogeneity of ripeness, they were selected with a total soluble solid content, measured as °Bx, of 13.0 ± 0.5 °Bx and pH of 4.8 ± 0.6. The initial moisture content (W_0), obtained by using the AOAC method N° 934.06,[11] was 5.8 ± 0.4 kg kg d.m.$^{-1}$ The fruits were peeled and the seedless and coreless pulp was shaped into parallelepipeds of 1.0 x 1.0 x 0.5 cm.

Acoustically assisted low-temperature drying experiments

Drying experiments were carried out in a convective dryer with air recirculation, air velocity, temperature control, and an ultrasonically activated drying chamber. The whole system is assembled into an industrial upright fridge ACRV-125-2 (Coreco, Spain). A schematic layout of the drying system is shown in Fig. 1.

Air flow is driven by a medium-pressure fan TD-800/200 ECOWATT (Soler & Palau, Spain) and its temperature and flow rate is measured near the drying chamber by a flow sensor SS 20.250 (Schmidt, Germany). The air velocity (from 0.1 to 2.0 m s^{-1}) is controlled by a proportional-integral-derivative algorithm, using an integrated intelligent real-time controller cRio-9092/3/4 (National Instruments, USA), which controls the fan speed, comparing the flow sensor signal to the set air velocity. The air temperature and relative humidity are measured in the air duct near to the drying chamber, using a DKK humidity and temperature sensor (Galltec+Mela, Germany). To keep the relative humidity low, the air is forced through a tray containing desiccant material, activated alumina pellets 1/4 (Alphachem, Spain), which are periodically renewed.

A high-power ultrasonic application system is assembled, being connected to the convective dryer used as the drying chamber. It mainly consists of a cylindrical radiator (internal diameter 100 mm, height 310 mm, thickness 10 mm) driven by a power ultrasonic transducer (frequency 21.9 kHz, impedance 369 Ω, power capacity 90 W). An ultrasonic signal is generated and fitted to minimize the phase between electric voltage and intensity by a dynamic resonance controller APG-AC01 (Pusonics, Spain) and the power capacity is maintained through a power amplifier RMX 4050HD (QSC, USA). Finally, an impedance matching unit APG-AC01 (Pusonics, Spain) (impedance from 50 to 500 Ω and inductance from 5 to 9 mH) is used to optimize the ultrasonic application electronically. The ultrasonic system provides an average sound pressure level in the drying chamber of 155 dB.

Air flows through the cylindrical radiator, where the samples are placed on a hanging stainless steel tree. The determination of the drying kinetics was carried out by weighing the samples at selected times using an electronic scale C-6200 CBC (Cobos, Spain) connected to the Compact FieldPoint programable automation controller system (National Instruments, USA) by an interface RS-232. A weighing sequence was programmed in the controller to provide an accurate measurement. The fan was stopped and the ultrasonic system set to a minimum electric voltage (ca. 1.0 V) by means of the RS-232 interface. The weight measurement was taken 20 times and the average was considered as the definitive figure. This was done in order to avoid the excess noise produced by the vibration of the cylindrical radiator.

An application was developed to provide overall control and monitoring of the drying process using LabVIEW 2013 programming code (National Instruments, USA). This application provides information on the air flow, air temperature, drying time, and sample weight during the drying process.

Two sets of drying experiments were carried out at temperatures of 5, 10, and 15 °C, air velocity of 1 m s^{-1} and relative air humidity of 32 ± 7%. In set 1, drying took place without ultrasound assistance (AIR experiments). In set 2, power ultrasound of 50 W (20.5 kW m^{-3}) was applied during the drying experiments (AIR + 80% weight loss was achieved, which corresponded to a final moisture content of ca. 0.5 kg kg d.m.$^{-1}$ Finally, each experiment was carried out in triplicate.

Diffusion model

The drying process was described by a mathematical model considering the liquid diffusion as the main transport mechanism. Thus, the model consisted of the microscopic mass transfer balance combined with Fick's diffusion second law. Moreover, the process was considered to be isothermal. The governing equation for a differential element of the parallelepiped shape was formulated (Eqn (1)):

$$D_e\left(\frac{\partial^2 W_l}{\partial x^2} + \frac{\partial^2 W_l}{\partial y^2} + \frac{\partial^2 W_l}{\partial z^2}\right) = \frac{\partial W_l}{\partial t} \tag{1}$$

The constant, isotropic, and effective diffusion coefficient (D_e), representative of the global transport process, might include molecular diffusion, liquid diffusion through the solid pores, vapor diffusion and all other factors that affect drying characteristics.[12] It was also assumed that no contraction or deformation of the solid particle occurred during the process. As an initial condition, the moisture distribution inside the solid was considered to be uniform at the beginning of the process (Eqn (2)). As boundary conditions, the moisture distribution symmetry (Eqn (3)) and the external mass transfer at the solid surface (Eqn (4)) were assumed.

$$W_{l(x,y,z)}\Big|_{t=0} = W_0 \tag{2}$$

$$\frac{\partial W_{l(x,y,z)}}{\partial x}\Big|_{x=0} = \frac{\partial W_{l(x,y,z)}}{\partial y}\Big|_{y=0} = \frac{\partial W_{l(x,y,z)}}{\partial z}\Big|_{z=0} = 0 \tag{3}$$

$$\begin{aligned} -D_e\rho_{dm}\frac{\partial W_{l(x,y,z)}}{\partial x}\Big|_{x=L} &= h_m\left(\varphi_e - \varphi_\infty\right)\\ -D_e\rho_{dm}\frac{\partial W_{l(x,y,z)}}{\partial y}\Big|_{y=L} &= h_m\left(\varphi_e - \varphi_\infty\right)\\ -D_e\rho_{dm}\frac{\partial W_{l(x,y,z)}}{\partial z}\Big|_{z=L} &= h_m\left(\varphi_e - \varphi_\infty\right) \end{aligned} \tag{4}$$

The sorption isotherm for kiwifruit reported by Moraga *et al.*[13] and the psychometric data were considered to complete the model.

COMSOL Multiphysics® 5.1 (COMSOL Inc., Sweden) software was used to solve the mathematical model, applying the finite elements method. The complete mesh consists of 9902 elements resulting in 2110° of freedom. Matlab 2014a® (The Mathworks, Inc., USA) software was used to develop the algorithm to identify both the effective diffusion (D_e) and the external mass transfer (h_m) coefficients by using the fminsearch function of Matlab, which uses the simplex search method described by Lagarias *et al.*[14] The coefficients were identified from each drying curve through the minimization of the objective function (mean relative error, MRE) given by Eqn (5), which relates experimental and calculated average moisture content.

$$\mathrm{MRE} = \frac{100}{n}\sum_{i=1}^{n}\left|\frac{W_{\exp_i} - W_{\mathrm{cal}_i}}{W_{\exp_i}}\right| \tag{5}$$

Determination of ascorbic acid content

The experimental procedure used to determine ascorbic acid content (AAC), as a reduced form of Vitamin C, in fresh and dried kiwifruit samples was the procedure proposed by Salkić, *et al.*[15] A sample (ca. 1.0 g) was homogenized with 10 mL of 0.056 mol L^{-1} sodium oxalate with an Ultra-Turrax T25 Digital (IKA, Germany) at 13 000 rpm for 30 s. The extraction mixture was left standing for 5 min. The homogenate was filtered and an aliquot of 1.0 mL of the extract was diluted to 10 mL with 0.056 mol L^{-1} sodium oxalate. Absorbance readings were made in an UV–Vis spectrophotometer UV-2401 (Shimadzu, Japan) at 266 nm at 25 °C, using 0.056 mol L^{-1} sodium oxalate as blank. Calibration curves were made using L-ascorbic acid as standard. The results were expressed as mg of L-ascorbic acid equivalent g d.m^{-1}.

Determination of vitamin E content

Determination of the vitamin E content (VEC) in fresh and dried kiwifruit samples was carried out according to the methodology proposed by Fernandes *et al.*[16] The sample (ca. 1.0 g) was homogenized with 10 mL of distilled water with an Ultra-Turrax T25 Digital (IKA) at 13 000 rpm for 1 min. Then, 1 mL of sodium hydroxide 0.5 mol L^{-1} was added to the sample and then heated at 70 °C for 30 min in a water bath. The mixture was cooled down using an ice bath, and 5 mL of hexane was added and the mixture was vigorously shaken for 1 min using a vortex. The supernatant (hexane phase) was collected and analyzed spectrophotometrically at 215 nm with a UV– Vis spectrophotometer UV-2401 (Shimadzu) using hexane as blank. Calibration curves were made using α-tocopherol as standard. The results were expressed as mg of α-tocopherol equivalent g

Total polyphenol content and antioxidant activity determinations

Methanol extracts from the kiwifruit samples were prepared according to the methodology described by Heredia and Cisneros-Zevallos.[17] Samples were accurately weighed (ca. 1 g fresh samples or ca. 0.1 g dried samples) and 20 mL of methanol extraction solvent was added. The mixture was homogenized using a T25 Digital Ultra-Turrax (IKA) at 13 000 rpm for 1 min at 4 °C and the solution obtained was refrigerated overnight. Mixtures were centrifuged at 4000 rpm for 10 min and then filtered. The extracts were refrigerated at 4 °C until analysis. At least four methanol extracts were prepared for each sample.

The total polyphenol content (TPC) was determined by means of the Folin– Ciocalteu assay according to Singleton and Rossi.[18] The antioxidant activity (AA) was spectrophotometrically determined using the ferric reducing antioxidant power (FRAP), cupric reducing antioxidant capacity (CUPRAC), and 2 2′-azino-bis(3-ethylbenzothiazoline-6-sulfonic acid) (ABTS) methods as described by Benzie and Strain,[19] Apak *et al.,*[20] and Re *et al.,*[21] respectively. Absorbance measurements were carried out at 25 °C in a UV/Vis/NIR spectrophotometer Multiskan Spectrum (Thermo Scientific, Finland) at 745 nm (TPC), 593 nm (FRAP), 450 nm (CUPRAC), and 734 nm (ABTS). Absorbance measurements were correlated with standard curves (0–250 mg L^{-1} gallic acid for TPC and 0–400 mg L^{-1} Trolox for AA). The results were expressed as mg of gallic acid equivalent g d.m^{-1}. for the TPC, while the AA was expressed as mg of Trolox equivalent g d.m^{-1}.

Statistical analyses

All quality determinations were carried out in triplicate and results were expressed as the percentage loss (%) of the quality attribute using the figures determined for the fresh sample as reference (Eqn (6)):

$$\text{Loss (\%)} = \frac{[\text{Fresh} - \text{Dried}]}{\text{Fresh}} \times 100 \qquad (6)$$

Data were averaged from replicates and reported as an average figure ± standard deviation. An analysis of variance (ANOVA) was applied to analyze the effects of both the drying temperature and the acoustic assistance during drying on the ascorbic acid and vitamin E contents, the total polyphenol content, and the antioxidant activity. Means were compared using Tukey's test at $P < 0.05$. Statistical analyses were carried out using Language and Environment for Statistical Computing R (R Core Team, Austria).

The percentage of explained variance (var) was also used to evaluate further the accuracy of the simulation obtained (Eqn (7)):

$$var = \left[1 - \frac{S_{yx}}{S_y}\right] \times 100 \qquad (7)$$

RESULTS AND DISCUSSION

Drying kinetics

Figure 2 shows the experimental drying curves (dots) for the different drying temperatures (5, 10, and 15 °C) without (AIR) and with an acoustic assistance of 20.5 kW m^{-3} (AIR + US). Although low-temperature drying is a time-demanding process, the use of acoustic energy promoted a remarkable reduction of the drying time. As an example, to reach a moisture content of 0.65 ± 0.03 kg kg d.m.$^{-1}$, the drying time for the AIR samples dried at 5, 10 and 15 °C were of ca. 60, 34, and 19 h, respectively, whereas when ultrasound was applied (AIR + US), the drying time decreased by 62%, 65%, and 55% at 5, 10, and 15 °C, respectively. Santacatalina *et al.*[22] also studied the influence of acoustic assistance during the low-temperature drying at 1 m s^{-1} of eggplant cubes. These authors reported reductions in the drying time of 80% and 58% when an acoustic assistance of 20.5 kW m^{-3} was applied at drying temperatures of 0 and 10 °C, respectively. Similarly, in the low-temperature drying of apple cubes from 0 to 10 °C (2 m s^{-1}), Santacatalina *et al.*[4] observed that the acoustic assistance (20.5 kW m^{-3}) increased the drying rate of apples at every drying temperature tested. In this case, the reduction of the drying time promoted by ultrasound application was similar (60%) in the experiments carried out at 0, 5, and 10 °C.

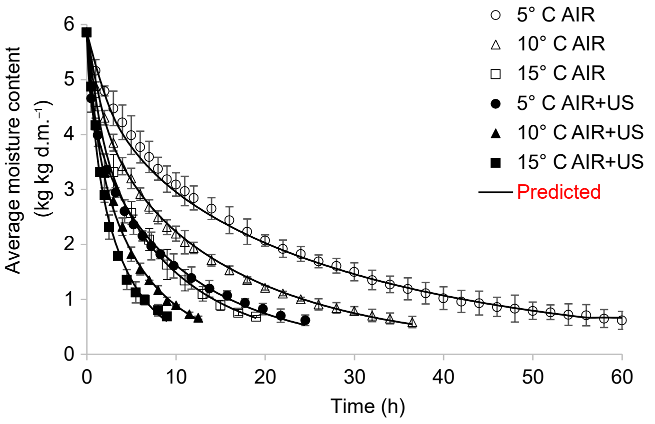

Figure 2. Experimental and predicted drying kinetics of kiwifruit without (AIR) and with 20.5 kW m^{-3} of acoustic assistance (AIR + US) at 5, 10, and 15 °C. Average values ± standard deviations.

Diffusion model

As described above, the diffusion model was designed for a parallelepiped. By minimizing the differences between the experimental drying curves and the calculated ones, the effective diffusion coefficient D_e and the external mass transfer coefficient h_m, were simultaneously determined for each experiment. Results are presented in Table 1, together with the average MRE and var, obtained by comparing the experimental and simulated drying curves.

The identified D_e in the AIR experiments ranged from 1.37 (5 °C) to 4.30 × 10^{-11} m^2 s^{-1} (15 °C), but in the AIR + US experiments this coefficient ranged from 3.67 (5 °C) to 9.45 × 10^{-11} m^2 s^{-1} (15 °C). These figures were within the range of those observed by Santacatalina *et al.*[4] in the low-temperature drying of apple (2 m s^{-1}). Santacatalina *et al.*[4] reported D_e figures from 3.3 (0 °C) to 8.8 × 10^{-11} m^2 s^{-1} (10 °C), when drying was carried out without acoustic assistance, and from 8.6 (0 °C) to 22.3 × 10^{-11} m^2 s^{-1} (10 °C), when an acoustic power of 20.5 W m^{-3} was applied. Higher figures were reported by Darıcı and Şen[23] in the hot-air drying of kiwifruit slices of 4 mm (2.3–7.0 × 10^{-10} m^2 s^{-1}) and 6 mm thickness (2.8–5.9 × 10^{-10} m^2 s^{-1}) dried between 50 and 80 °C and with an air velocity of 0.5 m s^{-1}. Thus, ten times higher effective diffusion coefficients were obtained at hot air drying, probably due to faster water diffusion inside the solid matrix at higher temperatures.

As expected, the higher the drying temperature, the higher the effective diffusion coefficient. The identified effective diffusion coefficient increased by 214% and 157% in the AIR and AIR + US experiments, respectively, when the temperature was increased from 5 to 15 °C. The effective diffusion coefficient increment was higher in AIR experiments than in AIR + US experiments as was also reported by Santacatalina *et al.*[4] (167% and 160% in AIR and

Table 1. Identified effective diffusion (D_e) and external mass transfer (h_m) coefficients together with the MRE and var, for each set of drying experiments without (AIR) and with 20.5 kW m^{-3} of acoustic assistance (AIR + US) at different temperatures

	AIR						AIR + US					
T (°C)	5		10		15		5		10		15	
$D_e \cdot 10^{11}$ (m^2 s^{-1})	1.37	±0.05	2.37	±0.11	4.30	±0.09	3.67	±0.11	6.52	±0.52	9.45	±0.48
$h_m \cdot 10^5$ (kg water m^{-2} s^{-1})	3.86	±0.11	6.40	±0.06	9.36	±0.17	12.76	±0.59	15.29	±0.42	19.01	±0.19
MRE (%)	3.2	±1.2	2.3	±0.9	2.2	±0.7	3.7	±0.5	2.7	±1.3	5.5	±0.6
var (%)	99.4	±0.1	99.8	±0.2	99.9	±0.1	99.6	±0.1	99.8	±0.1	99.4	±0.1

Average values ± standard deviations.

AIR + US experiments, respectively) and by Santacatalina *et al.*[22] (105% and 33% in AIR and AIR + US experiments, respectively) when increasing drying temperature from 0 to 10 °C. Thus, it seems that temperature had less influence in the AIR + US experiments than in the AIR experiments.

Moreover, AIR + US samples exhibited higher D_e coefficients compared with AIR samples, as a consequence of the acoustic assistance and its contribution to the reduction of the internal mass transfer resistance. As was pointed out in other researchers' work, the effective diffusion coefficient increment in AIR + US experiments is mainly linked to mechanical effects provoked in the material. Ultrasound generates a series of rapid and cyclic compressions and expansions of the material, which can be compared to a sponge being squeezed and released repeatedly, thus improving the water diffusion in the solid.[6] The D_ϵ coefficient increment was 168% at 5 °C; meanwhile, at 15 °C, it was lower (120%). Thus, the increment was higher at the lowest temperature. Similar behavior was reported by Santacatalina *et al.*[4] and by Santacatalina *et al.*[22] when ultrasound was applied in apple (148% and 136% of D_e increment at 0 and 10 °C, respectively) and eggplant (389% and 264% of D_e increment at 0 and 10 C, respectively) low-temperature drying. It seems that ultrasound mechanical effects were more effective at lower temperatures, as García-Pérez *et al.*[24] and Gamboa-Santos *et al.*[25] reported in hot air drying of carrot (at 30–70 °C) and strawberry (at 40–70 °C), respectively. These authors also observed an increment of the ultrasound influence on the effective diffusion coefficient as the temperature decreased. In fact, at the highest drying temperature (70 °C) no significant differences in D_e were observed between AIR and AIR + US experiments. It seems that ultrasound application introduces a given amount of energy into the solid thus affecting water mobility. As temperature increases, the mobility linked to temperature increases and the relative influence of ultrasound energy on the internal resistance diminishes.

The effective diffusion coefficient temperature dependency was satisfactorily correlated to an Arrhenius type equation (Eqn (8)) in the AIR and AIR + US experiments as was also done in low-temperature drying by Ozuna *et al.*[6] The Arrhenius linear correlation of D_e is represented in Fig. 3.

$$D_e = D_o \exp\left[-\frac{E_a}{R\,(T + 273.15)}\right] \qquad (8)$$

Correlation coefficients close to the unit were obtained in both cases (0.999 and 0.987 in AIR and AIR + US experiments, respectively). Thus, the adjustment to the Arrhenius-type equation was satisfactory. The D_o coefficient obtained was significantly lower (99% of decrease) in the AIR + US experiments (27 ± 2 m^2 s^{-1}) than in the AIR experiments (2636 ± 132 m^2 s^{-1}). Moreover, the estimated activation energy E_a for AIR and AIR + US experiments was of 77.0 ± 0.1 and 63.8 ± 0.2 kJ mol^{-1}. These figures were significantly different ($P < 0.05$) between them, the Ea for the AIR + US experiments being 17% lower than that for the AIR experiments. Similar results were also reported by Gamboa-Santos *et al.*[25] in strawberry drying (at 40–70 °C and 2 m s^{-1}) and by Do Nascimento *et al.*[26] in passion-fruit peel drying (at 40–70 °C and 1 m s^{-1}). According to these authors, the influence of the temperature on the D_e seemed to be more limited when ultrasound was applied. The application of ultrasound provided additional energy with which to facilitate the drying, the relative importance of which decreased as the drying temperature rose. The mechanical force given by the acoustic waves can create microscopic channels due to the 'sponge effect', which allows an easier inner water movement without significant overheating of the material being dried taking place.[27]

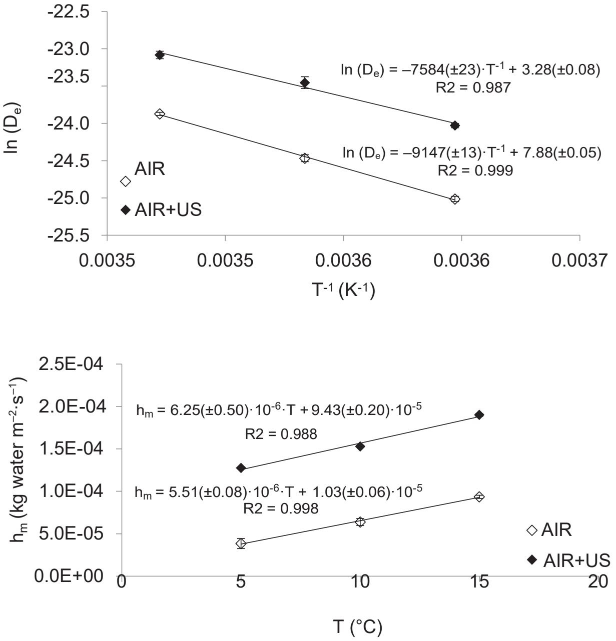

Figure 3. Influence of drying air temperature on the average effective diffusion and external mass transfer coefficients identified for kiwifruit drying without (AIR) and with 20.5 kW m^{-3} of acoustic assistance (AIR + US) at 5, 10, and 15 °C. Average values ± standard deviations.

As can be seen in Table 1, the external mass transfer coefficient h_m was affected by both the drying temperature and the acoustic assistance. The identified h_m figures ranged from 3.86 × 10^{-5} kg water m^{-2} s^{-1} at 5 °C (AIR) to 19.01 × 10^{-5} kg water m^{-2} s^{-1} at

15 °C (AIR + US). In low-temperature drying of apples at $2\ \mathrm{m\ s^{-1}}$ of air velocity and temperatures of 0 and 10 °C[4] and 10 °C[28], with and without ultrasound application, higher h_m figures were reported: $2.7–9.1 \times 10^{-4}$ and $4.3–10 \times 10^{-4}\ \mathrm{kg\ water\ m^{-2}\ s^{-1}}$, respectively, probably due to higher air velocity figures than in the present study ($1\ \mathrm{m\ s^{-1}}$). Thus, external resistance to moisture removal was significantly different at $2\ \mathrm{m\ s^{-1}}$ of air velocity than $1\ \mathrm{m\ s^{-1}}$ of air velocity.

The increase in the drying temperature from 5 to 15 °C caused an increase of h_m by 142% in AIR experiments and by 49% in AIR + US experiments. Thus, at higher temperatures, an increase in the external mass transfer coefficient was observed, being higher in AIR experiments than in AIR + US experiments. Santacatalina *et al.*[22] also observed a higher external mass transfer coefficient increase in AIR experiments (63%) than in AIR + US experiments (30%). The AIR experiments therefore presented a more important temperature effect than AIR + US experiments.

Acoustic assistance induced a decrease in the external resistance to the mass transfer due to the pressure variations at the solid/gas interfaces, and so it increased the surface moisture evaporation rate.[7] The sample vibrates in a microscale due to the ultrasound effects, which might also affect the external resistance. Thus, the acoustic assistance increased the external mass transfer coefficient h_m. Similarly to as was observed in the effective diffusion coefficient, this effect was more evident at 5 °C with an increase of 231% in this coefficient, while at 15 °C, the increment was 103%, probably due to the relative amount of thermal and acoustic energy. The effect of acoustic assistance on the external mass transfer coefficient was also studied during acoustically assisted ($20.5\ \mathrm{kW\ m^{-3}}$) low-temperature drying, at an air velocity of $1\ \mathrm{m\ s^{-1}}$, of eggplant by Santacatalina *et al.*,[22] respectively. In this study, higher increments of external mass transfer coefficient were also observed at 0 °C (383%) than at 10 °C (262%) when applying ultrasound.

The temperature dependency of the external mass transfer coefficient was linearly correlated (Eqn (9)) in AIR and AIR + US experiments. This is represented in Fig. 3.

$$h_m = h_k \cdot T + h_o \tag{9}$$

The adjustment to a linear type equation was suitable because in both cases correlation coefficients close to the unit were obtained (0.998 and 0.988 in AIR and AIR + US experiments, respectively).

The h_o coefficient significantly increased (by 811%) in AIR + US experiments ($9.43 \pm 0.20 \times 10^{-5}\ \mathrm{kg\ m^{-2}\ s^{-1}}$) compared with AIR experiments ($1.03 \pm 0.06 \times 10^{-5}\ \mathrm{kg\ m^{-2}\ s^{-1}}$). Moreover, in AIR + US experiments, a significantly higher (14%) h_k coefficient was obtained ($6.25 \pm 0.5 \times 10^{-6}\ \mathrm{kg\ m^{-2}\ s^{-1}\ {}^{\circ}C^{-1}}$) than in AIR experiments ($5.51 \pm 0.08 \times 10^{-6}\ \mathrm{kg\ m^{-2}\ s^{-1}\ {}^{\circ}C^{-1}}$). Thus, when ultrasound was applied, the surface moisture evaporation rate was enhanced and the external mass transfer coefficient increased. Not only was the external mass transfer coefficient in AIR + US experiments higher but it was also more affected by the temperature factor.

The drying curves were predicted by using the figures for D_e and h_m coefficients corresponding to Arrhenius (Eqn (8)) and linear (Eqn (9)) correlations, respectively. They are represented in Fig. 2 by continuing lines. The simulation was evaluated mathematically using the MRE (%) and var (%) figures, included in Table 1. As the MRE was lower than 6% and var was higher than 99% in all experiments, it could be concluded from Fig. 2 and Table 1 that the drying curves of kiwifruit dried at 5, 10 and 15 °C without and with acoustic assistance ($20.5\ \mathrm{kW\ m^{-3}}$) could be satisfactorily simulated by using the proposed model.

The use of the proposed model allowed us to evaluate the influence of ultrasound application on both the internal and external mass transfer resistance. From the figures obtained for the diffusion coefficient and the mass transfer coefficient, it could be concluded that the use of acoustic energy contributed to the acceleration of the drying process, not only decreasing the external resistance but also increasing the water mobility inside the food. The mechanical vibration produced by the ultrasound application affected both the internal resistance to the mass transport, by successive compressions and expansions of the material ('sponge effect'), and the external resistance to the mass transport due to the reduction of the boundary layer, which eased the vapor transfer rate from the solid surface to the drying air.[29] The sum of both effects led to an improvement in the water release during the drying process.

Bioactive compounds determinations

To determine the influence of the drying temperature and the ultrasound application during drying on the main bioactive compounds of kiwifruit, ascorbic acid content (AAC), vitamin E content (VEC), and total polyphenol content (TPC) were determined before and after drying.

In the fresh sample, the AAC and VEC were of 4.4 ± 0.2 mg L-ascorbic acid equivalent g d.m.$^{-1}$ and 0.098 ± 0.002 mg □-tocopherol equivalent g d.m^{-1}., respectively. Similar figures for AAC and VEC in fresh Zespri® Sweet Green Kiwifruit were reported by Sivakumaran *et al.*[30] (4.3 – 7.6 mg L-ascorbic acid equivalent g d.m.$^{-1}$ and 0.059 – 0.114 mg □-tocopherol equivalent g d.m.$^{-1}$, respectively). The TPC of fresh sample was of 10.0 ± 0.4 mg gallic acid equivalent/g d.m.$^{-1}$, which was in the range of the TPC proposed by Pal *et al.*[31] for fresh Hayward cultivar kiwifruit in three different fruit-harvesting months (7.9 – 11.3 mg gallic acid equivalent g d.m.$^{-1}$).

Figure 4 shows the AAC, VEC, and TPC losses (%) of kiwifruit samples after drying at 5, 10, and 15 °C without (AIR) and with ultrasound application (AIR + US), compared with the fresh sample. Drying without ultrasound application (AIR) at 5, 10, and 15 °C promoted AAC, VEC, and TPC losses of 14–26%, 28–54%, and 14–39%, respectively. Thus, as can be observed in Fig. 4, VEC losses were higher than AAC losses in dried kiwifruit at 5, 10 and 15 °C. As reported by Ball,[32] the main factors contributing to vitamin losses during processing are light, metal ions, and oxidation, due to air exposure that occurs during convective drying. Vitamin E is fat-soluble and is represented by four tocopherols and four tocotrienols.[32] Ascorbic acid is water-soluble and is a generic descriptor for all compounds exhibiting qualitatively the biological activity of ascorbic acid.[32] Thermal stability of vitamin E depends on processing time and conditions; meanwhile, ascorbic acid is stable on exposure to air and daylight at normal room temperature for long periods of time.[32] It seems, therefore, that ascorbic acid was more stable than vitamin E to air exposure during kiwifruit drying at 5, 10, and 15 °C.

No studies of quality changes in kiwifruit dried at low temperatures have been found in the literature, so we have referred instead to those regarding changes in the quality of kiwifruit as a consequence of drying with hot air. Higher AAC losses (49–88%) were observed after the convective drying of kiwifruit slices at 35 – 65 °C, compared to the fresh sample.[33] Nothing has been found in the literature about VEC changes after kiwifruit drying, either. Regarding TPC losses, similar figures (11–49%) were observed by Izli *et al.*[34] when kiwifruit slices were dried at 60, 70, and 80 °C and $1.5\ \mathrm{m\ s^{-1}}$.

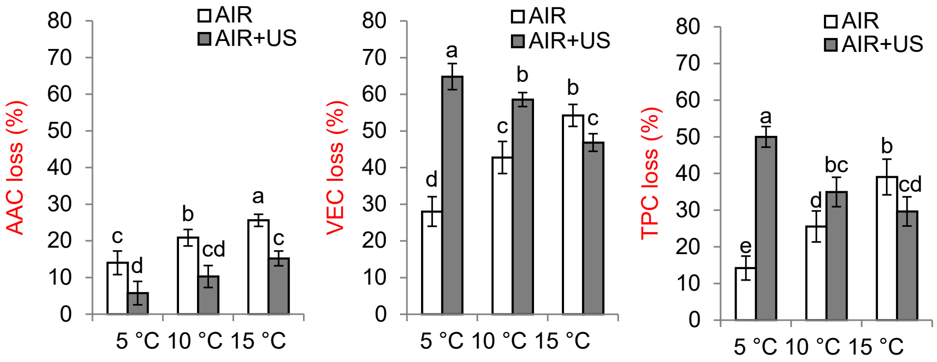

Figure 4. Kiwifruit losses (%) of ascorbic acid content (AAC), vitamin E content (VEC) and total polyphenol content (TPC) after drying at 5, 10 and 15 °C without (AIR, white bars) and with 20.5 $\mathrm{kW\,m^{-3}}$ of acoustic assistance (AIR + US, grey bars). Average values ± standard deviations. Means with different letters for AAC, VEC or TPC losses showed significant differences according to Tukey's test ($P < 0.05$).

Among all dried samples without ultrasound application (AIR), the highest losses in AAC, VEC, and TPC were observed in samples dried at 15 °C, probably due to higher bioactive compounds degradation at higher temperatures. Similar results were obtained by Santacatalina *et al.*:[4] TPC exhibited slight but significantly higher ($P<0.05$) losses in apple dried samples at 10 °C (40%) than at 0 °C (36%). In hot-air drying of kiwifruit, higher AAC losses were also observed with the increase of the drying temperature by Kaya *et al.*[33] at 35–65 °C (49 – 88% losses).

Samples dried at 5, 10, and 15 °C with ultrasound application (AIR + US) exhibited AAC, VEC and TPC losses of 6–15%, 47–65% and 30–50%, respectively, compared with the fresh sample. Thus, also in this case, the VEC losses were higher than AAC losses in dried samples with ultrasound application at 5, 10 and 15 °C. Furthermore, the TPC losses were also higher than the AAC losses in these samples.

In the case of samples dried at 5 and 10 °C with ultrasound application (AIR + US), the VEC and the TPC losses were significantly higher ($P<0.05$) than the corresponding dried samples without ultrasound application (AIR). This behavior was also observed by Santacatalina *et al.*[4] in TPC when drying apple cubes at temperatures of 0, 5, and 10 °C with and without ultrasound application (at 20.5 $\mathrm{kW\,m^{-3}}$). According to this study, this greater degradation could be linked to the structural damage of cells brought about by ultrasound. The mechanical stress linked to ultrasonic wave propagation could therefore aid the release of oxidative enzymes and intra-cellular compounds into the solvent, contributing to the degradation of polyphenol in a similar way to freezing. In hot-air drying, high degradation of VEC[16,35] and TPC[26,36] were also reported by different studies when ultrasound was applied.

However, samples dried at 15 °C with ultrasound application (AIR + US) exhibited significantly lower ($P<0.05$) losses of AAC (as well as samples dried at 5 and 10 °C), VEC, and TPC, than the corresponding dried samples without ultrasound application (AIR). It seems that ultrasound application led to a better retention of TPC in these cases, probably due to the shortening of the drying time, which reduces the thermal exposure of the samples and, consequently, the bioactive compound degradation. According to Moreno *et al.*,[37] the application of ultrasound can activate a response mechanism in the tissue that induces the formation of new phenolic compounds, not only through the combination of existing compounds but also via the activation of secondary metabolic pathways. Furthermore, the fact that the ultrasonic treatment produced a possible inactivation of oxidative enzymes must also be considered. Similar effects in AAC,[38] VEC[16,35] and TPC[26] were also reported in the bibliography of hot-air drying when ultrasound was applied.

Antioxidant activity

Antioxidant activity (AA) in kiwifruit samples was determined using the FRAP, CUPRAC, and ABTS methods to evaluate the effects of drying temperature and ultrasound application. In each AA method used, the measurement is based on a single electron transfer, but the antioxidants present in the medium may be hydrophilic or lipophilic in nature and this will aid the reaction to a greater or lesser extent. It should be noted that, as each method is based on a different chemical system and / or reaction, the AA figures clearly varied for each sample extract, depending on the method.[39] However, the results of AA according to FRAP, CUPRAC, and ABTS correlated highly with each other, the correlation coefficient being higher than 0.89.

The AA of the fresh sample, according to the FRAP, CUPRAC, and ABTS methods, was 42 ± 3, 26 ± 1 and 34 ± 2 mg Trolox equivalent $\mathrm{g\ d.m.^{-1}}$, respectively. Similar values of AA, according to the FRAP method, were reported by Pal *et al.*[31] in fresh kiwifruit of the Hayward cultivar at three different fruit-harvesting months (38–50 mg Trolox equivalent $\mathrm{g\ d.m.^{-1}}$). Similar values of AA, according to the CUPRAC and ABTS methods, were reported by Leontowicz *et al.*[40] in kiwifruit (22 ± 3 and 41 ± 4 mg Trolox equivalent $\mathrm{g\ d.m.^{-1}}$, respectively).

Loss (%) (Eqn (6)) of the AA, according to the FRAP, CUPRAC and ABTS methods, in the kiwifruit samples after drying at 5, 10, and 15 °C without (AIR) and with ultrasound application (AIR + US), compared with the fresh sample, are shown in Fig. 5. In general, when samples were dried without ultrasound assistance (AIR), the AA losses were higher after drying at 15 °C than at 5 °C, as was also observed in bioactive compounds losses, which might be related to higher bioactive compounds degradation at higher temperatures. Santacatalina *et al.*[4] also reported significantly higher ($P<0.05$) AA Loss (%) according to the CUPRAC method in apple-dried samples at 10 °C (21%) than at 0 °C (18%).

Antioxidant activity Loss (%) were significantly higher ($P < 0.05$) in samples dried at 5 and 10 °C with ultrasound application (AIR + US) than the corresponding dried samples without ultrasound application (AIR). As was mentioned above, this greater degradation could be linked to the structural damage to cells brought about by ultrasound. Santacatalina *et al.*[4] also reported lower AA according to ABTS and CUPRAC methods when drying apple cubes at low temperatures of 0, 5 and 10 °C without and with ultrasound

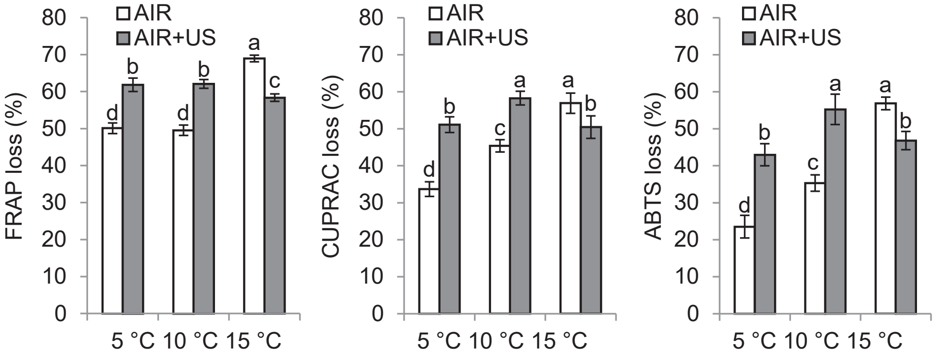

Figure 5. Kiwifruit losses (%) of antioxidant activity (AA), according to FRAP, CUPRAC and ABTS methods, after drying at 5, 10 and 15 °C without (AIR, white bars) and with 20.5 kW m^{-3} of acoustic assistance (AIR + US, grey bars). Average values ± standard deviations. Means with different letters for AA losses, according to FRAP, CUPRAC or ABTS methods, showed significant differences according to Tukey's test ($P < 0.05$).

application (20.5 kW m^{-3}). In hot air drying, Do Nascimento *et al.*[26] also observed lower AA (according to FRAP method) in dried passion-fruit peel (at 60 and 70 °C and 1 m s^{-1}) with ultrasound application (30.8 kW m^{-3}) than in corresponding samples without ultrasound application.

However, significantly lower ($P < 0.05$) AA losses were observed in samples dried at 15 °C with ultrasound application (AIR + US) than the corresponding dried samples without ultrasound application (AIR). It seems that ultrasound application leads to a better retention of AA in these cases, as was mentioned with regard to bioactive compounds. This better retention of AA was probably due to the shortening of the drying time, which reduces the thermal exposure of the samples and, consequently, the antioxidant activity degradation; or it might be related to a response mechanism of the tissue activated by ultrasound as reported by Moreno *et al.*[37] These results therefore correlated better with the retention of bioactive compounds mentioned above when ultrasound was applied at 15 °C. In hot air, significantly higher ($P < 0.05$) AA (FRAP method) was observed in passion fruit peel dried at 40 and 50 °C and 1 m s^{-1} with ultrasound application (at 30.8 kW m^{-3}) than without ultrasound application.[26]

CONCLUSIONS

The effects of acoustic assistance on a low-temperature drying process of kiwifruit have been studied. The intensification of the drying process was achieved by applying power ultrasound. Reductions of 55 – 65% in drying time were observed. A diffusion model considering both internal and external resistance satisfactorily simulated the drying kinetics (MRE = 3.3 ± 1.3%, var = 99.7 ± 0.2%). The acoustic energy caused an increment in the effective diffusion coefficient D_e and the external mass transfer coefficient h_m by up to 120 – 175% and 103 – 231%, respectively, which indicates an improvement in the drying rate caused by the application of power ultrasound. Significantly lower ($P < 0.05$) bioactive compound content (AAC, VEC and TPC, 14–54% of loss) and AA (23–69% of loss) were observed in all dried kiwifruit samples compared with the fresh sample. Ultrasound applied during drying at 5 and 10 °C promoted higher ($P < 0.05$) biocompound losses (VEC and TPC) and AA (35–65% and 43 – 62%, respectively) than those in corresponding samples without ultrasound application (14–43% and 23–50%, respectively). However, when drying was carried out at 15 °C, ultrasound contributed to the preservation of these biocompounds and antioxidant activity (30–47% and 47–58%, respectively) better ($P < 0.05$) than in samples obtained without using ultrasound (39–54% and 57–69%, respectively). Thus, the use of ultrasound when drying at 15 °C allowed the shortest drying time and better maintained biocompound content and antioxidant activity.

NOMENCLATURE

D_e	Effective water diffusion coefficient (m^2 s^{-1})
D_o	Parameter in the effective diffusivity model (m^2 s^{-1})
E_a	Activation energy (kJ mol^{-1})
h_m	External mass transfer coefficient (kg water m^{-2} s^{-1})
L	Length (m)
n	Number of experimental data
MRE	Mean relative error (%)
R	Universal gas constant (J mol^{-1} K^{-1})
S_x	Standard deviation (sample)
S_{yx}	Standard deviation (estimation)
T	Temperature (°C)
t	Time (h)
var	Percentage of explained variance (%)
W	Moisture content (kg kg d.m.$^{-1}$)
x, y, z	Spatial coordinates (m)
□$_{dm}$	Dry matter density (kg d.m. m^{-3})
□	Relative humidity
Subscripts	
0	initial
∞	drying air
cal	calculated
e	equilibrium at the surface
Abbreviations	
AIR	Convective air experiments
AIR + US	Convective air experiments assisted by ultrasound
MRE	Mean relative error
AAC	Ascorbic acid content
VEC	Vitamin E content
TPC	Total polyphenol content
AA	Antioxidant activity
var	Percentage of explained variance

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