Osmotic dehydration and freezing as a suitable pretreatment in the process of vacuum drying kiwiberry: drying kinetics and microstructural changes**

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A b s t r a c t. This study investigated the effects of osmotic dehydration and freezing on the kinetics and microstructure of vacuum-dried kiwiberry. Both fresh and previously frozen fruit were dehydrated in sucrose, maltitol and xylitol. Freezing and osmotic dehydration were selected as possible ways to improve the drying kinetics and positively influence the taste of the fruit. This experiment focused on the analysis of microstructural changes induced by applied processing methods using the X-ray microtomography technique. The results showed that the fruit pretreated in sucrose suffered the least structural damage as expressed by the largest condensation of small pores and thin cell walls. Freezing and xylitol resulted in the accumulation of larger pores and thicker walls. The most rapid drying time of 678-688 min was observed for unfrozen samples, dehydrated in sucrose and maltitol. Freezing slowed down the drying process, by 60-100 min, in comparison to the unfrozen samples. The applied mathematical models proved useful in predicting the kinetics of the drying process. The equation proposed by Midilli *et al.* provided the best fit for predicting the kinetics of the process.

K e y w o r d s: *Actinidia arguta*, X-ray microtomography, mathematical modelling, drying kinetics

INTRODUCTION

The limitations of conventional drying methods are driving the search for a better way of improving mass transfer, reducing energy consumption as well as retaining maximum product quality (Szadzińska *et al.*, 2018). One possible solution may be a combination of different treatment and pretreatment techniques in one process. Pretreatment is a process aimed at changing selected parameters of the final product and it usually has a positive effect on its quality, which may be determined by measuring its organoleptic and physicochemical parameters (Cichowska *et al.*, 2018). The type and parameters of the pretreatment should be selected depending on the raw material used or the expected result. Assessment of the changes induced by a series of technological processes and in particular, the effects of pretreatment may be difficult or require sophisticated equipment. For example, the internal microstructure of dried food products can be analysed using the X-ray microtomography technique (X-ray micro-CT). X-ray micro-CT has been used to nondestructively observe the structure of various porous dried materials, such as freeze-dried carrots (Voda *et al.*, 2012), chokeberries (Calín-Sánchez *et al.*, 2015) and raspberries (Szadzińska *et al.*, 2018). Kiwiberry (*Actinidia arguta*) is a plant that produces fruit which contains numerous bioactive substances and the preservation of this fruit during the drying process is a challenge for currently available food technology (Latocha, 2017). Vacuum drying is a widely used method of drying products possessing such properties. The use of osmotic dehydration preceding the drying process can in many cases improve the kinetics of the process, and also, osmotic substances positively affect the retention of bioactive components (Kowalska *et al.*, 2018). Additionally, it affects the cell structure of fruit, by

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modifying the cell walls, splitting the middle lamella, as well as causing the lysis of membranes and tissue shrinkage (Joardder *et al.*, 2017). The other possible pretreatment method is freezing which allows for the more effective processing of fruit during their harvest and can also improve the taste of the final product (Duffy *et al.*, 2016). However, during freezing, the growth of ice crystals compresses and ruptures cells, which causes cell damage (Joardder *et al.*, 2017). That is why freezing is not only destructive to the structure of the fruit but also influences its bioactive components despite the use of rapid methods of lowering temperature (Phothiset and Charoenrein, 2014). To the best of the authors' knowledge, only the impact of drying methods on the parameters concerning the physicochemical properties of dried kiwifruit has been investigated. To date, no studies have been conducted which describe the impact of pretreatment on the microstructure of these fruit. This study aimed to experimentally determine the effect of freezing and osmotic dehydration on the structural properties of kiwiberry. The secondary goal was to determine the feasibility of both preprocessing methods on the drying kinetics of the berries.

MATERIAL AND METHODS

The study was conducted using *Actinidia arguta* 'Weiki'. The plants were grown in a commercial orchard under the supervision of scientists from the Faculty of Environmental Protection of the Warsaw University of Life Sciences. The fruit were collected at the maturity phase and were stored at 2°C in darkness. Before each experiment, the fruit were removed from cold storage, and after reaching room temperature (21°C), they were washed with tap water and cut into halves.

The fruit halves were laid out on perforated aluminium trays and frozen at -40°C in an IRINOX SpA (Italy) shock freezer. The test material was then transferred to sealed packages of barrier film and stored in a laboratory freezer at -22°C. The frozen fruit was thawed and used after reaching room temperature (21°C).

Dehydration was performed using 60% w/w solutions of sucrose, xylitol, and maltitol. The concentrations of these solutions were determined during preliminary experiments, which were based on the results presented in the literature (Bialik *et al.*, 2018). All of the samples were dehydrated as a way to lessen the negative aftertaste of the dried fruit. OD was performed in a thermostatically controlled bath of hot water for 180 min, with a stirring speed of 1 Hz and a water temperature of 40°C (± 1°C). Depending on the experiment, 22 g (± 2.5 g) of fresh or previously frozen kiwi halves were placed in 250 ml beakers containing 80 ml osmotic solutions. After reaching the setup time, the material was removed from the bath, washed with 200 g of distilled water and gently placed on filter paper to remove the liquid residue.

The drying procedure was carried out in a vacuum laboratory dryer Christ Gamma 1-16 LSC (Germany) at 55°C under a pressure of 4 kPa. The fruit halves were placed on trays and dispersed to produce an area density of 3.95 kg m^{-2} with the exposed flesh facing up. The process was carried out to achieve a moisture ratio (MR) of 0.02. The weight of the material was recorded every 2 minutes, with an accuracy of 0.01 g. The dry matter content of the raw and dried fruit was measured following AOAC 920.1511 (Latimer and AOAC International, 2016). The drying process was carried out in duplicate, a detailed description of the methods used is presented in Table 1.

Table 1. A description of pretreatment methods used in this experiment

Sample code	Pretreatment description
M	Dehydration in maltitol
S	Dehydration in sucrose
X	Dehydration in xylitol
FR+M	Pre-frozen fruits dehydrated in maltitol
FR+S	Pre-frozen fruits dehydrated in sucrose
FR+X	Pre-frozen fruits dehydrated in xylitol

The SkyScan 1272 system (Bruker microCT, Kontich, Belgium) was used for the X-ray micro-CT measurements. It had a pixel resolution of 25.0 µm and was operated using a 40 keV source voltage, and a 193 µA current. A stack of approximately 1500 flat projection images (1008 × 1008 pixels) was obtained after a 180° rotation with 0.4° steps, which averaged 4 frames for each step. Each sample was scanned for 35 min. Three randomly selected individual samples of every variant were scanned. The projection images were loaded in NRecon 1.6.3.2 software (Bruker, Kontich, Belgium) to reconstruct virtual cross-sections of the sample. The images were corrected for rings and beam hardening, and common artifacts in the X-ray CT images.

The 3D visualization of individual samples was performed using CTvox software (Bruker, Kontich, Belgium). Reconstructed datasets were loaded in CTAnn software (Bruker, Kontich, Belgium) and binarized in a threshold range of 50-255. Voxels with a grey value higher or lower than that threshold value were considered to be sample tissue or background/pores, respectively. The region of interest was adjusted by custom processing using a shrink-wrap internal plugin. In order to calculate the total porosity and thickness of the walls, a 3D analysis was performed. Afterwards, reversed thresholding was performed using a bitwise operations plug-in, which was followed by a 3D analysis that resulted in the determination of the thickness of the pores.

Table 2. Mathematical models used in the experiment

No.	Name	Model Equation
1	Logarithmic	$MR = a\exp(-k\tau) + b$ (Sarimeseli, 2011)
2	Logistic	$MR = \dfrac{b}{(1 + a\exp(k\,\tau))}$ (Soysal et al., 2006)
3	Midilli et al.	$MR = a\exp(-k\tau^c) + b\tau$ (Midilli et al., 2002)
4	Fick's second law of diffusion (simplified)	$MR = \dfrac{8}{\pi^2}\exp\left(-\dfrac{\pi\,D_{eff}\,\tau}{4\,L^2}\right)$ (Ramaswamy and Nsonzi, 1998)
5	Two-factor	$MR = a\exp(-k\tau) + b\exp(-k_i\tau)$ (Arslan et al., 2010)
6	Wang and Singh	$MR = 1 + a\tau + b\tau^2$ (Wang and Singh, 1978)

k, k_i drying rates (min^{-1}); a, b – model parameters, τ – time (s); D_{eff} – effective water diffusion coefficient (m^2 s^{-1}); L – half of the material thickness (m).

Mathematical modelling provides a means for better understanding and describing drying kinetics. Table 2 lists the models used for the evaluation of the drying process. The drying curves were plotted as a function of time and the dimensionless moisture ratio (MR):

$$MR = \frac{u_\tau}{u_0}, \qquad (1)$$

where: u_0 is the initial moisture content (g H$_2$O g^{-1} d.m.), and u_τ is the moisture content at a specific time (g H$_2$O g^{-1} d.m.).

Regression analysis was performed using Table Curve 2D v5.01 software (SYSTAT Software, Inc., Chicago, IL, USA). In order to evaluate the model fitting, the reduced chi-squared statistic (χ^2), and the root mean square error (RMSE) were used. χ^2 and RMSE were calculated as follows:

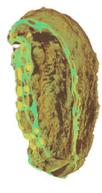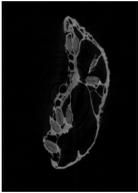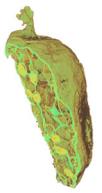$$RMSE = \sqrt{\frac{\sum_{i=1}^{N}\left(MR_{i,p} - MR_{i,e}\right)^2}{N}}, \qquad (2)$$

$$\chi^2 = \frac{\sum_{i=1}^{N}\left(MR_{i,p} - MR_{i,e}\right)^2}{N-n}, \qquad (3)$$

where: $MR_{i,p}$ is the predicted dimensionless moisture ratio, $MR_{i,e}$ is the experimental dimensionless moisture ratio, N is the number of observations and n is the number of constan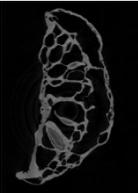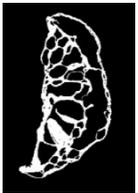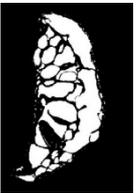ts in the model equation. The best-fitting model for the experimental data is characterized by χ^2 and RMSE values nearing 0. Additionally, for a better description of the process, the effective water diffusion coefficient based on the simplified equation for Fick's second law of diffusion was calculated.

RESULTS AND DISCUSSION

The porosity and the pore volume of the samples were determined from X-ray images, as shown in Table 3 and Figs 1-3. Various pretreatments have a different impact on the porosity of the obtained products (Table 3). The highest porosity value of about 69% was observed for the frozen and FR+S samples (Fig. 2b). In the case of M (Fig. 1a), the porosity differs by only 5% concerning the FR+S drying process (Fig. 2b). A decrease in porosity with an average value of 55-58% was measured for S, X, FR+M (Table 3). The most compacted structures were presented by the kiwiberry samples prefrozen and dehydrated in xylitol (Fig. 3b). The S samples, were characterized by the highest volume (18%) of thin walls (<0.2 mm), and pores < 2.4 mm (Fig. 2a). In comparison, FR+S samples were described by a 16% share of <0.2 mm walls but the most common pores were larger than 2.0 mm (Fig. 2b). The polar opposite was the case with the X and FR+X samples where about 10% of the walls were 0.3 mm or thinner (Fig. 3a, b). The largest pores were observed for FR+X (Fig. 3b) where over 10% of their volume was above 3 mm. Joardder et al. (2016), reported that drying methods and conditions affect the porosity of the final product. The higher porosity of the final product may indicate that less structural damage has occurred in the product during drying (Szadzińska et al., 2018). Osmotic dehydration affects cell walls, splits lamella and causes the lysis of membranes and tissue shrinkage (Ayala-Aponte et al., 2014). The observed differences in total porosity between the osmotic agents are partially a result of the differences in their osmotic pressure and effectiveness of dehydration (Bialik et al., 2018). In the dehydration experiment, performed by Bialik et al. (2018) researchers

Table 3. 3D rendered, raw, and binarized images as well as the porosity of dried kiwiberry

Sample code	Raw image	3D visualization	Cross section raw image	Binarization for porosity and wall thickness analysis	Reversed binarization for pore thickness analysis	Total porosity (%)
M						$62.5^{ab} \pm 4.8$
S						$57.8^{ab} \pm 3.6$
X						$55.0^{a} \pm 3.6$
FR+M						$57.9^{ab} \pm 1.5$
FR+S						$68.0^{b} \pm 6.3$
FR + X						$52.1^{a} \pm 6.3$
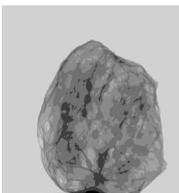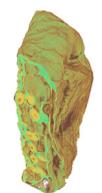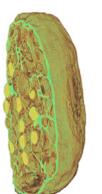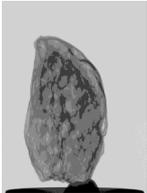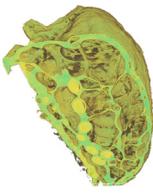
Means sharing the same superscript are not significantly different from each other (Tukey's HSD, $p < 0.05$).
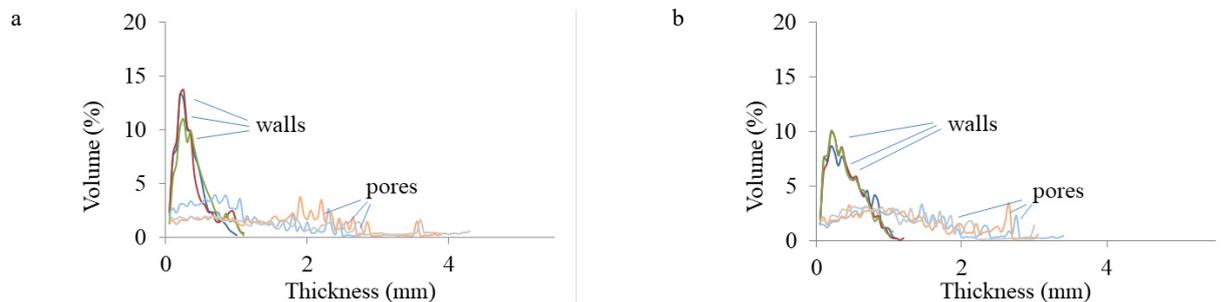
Fig. 1. Percentage of walls and cells of specific thickness in the sample dehydrated (a), pre-frozen and dehydrated (b) in maltitol.
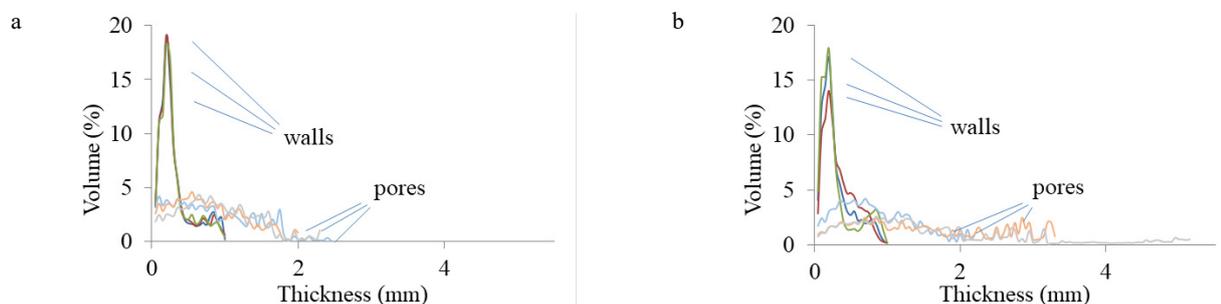

Fig. 2. Percentage of walls and cells of specific thickness in the sample dehydrated (a), pre-frozen and dehydrated (b) in sucrose.

Fig. 3. Percentage of walls and cells of specific thickness in the sample dehydrated (a), pre-frozen and dehydrated (b) in xylitol.

found no differences in the dehydration effectiveness of kiwiberry using sucrose and maltitol. In this experiment, different porosity values were found for the dried samples pretreated in both of these osmotic agents. This suggests that the utilization of these osmotic agents has different effects on the pretreatment and drying phases. It may be anticipated however, that the glass transition temperature of these osmotic agents may play an important role. The glass transition temperatures (T_g) of maltitol, xylitol and sucrose were 39, 29 and 62-70°C, respectively (Hartel *et al.*, 2011). As mentioned above, the highest degree of porosity was determined for samples subjected to freezing followed by osmotic dehydration that was carried out in sucrose (FR+S) whereas the porosity of all of the other variants, that were not prefrozen before osmotic dehydration, varied between 55.0 and 62.5%. Freezing ruptured the cellular structure

of the fruit thereby allowing an increased incorporation of solids (solid gain). Considering the glass transition temperature of the utilized osmotic agents and the drying temperature (55°C) it may be expected that the structure impregnated by maltitol and xylitol collapsed whereas the fruit impregnated by sucrose maintained its structure since the process was performed below T_g for sucrose. However, the proposed explanation is only a proposition and requires further investigation.

Vallespir *et al.* (2019) performed an experiment combining different freezing temperatures and convective drying using beetroot, apple, and eggplant. He concluded that the final microstructure depends on the initial microstructure of the product and the freezing rate. In the current experiment, the product used has a jelly structure, which is probably damaged during the freezing phase. This phenomenon is
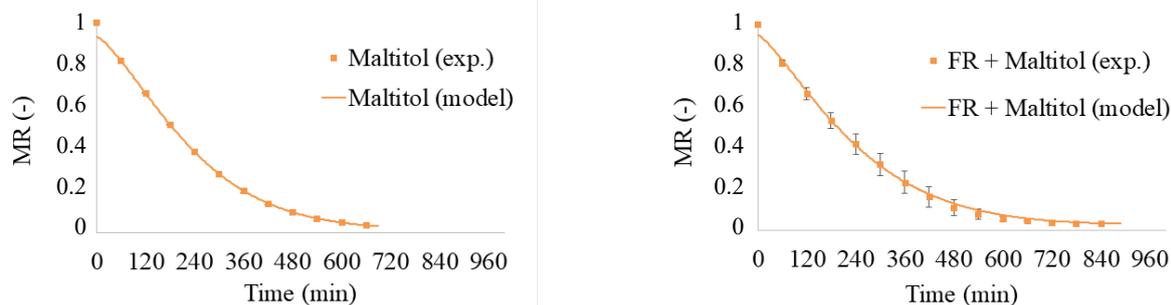
Fig. 4. Drying curves from the samples dehydrated (a), pre-frozen and dehydrated (b) in maltitol.
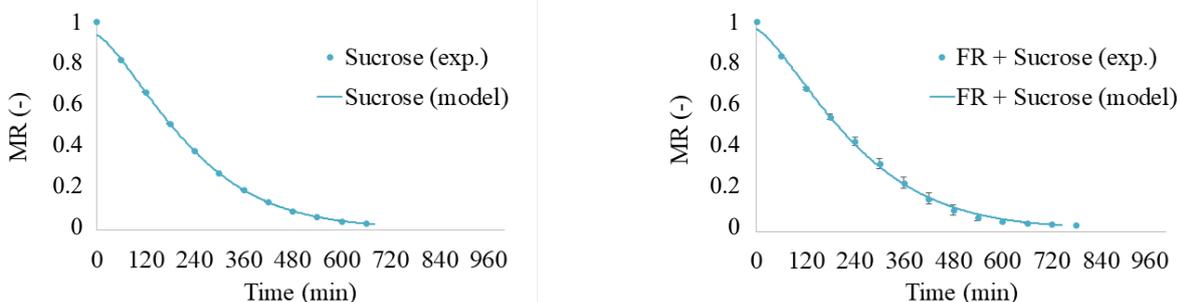

Fig. 5. Drying curves from the samples dehydrated (a), pre-frozen and dehydrated (b) in sucrose.
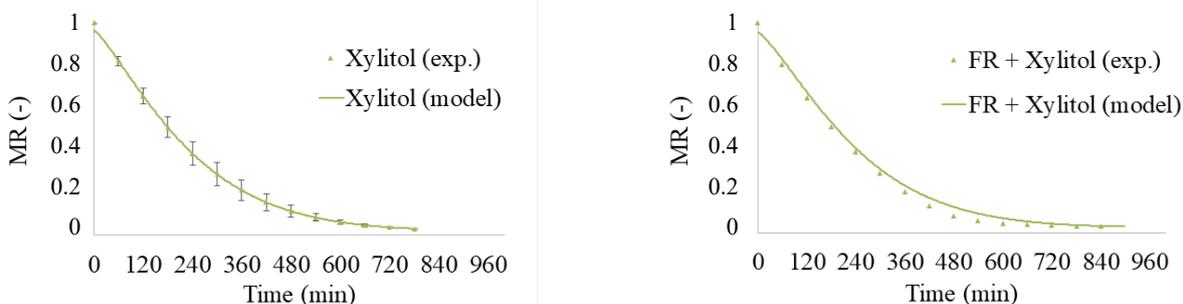

Fig. 6. Drying curves from the samples dehydrated (a), pre-frozen and dehydrated (b) in xylitol.

related to the size, shape, and distribution of the ice crystals (Li *et al.*, 2018). Freezing pretreatment influenced the pore volume and porosity, this was caused by ice crystal formation. The formation of ice crystals reduced the number of small-sized walls and pores, this effect was particularly pronounced in FR+M and FR+S samples.

Drying curves established during the experiment and from the best fitting model are presented in Figs 4-6. Experimental results indicate that the fastest drying kinetics were observed for the samples, which were predehydrated in osmotic solutions only. MR = 0.02 for the control sample (S) was reached after about 713 min (Table 4). Kiwiberries dehydrated in maltitol (M) showed more favourable kinetics and were dried after 675 ± 15 min. Freezing resulted in an adverse effect on the drying speed, especially during its second phase. FR+S samples reached the desired MR value after about 872 min. Somewhat better results from the prefrozen samples were obtained for FR+M and FR+X

with drying times averaging 832 – 842 min. The previously described changes induced by osmotic dehydration lowered the initial moisture content, and reduce the ending drying rate (Nahimana *et al.*, 2011). The slower kinetics of the prefrozen samples may be connected to the structural damage caused by the nucleation of the ice crystals (Li *et al.*, 2018). Statistical analysis (Table 2) has shown the significant influence of dehydration or freezing on the drying kinetics. There was no significant difference when both treatments were applied.

In an experiment performed by Dermesonlouoglou *et al.* (2019) the researchers explored the usage of different osmotic reagents (glycerol, maltodextrin, trehalose, sodium chloride, calcium chloride, and citrus extract) as a method used to obtain novel food from tomatoes and cucumber slices. They combined osmotic dehydration with conventional drying (at 55°C), which resulted in the achievement of some very promising results from the cucumbers. Depending on the osmotic agent used and the temperature

Ta b l e 4. Additional drying parameters of the tested samples

Sample code	Moisture content (g H₂O g⁻¹ dry mass)		Experimental drying time (min)	Best fitting model	RMSE	χ^2	CRV (%)
	Initial	Final					
M	$1.998^c \pm 0.047$	$0.057^{bc} \pm 0.001$	$675^c \pm 15$		0.00896	0.00009	5.35
S	$1.947^{bc} \pm 0.174$	$0.063^c \pm 0.002$	$713^{cd} \pm 33$		0.00879	0.00008	6.11
X	$1.709^{abc} \pm 0.171$	$0.050^{ab} \pm 0.003$	$772^{ad} \pm 36$	Midilli *et al.*	0.00636	0.00004	5.60
FR + M	$1.650^{ab} \pm 0.055$	$0.047^{ab} \pm 0.002$	$842^{ab} \pm 20$		0.00818	0.00007	5.77
FR + S	$1.306^a \pm 0.096$	$0.040^a \pm 0.004$	$834^{ab} \pm 24$		0.00874	0.00007	6.00
FR + X	$1.574^a \pm 0.001$	$0.046^a \pm 0.009$	$872^b \pm 30$		0.00809	0.00006	5.82

Means sharing the same superscript are not significantly different from each other (Tukey's HSD, $p < 0.05$).

applied, a reduction in the total time required for drying of between 20 and 40% was observed. Similar results were obtained from the experiment performed by Teles *et al.* (2006) where osmotic pretreatment was preceded by the air drying of melon cubes. The lowest drying times were established when the samples were dehydrated for between 175 and 220 min at 65°C. In this experiment, the temperature applied was lower due to the delicate structure of the kiwiberry.

The mathematical models applied allowed us to obtain a very good fit for the kinetics of drying. In all cases, the best model fit parameters for experimental data, as expressed by RMSE, χ^2, and CRV, were obtained by the Midilli *et al.* (2002) model (Table 2). The overall effectiveness of this model was also observed by Soysal *et al.* (2006), and Wiktor *et al.* (2016). Although closely matching parameters are maintained, it is worth focusing on the significant process time discrepancy for modelling the FR + S samples. In this case, the difference between the average time required to achieve the desired MR (equal to 0.02) and the value determined by the best-suited model was almost 90 min.

CONCLUSIONS

1. The results obtained from this experiment suggest that where drying kinetics is concerned the optimal pretreatment for vacuum drying is osmotic dehydration in sucrose or maltitol.

2. In terms of the preservation of the structure by means of porosity, wall, and cell thickness, the best results were obtained by sucrose pretreatment.

3. The freezing of kiwiberry significantly influenced the drying kinetics by slowing it down by almost 15%, this is due to the significant destruction of the cell structure which was observed and measured using X-ray micro-CT.

4. The mathematical modelling of drying has proven its usefulness in the prediction of drying kinetics, with the best fit obtained using the Midilli *et al.* model.

Conflict of interest. The Authors do not declare any conflict of interest.

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