

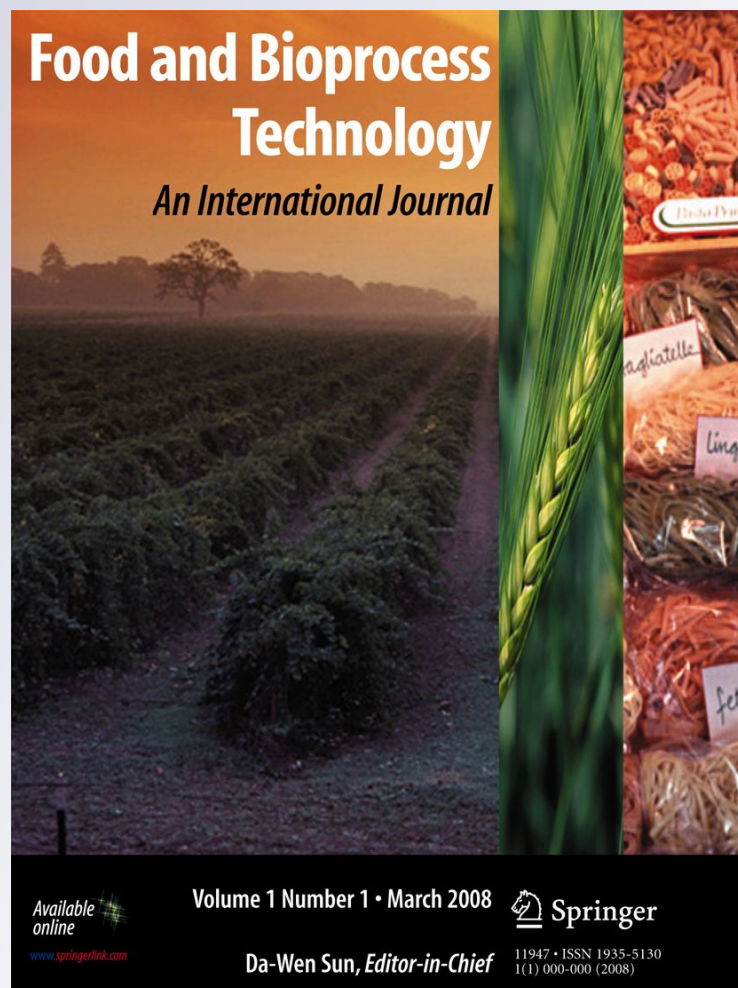
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Abstract The effects of different pretreatments on the convective drying kinetics of blueberries (var. O'Neil) at 70 °C were investigated. The sodium hydroxide, enzymatic, microwaves, and high hydrostatic pressure pretreatments were applied and compared with control samples (non-pretreated). In order to simulate experimental drying data, the following mathematical models were selected: logarithmic, two terms, modified Henderson–Pabis, Midilli–Kucuk, and Weibull. Fick's second law of diffusion was applied to determine the water diffusion coefficient for all the treatments. All pretreatments decreased significantly the drying time of the control samples. High hydrostatic pressures together with microwave pretreatments presented lower

drying times than non-pretreated samples. Moreover, based on statistical test results, Weibull and modified Henderson–Pabis models presented the best fit for the experimental drying curves. Thus, both models can be satisfactorily applied to estimate the drying time of blueberries as well as to evaluate the effects of different pretreatments on fruit drying rates.

Keywords High hydrostatic pressure · Blueberry · Pretreatments · Drying kinetics · Mathematical modeling

Introduction

Blueberry, which belongs to the genus *Vaccinium*, family Ericaceae, matures between December and late January in Chile, depending on the cultivation zone and extending over 30–40 days (Vega-Gálvez et al. 2009). They contain many compounds with strong antioxidant activities, such as phenolic compounds, anthocyanin pigments, flavonoids, as well as vitamin C (López et al. 2010), which play important roles in human nutrition due to free radical scavenging activities, resulting in a source protection against diseases such as memory loss, cancer, heart disease, and aging, among others (Skrede et al. 2000; Lee and Wrolstad 2004; Giovanelli and Buratti 2009; Shi et al. 2008). In recent years, the consumption of these fruits has increased due to its bioactive compounds. Therefore, it is fundamental for this fruit processing industry to apply new technologies to improve the post-harvest fruit shelf life in order to maintain the original quality attributes and to minimize operation costs (Moraga et al. 2006).

Convective hot air drying is one of the most common methods used for preserving foods and extending their shelf life by reducing their moisture content (Vega-Gálvez et al.

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2011). Moreover, the use of this technology may contribute to the increase of local development, consumption, and productivity; to obtain high-quality export products; as well as an integral use of the material in epochal production (Vega-Gálvez et al. 2009). It is known that the dehydration process can lead to non-uniformity, slow drying rates, and changes in the quality of the final products. Nevertheless, these disadvantages can be reduced using a combination of convective drying with different pretreatments.

Pretreatments are carried out to improve food quality and to reduce process times (Fito and Chiralt 2003; Aguilera et al. 2003). When pretreatments are used, the final products differ from those without pretreatment because modifications in the cell membranes occurred, which play a key role during further processing (Pérez and Schmalko 2009). The changes in the state of the cell membranes may vary between partial and total permeabilization depending on the specific pretreatment (Taiwo et al. 2001). Within common pretreatments, water blanching, application of enzymatic and osmotic solutions, and microwaves can be mentioned (Al-Khuseibi et al. 2005).

In particular, high hydrostatic pressure technology is an emerging non-thermal treatment with a great potential for food processing (McInerney et al. 2007). Moreover, the application of a high hydrostatic pressure pretreatment can enhance the drying rates and improve the overall quality of foods (Rastogi and Niranjan 1998; Rastogi et al. 2000a; McInerney et al. 2007; Yücel et al. 2010; Katsaros et al. 2009; Keenan et al. 2010; Briones-Labarca et al. 2010). Among other pretreatments, the application of microwaves prior to hot air drying on fruits and vegetables has been known to improve product quality such as enhanced aroma, faster rehydration, considerable savings in energy, and shorter drying times (Soysal et al. 2009). Also, the applications of enzymes are used in the food industry to improve quality. The main commercial enzyme preparations usually contain hydrolytic enzymes such as pectinases, cellulases, and hemicellulases (Ortega et al. 2004).

Simulation of the drying process through mathematical modeling is an important tool for improving these processes and minimizing operative problems such as product damage and excessive consumption of energy, among others (Azzouz et al. 2002). The empirical equations frequently used to model drying kinetics are Newton, Henderson–Pabis, Page, modified Page, Wang–Singh, logarithmic, two terms, Midilli–Kucuk, and modified Henderosn–Pabis (Akpınar 2006; Doymaz 2007). Most of these equations have been derived from Fick's second law of diffusion for different geometries (Fito and Chiralt 2003).

The aim of this work was to experimentally study and mathematically model the effect of different pretreatments

such as sodium hydroxide, enzymatic, microwaves, and high hydrostatic pressure on the drying kinetics of blueberries (var. O'Neil) during hot air drying at 70 °C.

Materials and Methods

Raw Material and Drying Conditions

Blueberries (var. O'Neil) were cultivated and purchased in the province of Salamanca, Chile. The fresh samples (2.50 kg) were selected to provide a homogenous group based on their date of harvest, color, and freshness according to visual analysis. Then, they were stored at 4.5 ± 0.2 °C (temperature) and $92.3 \pm 0.4\%$ (relative humidity) in a refrigerator (Samsung SR-34RMB, Seoul, South Korea) before processing for a maximum time period of 5 days. The samples had a diameter of 12 ± 0.2 mm. The moisture content was determined following AOAC methodology no. 934.06 (AOAC 1990). All samples were dried in a convective tray dryer at 70 ± 0.2 °C (in triplicate), which includes a fan and a control panel that monitors air velocity and temperature. The air flow rate was 2.0 ± 0.1 m/s. The samples to be processed (100.12 ± 0.86 g) were arranged in a layer within a stainless steel basket, which is placed on a digital balance (Ohaus, SP402, USA) accurate to ± 0.01 g and connected to a PC using an electronic interfacing device (Ohaus, RS232). The computer recorded and stored all data from the balance in real time until the sample reaches an equilibrium condition (constant weight) using the Microsoft® Hyperterminal software.

Pretreatments

The characteristics of selected pretreatments are based on studies by Vega-Gálvez et al. (2011) for the enzymatic solution treatment, Yücel et al. (2010) for the high hydrostatic pressure treatment, Doymaz (2007) for the NaOH solution treatment, and Soysal et al. (2009) for the microwave drying treatment. Thus, blueberry samples were subjected to four different pretreatments prior to dehydration at 70 °C, which are described as follows:

CO: Fresh samples were loaded directly on to drying trays without any pretreatment. These were the non-treated or control samples.

SHP: Fresh samples were immersed in a NaOH solution (QUIMIS, Q.251.2, Sao Paulo, Brazil), 1.5% (w/v) at 45 °C for 10 s.

ESP: Fresh samples were immersed in an enzymatic solution called Pectinex 3XL (Novo Nordisk Ferment, Flawil, Switzerland), 8% (v/v) at 50 °C for 30 min.

MWP: Fresh samples were dried in a microwaves oven of 1,500 W (Samsung, model G245C, Seoul, South Korea) for 10 s.

HHP: Fresh samples were packed in polyethylene bags, then heat-sealed and exposed to high hydrostatic pressure (HHP) at 450 MPa for a time period of 30 s at 20 °C. HHP treatments were performed in an isostatic pressing system (Avure Inc., Kent, WA, USA) within a cylindrical pressure chamber with a length of 700 mm and a diameter of 600 mm. Water at room temperature was the pressurizing medium. Pressurized samples were immediately stored at 4 °C until further processing.

Water Diffusion Coefficient

In order to study the mass transfer phenomena during the dehydration of blueberry samples, Fick's second law of diffusion was used. This law has been widely applied to describe the drying process during the falling rate period for food and biological materials (Akpinar 2006; Doymaz 2009). In this law, the dependent variable is the moisture ratio (MR), which relates the gradient of the sample moisture content to both initial and equilibrium moisture contents, Eq. 1 (Doymaz 2005). Equation 2 presents the mathematical solution of Fick's second law when internal mass transfer is the controlling mechanism and one-dimensional transport if a spherical geometry is assumed (Crank 1975). For sufficiently long drying times, the first term ($i=1$) in the series expansion of Eq. 2 gives a good estimation of the solution and can be applied to determine the water diffusion coefficients for each working temperature and pretreatment method (Eq. 3).

$$MR = \frac{X_{wt} - X_{we}}{X_{wo} - X_{we}} \quad (1)$$

$$MR = \frac{6}{\pi^2} \sum_{i=1}^{\infty} \frac{1}{i^2} \cdot \exp\left(\frac{D_{we} i^2 \pi^2 t}{r^2}\right) \quad (2)$$

$$MR = \frac{6}{\pi^2} \cdot \exp\left(\frac{-D_{we} \pi^2 t}{r^2}\right) \quad (3)$$

where X_{wt} is the moisture content (grams water/gram d.m.), X_{wo} is the initial moisture content (grams water/gram d.m.), X_{we} is the equilibrium moisture content (grams water/gram d.m.), D_{we} is the water diffusion coefficient (square meters per second), t is the drying time (seconds), r is the product radius (meters), i is the number of terms, and d.m. is dry matter.

Mathematical Modeling of Drying Kinetics

Different mathematical models have been proposed to describe the drying kinetics of food and bioproducts which are derived from the diffusional model of Fick's second law for different geometries (Doymaz 2005; Akpinar 2006; Corzo et al. 2008; Doymaz 2009). In addition, these models have been commonly used in industrial designs for dryers and drying time determination (Kiranoudis et al. 1992). Also, they offer a compromise between theory and ease of use (Turhan et al. 1997). Experimental drying curves were fitted to five thin-layer drying models, namely logarithmic (Eq. 4), two terms (Eq. 5), modified Henderson–Pabis (Eq. 6), Midilli–Kucut (Eq. 7), and standardized Weibull (Eq. 8). The mathematical expressions of these models are as follows:

$$MR = C + n_1 \cdot \exp(-k_1 t) \quad (4)$$

$$MR = n_2 \cdot \exp(-k_2 t) + n_3 \cdot \exp(-k_3) \quad (5)$$

$$MR = n_4 \cdot \exp(-k_4 t) + n_5 \cdot \exp(-k_5) + n_6 \cdot \exp(-k_6) \quad (6)$$

$$MR = n_7 \cdot \exp(-k_7 t^{n_8}) + C t \quad (7)$$

$$MR = \exp\left(-\left(\frac{t}{\beta}\right)^\alpha\right) \quad (8)$$

In this work, the shrinkage and external resistance were assumed as negligible (Simal et al. 2005; Hii et al. 2009). The parameter k_i ($i=1 \dots 7$) is known as a kinetic parameter, which could be considered as a pseudo-diffusivity (Azzouz et al. 2002; Simal et al. 2005). Parameters n_i ($i=1 \dots 8$) and C are known as empirical parameters, which are proposed to depend on the existence of an external skin and air drying velocity (Babalís and Belessiotis 2004). The shape parameter (α) is related to the velocity of the mass transfer at the beginning, e.g., the lower the α value, the faster the drying rate at the beginning (Corzo et al. 2008). Parameter β can be interpreted as a kinetic reaction constant and represents the time when concentration, in this case, $X_{wt} - X_{we}$, attains a value corresponding to 36.8% of $X_{wo} - X_{we}$ (Marabi et al. 2003).

Statistical Analysis

For modeling the drying kinetics, the goodness of fit between the predicted and experimental data was evaluated

based on statistical analyses including the determination coefficient (Eq. 9), sum of squared error (Eq. 10), root sum squared error (Eq. 11), and chi-square (Eq. 12; Akpınar 2006; Doymaz 2007). The effect of pretreatments on the water diffusion coefficients and empirical parameters was estimated using Statgraphics Plus® 5.1 (Statistical Graphics Corp., Herndon, VA, USA). The results were analyzed by an analysis of variance (ANOVA). Differences between the media were analyzed using the least significant difference test with a significance level of $\alpha=0.05$ and a confidence interval of 95% ($p<0.05$). In addition, the multiple range test was used to demonstrate the existence of homogeneous groups.

$$r^2 = \frac{\sum_{i=1}^N (MR_{calc,i} - MR_{exp,i})}{\sum_{i=1}^N (MR_{exp,i} - MR_{exp,i})} \quad (9)$$

$$\chi^2 = \frac{\sum_{i=1}^N (MR_{exp,i} - MR_{calc,i})^2}{N - z} \quad (10)$$

$$SSE = \frac{1}{N} \sum_{i=1}^N (MR_{exp,i} - MR_{calc,i})^2 \quad (11)$$

$$RMSE = \left[\frac{1}{N} \sum_{i=1}^N (MR_{calc,i} - MR_{exp,i})^2 \right]^{\frac{1}{2}} \quad (12)$$

where MR_{exp} is the experimental moisture ratio, MR_{calc} is the calculated moisture ratio, N is the number of data values, z is the number of constants, and i is the number of terms.

Results and Discussion

Drying Kinetics

The initial water content of blueberries was 78.5 ± 1.27 g/100 g of fresh matter. The drying process was finished when changes in mass were negligible, which occurred at 0.02 g water/gram d.m. Figure 1 shows the experimental drying curves obtained for the four pretreatments. Control samples needed 780 min to reach final water content. All pretreatments enhanced the product drying rates, decreasing considerably the drying time, as can be observed in Fig. 1. These results show that pretreatments contributed to increase the permeability of the cell membranes of blueberries, leading to an increase in water diffusivity. Also, these results could indicate the

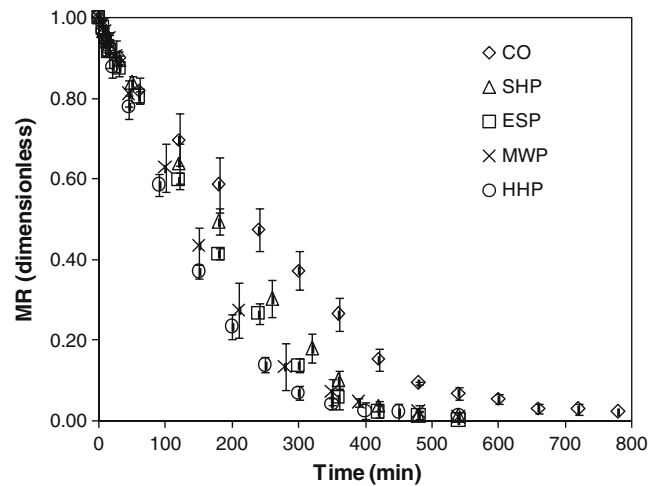


Fig. 1 Experimental drying curves of blueberries at 70 °C for different pretreatments. Values are the mean \pm standard deviation ($n=3$)

optimum characteristics for each pretreatment prior to hot air drying at a given temperature. Based on these experimental drying curves, samples pretreated with microwaves and HHP required 480 and 420 min to reach the final water content, respectively. Nevertheless, according to the ANOVA, the mentioned final process times did not show significant differences between HHP and MW ($p>0.05$). The observed pretreatment characteristics were also reported in previous investigations on seedless grapes (Di Matteo et al. 2000; Doymaz and Pala 2002), banana (Dandamrongrak et al. 2003), peach slice (Kingsly et al. 2007), and spinach (Karaaslan and Tunçer 2008). Improvement of drying rates due to HHP pretreatment could be related to the damage of the cell wall structure of the samples which results in an increase of the cells' permeability (Rastogi et al. 2000a, b; Andrés et al. 2004; Li and Ramaswamy 2006; Yücel et al. 2010).

Estimation of Water Diffusion Coefficients

Table 1 presents the water diffusion coefficients (from Eq. 3) for the different pretreatments as well as the

Table 1 Water diffusion coefficients (Eq. 3) of blueberry dried at 70 °C as a function of each pretreatment

Pretreatment	$D_{we}, \times 10^{-10}$ (m ² /s)	r^2
CO	9.51 \pm 2.19a	0.97
SHP	12.26 \pm 1.43a	0.97
ESP	15.38 \pm 1.26b	0.97
MWP	16.29 \pm 1.96b	0.98
HHP	17.71 \pm 0.88b	0.99

Values with different letters for the same column indicate significantly different D_{we} values ($p<0.05$)

Table 2 Kinetics and empirical parameters of the selected models used to simulate blueberry drying curves as a function of each pretreatment

Model name	Parameter (equation)	CO	SHP	ESP	MWP	HHP
Logarithmic	k_1	0.0028±0.0007a	0.0030±0.0006a	0.0040±0.0004b	0.0045±0.0004b	0.0059±0.0005c
	n_2	1.1737±0.1037a	1.3233±0.1185a	1.1933±0.0208a	1.1933±0.0462a	1.1033±0.0404a
	C	-0.1781±0.1087a	-0.3233±0.01253b	-0.1853±0.0232a,b	-0.1727±0.0426a	-0.0948±0.0421a
Two terms	k_2	4.8685±0.8280a	0.0050±0.0005b	9.0005±1.0642c	0.0067±0.0008d	4.7835±0.6343a
	n_2	-0.0115±0.0008a	0.4549±0.0674b	-0.0454±0.0346c	1.0346±0.0430d	-0.0355±0.0086e
	k_3	0.0036±0.0001a	0.0051±0.0005b	0.0059±0.0005b	29.595±8.4640c	0.0071±0.0001d
	n_3	1.0115±0.0007a	0.5717±0.0730b	1.0455±0.0346a	-0.0530±0.0166c	1.0355±0.0092a
Mod. H-P	$k_4 (\times 10^3)$	6.2560±0.4061a	0.2874±0.0089b	0.2450±0.0035b	0.0080±0.0004c	0.0062±0.0001c
	n_4	-0.0149±0.0057a	-0.0322±0.0104b	-0.0157±0.0073c	-0.0554±0.0132d	-0.0168±0.0017c
	k_5	0.0040±0.0003a	0.0053±0.0003b	0.0056±0.0002b	0.0059±0.0003b	0.0078±0.0001c
	n_5	0.7185±0.1972a	0.2605±0.0068b	0.3155±0.0827c	0.1880±0.0094d	0.1367±0.0221e
	k_6	0.2965±0.2029a	0.0053±0.0003b	0.0056±0.0002b	0.0059±0.0003b	0.0078±0.0001c
	n_6	0.0040±0.0003a	0.7718±0.0172b	0.7005±0.0898c	0.8673±0.0038d	0.8801±0.0043e
Midilli-Kucuk	k_7	0.0013±0.0010a	0.0006±0.0001a	0.0005±0.0001a	0.0009±0.0002a	0.0020±0.0007a
	n_7	0.9749±0.0194a	0.9672±0.0133a	0.9657±0.0238a	0.9903±0.0026b	0.9783±0.0060a
	n_8	1.2159±0.1453a	1.3891±0.3152a,b	1.4224±0.0441b	1.3441±0.0672a	1.2422±0.0578a
	$C (\times 10^{-5})$	-3.7500±2.0010a	-10.900±0.3300b	-5.4000±0.2420c	-2.4400±0.1470d	-2.6400±0.2140d
Weibull	α	1.0172±0.1718a	1.0849±0.0968a	1.2110±0.2931a	1.2411±0.0935a	1.1096±0.0717a
	β	241.32±8.4665a	176.87±17.835b	155.05±4.5026b,c	161.21±20.094b	133.71±10.954c

Values followed by different letters in the same line indicate significant difference ($p < 0.05$)

determination coefficients (from Eq. 9). Although the different pretreatments showed higher D_{we} values when compared with the control sample ($p < 0.05$), SHP did not

show statistically significant differences related to the untreated sample. Moreover, ESP, MWP, and HHP presented a homogenous group from a statistical point of view.

Table 3 Statistical tests for each empirical model as function of each pretreatment

Model name	Statistics	CO	SHP	ESP	MWP	HHP
Logarithmic	r^2	0.9921	0.9949	0.9936	0.9966	0.9976
	SSE	0.0021	0.0033	0.0025	0.0026	0.0014
	RMSE	0.0463	0.0571	0.0497	0.0506	0.0370
	χ^2	0.0032	0.0061	0.0043	0.0048	0.0021
Two terms	r^2	0.9770	0.9792	0.9813	0.9897	0.9849
	SSE	0.0021	0.0033	0.0025	0.0026	0.0024
	RMSE	0.0463	0.0571	0.0497	0.0506	0.0370
	χ^2	0.0028	0.0047	0.0035	0.0037	0.0018
Mod. H-P	r^2	0.9989	0.9941	0.9844	0.9902	0.9941
	SSE	0.0007	0.0010	0.0010	0.0010	0.0012
	RMSE	0.0266	0.0312	0.0311	0.0324	0.0346
	χ^2	0.0009	0.0013	0.0012	0.0014	0.0014
Midilli-Kucuk	r^2	0.9965	0.9923	0.9822	0.9897	0.9897
	SSE	0.0019	0.0022	0.0023	0.0019	0.0025
	RMSE	0.0373	0.0387	0.0482	0.0363	0.0440
	χ^2	0.0021	0.0025	0.0034	0.0026	0.0032
Weibull	r^2	0.9995	0.9897	0.9858	0.9943	0.9936
	SSE	0.0009	0.0013	0.0020	0.0013	0.0019
	RMSE	0.0303	0.0360	0.0450	0.0363	0.0440
	χ^2	0.0011	0.0015	0.0024	0.0016	0.0022

The microwave process resulted in a differential increase of vapor pressure between the product center and surface, allowing a higher mass transfer from food (Sham et al. 2001). The maximum and minimum D_{we} values of 17.71×10^{-10} and 9.51×10^{-10} m²/s were observed when HHP and CO were applied, respectively. Comparable results were reported in previous investigations of high-pressure treatment on pineapple (Rastogi et al. 2000a, b) and *Aloe vera* (Vega-Gálvez et al. 2011). Since improving drying times has been a major challenge for food engineers and the food processing industry, different pretreatments have been addressed in several works: microwave (Andrés et al. 2004; Alibas 2007), blanching (Walde et al. 2006; Arévalo-Pinedo and Xidieh Murr 2007), and different chemical pretreatments (Doymaz 2007; Falade et al. 2007).

Modeling of Drying Kinetics

Figure 1 shows that the decrease of moisture ratio has a clear exponential tendency; thus, the use of the empirical models proposed in this study for the complete drying process is highly recommended (Simal et al. 2005; Akpinar 2006; Doymaz 2009). Therefore, experimental data were fitted by means of the mathematical models formerly mentioned. Table 2 shows the kinetic as well as the empirical parameter values of these models for each evaluated pretreatment. In some models, there was a clear positive increase of k_i ($i=1..7$) values with the pretreatment used in comparison to the control sample, while the n_i ($i=1..8$) values remained relatively unchanged. A p value >0.05 was obtained from the ANOVA on the averages of parameters $n_i=1-8$, α , and C , suggesting that there was no significant influence of the treatment used on these empirical parameters. The same statistical evaluation (ANOVA) was carried out on the averages of the kinetic parameters $n_i=4-6$, $k_i=1-7$, and β (Table 3), obtaining a p value <0.05 , which suggests a significant influence of the treatment used on these kinetic parameters.

Statistical Analysis of Models

Table 3 shows the average values of the statistical tests performed for all the proposed models. The models showed a good fit with high values of r^2 (>0.90) and values close to zero for SSE, RMSE, and χ^2 . According to these results, the models that best fitted the experimental data, considering the statistical tests applied, were the modified Henderson–Pabis model ($r^2 \leq 0.9842$, $SSE \leq 0.0012$, $RMSE \leq 0.0346$, $\chi^2 \leq 0.0014$) followed by Weibull ($r^2 \leq 0.9858$, $SSE \leq 0.0020$, $RMSE \leq 0.0450$, $\chi^2 \leq 0.0024$). The fact that the best fit was obtained by the modified Henderson–Pabis model could be related to the model's

possession of three exponential parameters which provide a better mathematical approximation of the experimental drying curves which have exponential tendencies that decrease (Azzouz et al. 2002; Doymaz 2005). Thus, the goodness of fit of the modified Henderson–Pabis and Weibull models to approximately represent the experimental drying curves of blueberry can be observed in Fig. 2a, b, respectively, for all the pretreatments applied.

Conclusions

The results of this investigation have demonstrated that pretreatments such as enzymatic, sodium hydroxide, microwave, and high hydrostatic pressure significantly increased the drying rate of blueberries. All pretreatments reduced the drying time of the control samples

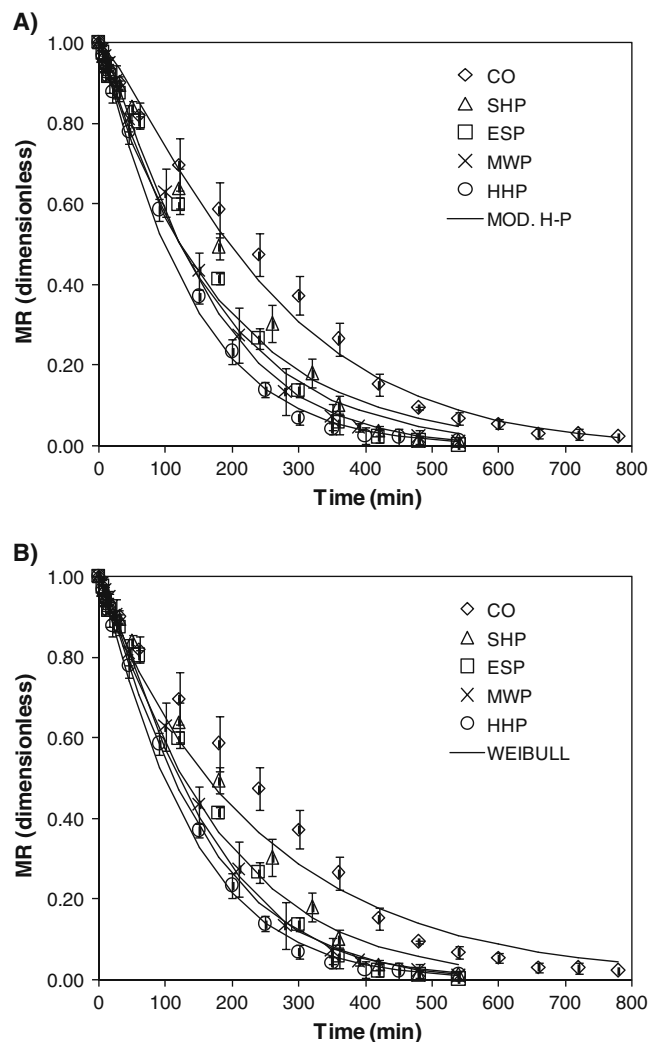


Fig. 2 Experimental and simulated drying curves of blueberries at 70 °C for different pretreatments: modified Henderson–Pabis (a) and Weibull (b). Values are the mean \pm standard deviation ($n=3$)

(780 min). In particular, HHP and MW pretreatments showed the lowest process time compared with the non-treated samples: 420 and 480 min, respectively ($p > 0.05$). The water diffusion coefficients of all pretreated samples were higher than the non-treated samples. At 70 °C, the water diffusion coefficients associated with the enzymatic solution, microwave, and HHP pretreatments did not show statistical differences, presenting values in the range of $15.38\text{--}17.71 \times 10^{-10} \text{ m}^2/\text{s}$. Mathematical modeling of experimental drying curves was satisfactorily achieved. Based on the statistical test results, the modified Henderson–Pabis model ($r^2 \geq 0.99$, $\text{SSE} \leq 0.0010$, $\text{RMSE} \leq 0.0288$, $\chi^2 \leq 0.0012$) followed by the Weibull model ($r^2 \geq 0.98$, $\text{SSE} \leq 0.0012$, $\text{RMSE} \leq 0.0312$, $\chi^2 \leq 0.0013$) can be appropriately used to simulate experimental drying curves. Taking into account all these considerations, the use of HHP could be an alternative to microwave and enzymatic pretreatments of convective dehydration to obtain final dried blueberry minimizing process time.

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