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Drying kinetics of blackberry leaves

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Key words:
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air speeds
Midilli model

ABSTRACT

Blackberry leaves have some pharmacological properties and one of the most widespread and studied uses is to relieve symptoms of the climacteric and other symptoms during the premenstrual period. Thus, drying becomes important for the conservation and storage of the product until its use or processing. The present study aimed to evaluate the drying kinetics of blackberry leaves, as well as to determine the effective diffusion coefficient and the activation energy during the drying process. Blackberry leaves were dried in an experimental fixed-bed dryer under four controlled temperature conditions (40, 50, 60 and 70 °C) and two drying air speeds (0.4 and 0.8 m s^{-1}). With the experimental data of moisture ratio, eight mathematical models were fitted to represent the process of thin-layer drying of agricultural products. Based on the obtained results, it was found that the Midilli model represented best the phenomenon of drying of blackberry leaves. The increase in temperature and air speed reduced the drying time of blackberry leaves and increased the values of the effective diffusion coefficient. This relation can be described by the Arrhenius equation, which has an activation energy for the liquid diffusion during drying of 65.94 and 66.08 kJ mol^{-1}, for drying air speeds of 0.4 and 0.8 m s^{-1}, respectively.

Palavras-chave:
Morus nigra L.
planta medicinal
velocidade do ar
modelo de Midilli

Cinética de secagem de folhas de amora preta

RESUMO

As folhas de amora preta possuem algumas propriedades farmacológicas e um dos usos mais difundidos e estudados é para alivio dos sintomas do climatério e de outros durante o período pré-menstrual. Desta forma, a secagem se torna importante para a conservação e o armazenamento do produto até o seu uso ou processamento. Diante do exposto, objetivou-se com o presente estudo avaliar a cinética de secagem de folhas de amora preta, bem como determinar o coeficiente de difusão efetivo e a energia de ativação durante o processo de secagem. As folhas de amora preta foram submetidas à secagem em um secador experimental de leito fixo, em quatro condições controladas de temperatura (40, 50, 60 e 70 °C) e duas velocidades do ar de secagem (0,4 e 0,8 m s^{-1}). Aos dados experimentais de razão de teor de água foram ajustados oito modelos matemáticos para representarem o processo de secagem em camada delgada de produtos agrícolas. Com base nos resultados obtidos, verificou-se que o modelo de Midilli foi o que melhor representou o fenômeno da secagem de folhas de amora preta. O aumento da temperatura e da velocidade do ar reduziu o tempo de secagem das folhas de amora preta, bem como aumentou os valores do coeficiente de difusão efetivo, sendo que esta relação pode ser descrita pela equação de Arrhenius, que apresenta uma energia de ativação para a difusão líquida durante a secagem de 65,94 e 66,08 kJ mol^{-1}, para as velocidades do ar de secagem de 0,4 e 0,8 m s^{-1}, respectivamente.

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Introduction

Blackberry (*Morus nigra* L.) is a species belonging to the genus *Morus*, of the family Moraceae, having about 24 species and one subspecies, with at least 100 known varieties, found in temperate and subtropical regions (Ercisli & Orhan, 2007). *Morus nigra* L. has medicinal properties in its fruits, leaves and roots (Grandi, 2014), being widely used in the popular medicine.

Among the parts of *Morus nigra* L., leaves play a relevant role and one of the examples of use is the tea made from the leaves to relieve symptoms of climacteric, headache and irritation which occur in the premenstrual period, due to the presence of flavonoid compounds, and especially isoflavones (Lorenzi & Matos, 2008). In this context, various researchers have studied the medicinal use of blackberry leaves (Miranda et al., 2010; Freitas et al., 2016; Rosa et al., 2016).

Leaves of medicinal plants normally have high water content. Water is the main responsible for the increase in metabolic activities and chemical and physical changes which occur in the product during storage. Thus, it is fundamental to reduce water content through drying to maintain the quality of medicinal plants after harvest (Goneli et al., 2014).

The study of thin-layer drying curves provides important data for the development of processes and dimensioning of equipment intended for the drying of agricultural products. These data allow the estimation of drying time, production planning and energy expenditure involved in the process, which will affect the final value of the product (Vilela & Artur, 2008).

Water diffusivity in a product can be understood as the ease with which water is removed from this product during the drying process, and diffusivity is not an intrinsic characteristic to the product, because it varies according to the changes in the conditions of drying, temperature and air speed (Oliveira et al., 2006). Thus, it becomes also fundamental to study the behavior of the diffusion coefficient, besides the drying curves as a function of the different drying air conditions.

In this context, this study aimed to fit mathematical models to predict the thin-layer drying curves of blackberry leaves at different temperatures and air speeds, as well as to determine the effective diffusion coefficient and activation energy during the drying process.

Material and Methods

The present study was carried out at the Laboratory of Pre-processing and Storage of Agricultural Products, at the Faculty of Agrarian Sciences - FCA of the Federal University of Grande Dourados - UFGD, in the municipality Dourados, MS, Brazil, from October to November 2016.

Blackberry leaves were collected from a single plant, located at the Unit 2 of the UFGD, at the facilities of the FCA (22º 11' 45" S and 54º 55' 18" W, at altitude of 446 m), to guarantee the homogeneity of the product. Only leaves with no injuries or apparent incidence of diseases were selected. The leaves were always collected in the morning, avoiding the collection of leaves after rains or with dewdrops on the surface, to not compromise the characterization of their drying curves.

Blackberry leaves were dried in an experimental fixed-bed dryer with automatic control of drying air speed and temperature (Figure 1). The thin-layer drying bed was composed of two 0.2 m-diameter trays with screened bottom, and the blackberry leaves were arranged in a thin layer on each tray of the experimental dryer.

After collection and selection, the blackberry leaves had initial water content of approximately 2.03 (decimal, on dry basis - d.b.). Initial and equilibrium moisture contents were determined by the gravimetric method in the oven, at 103 ± 1 ºC for 24 h, in triplicate (ASABE, 2010).

The drying tests of blackberry leaves were conducted for different drying air temperatures (40, 50, 60 and 70 °C) and speeds (0.4 and 0.8 m s^{-1}) in a completely randomized design, with four replicates. Blackberry leaves were dried until they reached the equilibrium moisture content, but for mathematical modelling purposes a final moisture content of 0.11 ± 0.01 (decimal, d.b.) was considered. The moisture content of the blackberry leaves during the drying under the different conditions of the air was determined by Eq. 1.

$$MR = \frac{M - M_e}{M_i - M_e} \tag{1}$$

where:

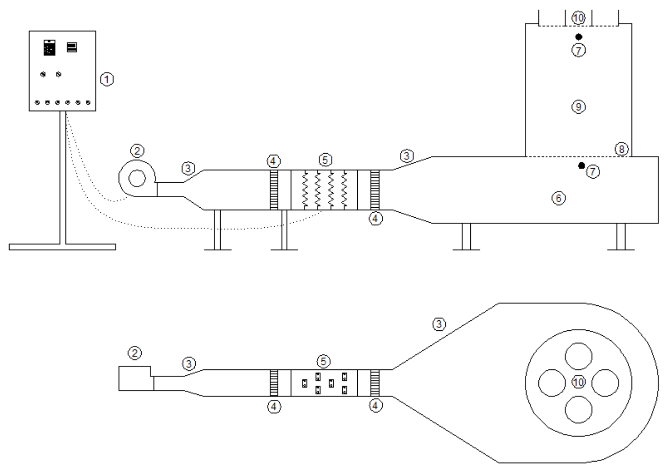
MR - moisture ratio of the product, dimensionless;

M - moisture content of the product at a certain time, decimal, d.b.;

M_e - equilibrium moisture content of the product, decimal, d.b.; and,

M_i - initial moisture content of the product, decimal, d.b.

Mathematical models traditionally used to predict the thin-layer drying of agricultural products were fitted to the moisture content data observed during the drying of blackberry leaves. The mathematical models presented in Table 1 have been used by various researchers in studies with leaves of medicinal plants (Prates et al., 2012; Goneli et al., 2014; Martins et al., 2015; Silva et al., 2015; Gasparin et al., 2017; Gomes et al., 2017).



1 - Control panel; 2 - Centrifugal fan; 3 - Expansions; 4 - Air homogenizers; 5 - Electric resistances; 6 - Plenum; 7 - Air temperature measurement point; 8 - Screened bottom; 9 - Drying bed; 10 - Trays for thin-layer drying.
Source: Adapted from Martins (2015)

Figure 1. Experimental dryer used in the drying of blackberry leaves

Table 1. Mathematical models used to estimate the thin-layer drying curves of blackberry leaves

Model designation	Model	Eq.
Approximation of diffusion	MR = a exp(-k θ) + (1 - a) exp(-k b θ)	(2)
Two terms	MR = a exp($-k_0$ θ) + b exp($-k_1$ θ)	(3)
Two-term exponential	MR = a exp(-k θ) + (1 - a) exp(-k a θ)	(4)
Henderson & Pabis	MR = a exp(-k θ)	(5)
Logarithmic	MR = a exp(-k θ) + c	(6)
Midilli	MR = a exp(-k $θ^n$) + b θ	(7)
Newton	MR = exp(-k θ)	(8)
Page	MR = exp(-k $θ^n$)	(9)

θ - Drying time; h; k, k_o, k_1 - Drying constants; h^{-1}; a, b, c, n - coefficients of the models

Effective diffusion coefficients of blackberry leaves were obtained by fitting the liquid diffusion mathematical model (Eq. 10), with eight terms, to the data observed during the drying under different air conditions.

$$MR = \frac{M - M_e}{M_i - M_e} = \frac{8}{\pi^2} \sum_{n_t=0}^{\infty} \frac{1}{(2n_t+1)^2} \exp\left[-(2n_t+1)^2 \pi^2 D_i \left(\frac{\theta}{4L}\right)^2\right] \quad (10)$$

where:

D_i - effective diffusion coefficient, $m^2\ s^{-1}$;
L - product thickness, m; and,
n_t - number of terms of the model.

Thickness (L) of blackberry leaves was measured using a digital caliper, with 0.01 mm resolution. Thickness was measured in 50 leaves, with six measurements at different points in each one. After the measurements, mean thickness was calculated and was equal to 0.427 mm. The effect of temperature on the effective diffusion coefficient was assessed using the Arrhenius equation, as described in Eq. 11.

$$D_i = D_0 \exp\left(\frac{E_a}{R\ T_a}\right) \quad (11)$$

where:

D_o - pre-exponential factor;
E_a - activation energy, kJ mol^{-1};
R - universal gas constant, 8.314 kJ $kmol^{-1}$ K^{-1}; and,
T_a - absolute temperature, K.

The mathematical models were fitted to the moisture ratio data observed during the thin-layer drying of blackberry leaves, under the different conditions of the air, through nonlinear regression analysis by the Gauss-Newton method using a statistical computer program.

The mathematical model to represent the drying of blackberry leaves was selected by assessing the degree of fit of each model, based on the magnitude of the adjusted coefficient of determination (R^2), mean relative error (P), standard deviation of the estimate (SE) and residual distribution behavior. P and SE values were calculated using Eqs. 12 and 13, respectively.

$$P = \frac{100}{n_o} \sum_{i=1}^{n_o} \left(\frac{|Y - \hat{Y}|}{Y}\right) \quad (12)$$

$$SE = \sqrt{\frac{\sum_{i=1}^{n_o} (Y - \hat{Y})^2}{DF}} \quad (13)$$

where:

n_o - number of experimental observations;
Y - value observed experimentally;
$\hat{Y}$ - value estimated by the model; and,
DF - degrees of freedom of the model.

Results and Discussion

To select mathematical models to represent the thin-layer drying of agricultural products, the mean relative error (P) is considered as a parameter of exclusion of models because, according to Mohapatra & Rao (2005), models with P higher than 10% are inadequate to represent the drying process. P values indicate the deviation of the observed data from the curve estimated by the model (Kashaninejad et al., 2007). Another parameter also considered as of exclusion of mathematical models is the residual distribution because, according to Goneli et al. (2009), if a model has biased residual distribution it is considered as inadequate to represent the phenomenon, but if it has random residuals (residual values distributed close to the horizontal strip around zero), it is considered as acceptable.

Thus, based on these two statistical parameters of the models fitted to the observed moisture ratio data of blackberry leaves (Table 2), the only acceptable model to represent the thin-layer drying, for all conditions of drying air temperature and speed evaluated in the present study, is the Midilli model (Eq. 7).

It can also be observed in Table 2 that, based on P values and residual distribution, for air speed of 0.8 m s^{-1} two models are acceptable to represent the thin-layer drying of blackberry leaves: Logarithmic (Eq. 6) and Midilli (Eq. 7). In these cases, the model that best fits to the observed data is selected considering also the highest coefficients of determination (R^2) and lowest standard deviations of the estimate (SE). For all air conditions, the Midilli model (Eq. 7) has the highest R^2 values and lowest SE values among all others, thus reinforcing its best fit to the data of thin-layer drying of blackberry leaves.

Gasparin et al. (2017), studying the drying kinetics of *Mentha piperita* leaves at different drying air temperatures and speeds, also found that the model which fitted best to the observed data was the Midilli model. Other researchers have also found that the Midilli model was the most adequate to represent the thin-layer drying of medicinal plants, such as Gomes et al. (2017) with *Cymbopogon citratus* leaves, Silva et al. (2015) with 'jenipapo' leaves, Martins et. al (2015) with 'timbó' leaves and Goneli et al. (2014) with 'aroeira' leaves.

The better fit of the Midilli model to the observed drying data of medicinal plants, according to Goneli et al. (2014), is probably related to the fast water loss in the initial stages of the process in this type of product, generating a drying curve that is steeper and better characterized mathematically by this model.

The moisture ratios estimated by the Midilli model were highly correlated with the observed drying data of blackberry leaves under the different drying air conditions. This is

Table 2. Statistical parameters obtained in the drying of blackberry leaves

Models	0.4 m s⁻¹ SE (decimal)	P (%)	R^2 (decimal)	Residual	0.8 m s⁻¹ SE (decimal)	P (%)	R^2 (decimal)	Residual
40 °C								
Approximation of diffusion	0.0169	8.4960	0.9971	BS	0.0067	4.9932	0.9995	RD
Two terms	0.0312	18.2692	0.9902	BS	0.0342	35.5409	0.9862	BS
Two-term exponential	0.0178	9.4502	0.9967	BS	0.0170	16.9787	0.9965	BS
Henderson & Pabis	0.0307	18.2690	0.9902	BS	0.0311	30.5449	0.9884	BS
Logarithmic	0.0098	5.1307	0.9990	RD	0.0079	6.6288	0.9992	RD
Midilli	0.0082	2.9980	0.9993	RD	0.0057	3.2213	0.9996	RD
Newton	0.0326	20.5178	0.9888	BS	0.0345	35.1279	0.9854	BS
Page	0.0194	9.2402	0.9961	BS	0.0172	15.0631	0.9964	BS
50 °C								
Approximation of diffusion	0.0289	25.7472	0.9906	BS	0.0081	5.5964	0.9992	RD
Two terms	0.0262	21.8166	0.9924	BS	0.0256	20.7900	0.9928	BS
Two-term exponential	0.0121	7.9068	0.9983	BS	0.0137	10.0025	0.9978	BS
Henderson & Pabis	0.0255	21.8170	0.9924	BS	0.0248	20.7895	0.9928	BS
Logarithmic	0.0135	11.6465	0.9980	BS	0.0094	7.1292	0.9990	RD
Midilli	0.0091	5.6663	0.9991	RD	0.0087	4.9119	0.9992	RD
Newton	0.0282	25.7474	0.9906	BS	0.0267	23.6281	0.9914	BS
Page	0.0131	6.8514	0.9980	BS	0.0148	9.5861	0.9974	BS
60 °C								
Approximation of diffusion	0.1484	48.7990	0.7620	BS	0.0192	12.7678	0.9963	BS
Two terms	0.0382	29.3516	0.9847	BS	0.0407	28.8397	0.9839	BS
Two-term exponential	0.0198	11.1774	0.9956	RD	0.0207	14.6769	0.9955	BS
Henderson & Pabis	0.0370	27.6202	0.9848	BS	0.0390	28.8398	0.9839	BS
Logarithmic	0.0210	14.6172	0.9952	RD	0.0138	9.4836	0.9981	RD
Midilli	0.0141	4.7146	0.9979	RD	0.0111	6.1239	0.9988	RD
Newton	0.0402	32.3802	0.9816	BS	0.0432	33.4536	0.9795	BS
Page	0.0197	7.6632	0.9957	RD	0.0194	11.2890	0.9960	BS
70 °C								
Approximation of diffusion	0.0280	20.4360	0.9931	BS	0.0469	30.9039	0.9808	BS
Two terms	0.0554	41.2041	0.9747	BS	0.0525	30.9043	0.9808	BS
Two-term exponential	0.0296	23.2203	0.9918	BS	0.0241	16.2139	0.9949	BS
Henderson & Pabis	0.0517	42.3852	0.9749	BS	0.0469	30.9039	0.9808	BS
Logarithmic	0.0190	14.8630	0.9968	RD	0.0125	7.4549	0.9988	RD
Midilli	0.0162	9.5931	0.9978	RD	0.0106	4.9279	0.9992	RD
Newton	0.0555	48.0328	0.9692	BS	0.0494	34.5807	0.9766	BS
Page	0.0258	15.9781	0.9938	BS	0.0213	12.2643	0.9960	BS

BS - Biased residual distribution; RD - Random residual distribution

demonstrated in Figure 2, in which it is possible to observe the proximity between the data estimated by the model and the data observed during the process, thus reinforcing the applicability of this model to estimate the drying curves of blackberry leaves.

The increase in drying air temperature considerably reduces the time required for blackberry leaves to reach the moisture content of approximately 0.11 (decimal, d.b.) (Figure 2). This phenomenon was observed by Radünz et al. (2011), who assessed the drying kinetics of 'carqueja', as well as other researchers studying the drying kinetics of other medicinal plants, such as Prates et al. (2012) with 'fruta-de-lobo' leaves, Goneli et al. (2014) with 'aroeira' leaves, Martins et al. (2015) with 'timbó' leaves, Silva et al. (2015) with 'jenipapo', Gasparin et al. (2017) with *Mentha piperita* leaves.

Still in Figure 2, it is also possible to note the effect of air speed on the thin-layer drying curves of blackberry leaves; the increase in air speed reduced the drying time. The effect of air speed is more accentuated at the lowest drying air temperatures; as air temperature increases, there is a reduction in the influence of the speed on the time spent to dry the product.

The more pronounced effect of drying air temperature, compared with its speed, on the reduction of blackberry leaves drying time can be attributed to the fact that the main cause of the drying process is the difference in vapor pressure between the product and the drying air. Vapor pressure difference increases with the increment in drying air temperature, and air speed does not cause alterations in the vapor pressure difference between air and product (Martins, 2015).

The greater influence of drying air speed at lower temperatures, as observed in Figure 2 for the air temperature of 40 °C, can be explained by the fact that water evaporation initially occurs on the product's surface, which causes drying air speed to have greater importance in the beginning of the process, as explained by Babalis et al. (2006). These authors also explain that water evaporation initially on the product's surface is replaced by an evaporation front which moves to the inside of the product, causing the effect of drying air speed to be followed by the liquid diffusion process, which becomes the most important factor for the drying process.

Since at lower temperatures the time required to remove water present on the product's surface is longer than at higher temperatures, it causes the effect of drying air speed to be more pronounced at lower temperatures, due to the longer time during which it contributes to removing water present on the surface. Thus, the higher the drying air speed, the greater the contribution of this factor to the removal of water from the product's surface.

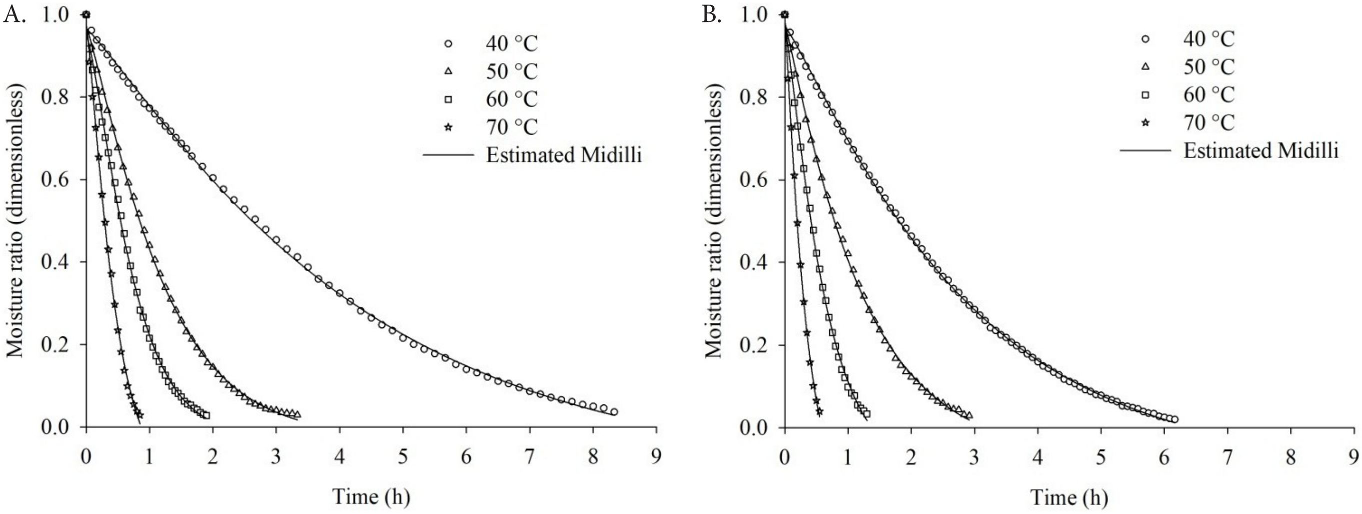


Figure 2 . Moisture ratio data observed and estimated by the Midilli model for the thin-layer drying of blackberry leaves at air speeds of 0.4 m s^{-1} (A) and 0.8 m s^{-1} (B)

The constant ‘k’ of the Midilli model increased with the increment in drying air temperature and speed (Table 3). The constant ‘k’ can be used as an approximation to characterize the effect of temperature and is related to the effective diffusivity in the decreasing period of the drying process, and liquid diffusion controls the process (Babalis & Belessiotis, 2004).

The coefficients “a”, “n” and “b” of the Midilli model (Table 3) do not exhibit a defined trend in their magnitudes as a function of the increase in drying air speed, except the coefficient ‘b’ in the drying tests conducted with air speed of 0.8 m s^{-1}.

As can be observed in Table 3, the effective diffusion coefficients (D_i) increase with the increment in drying air temperature and speed. According to Goneli et al. (2009), as temperature increases there is also an increase in the level of vibration of water molecules and reduction in water viscosity, which is a measure of resistance of a fluid to flowing. Variations in this property lead to alterations of water diffusion in the capillaries of agricultural products, which contribute to a faster diffusion along with more intense vibration of water molecules.

The increase in D_i with the increment in drying air speed can be attributed to the fact that the increment in air speed contributes to the evaporation of water, which moves to the product’s surface (Martins et al., 2015). Similar behavior was observed by Kaya & Aydin (2009) studying the drying curves of mint and nettle leaves.

The dependence of D_i values of blackberry leaves increased with the increment in drying air temperature and speed, as observed in Figure 3. A similar behavior was observed by Kaya & Aydin (2009).

The slope of the Arrhenius curve for the thin-layer drying of blackberry leaves (Figure 3) is used to obtain the E_a/R ratio, and its intersection with the Y-axis is used to obtain

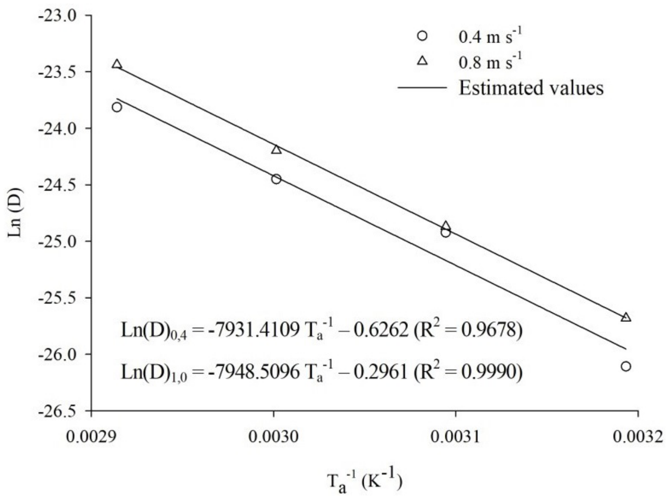


Figure 3. Arrhenius representation for the effective diffusion coefficient as a function of different conditions of the air used to dry blackberry leaves

the D_o value. Eqs. 14 and 15 present the coefficients of the Arrhenius equation fitted to the effective diffusion coefficients of blackberry leaves for the drying air speeds of 0.4 and 0.8 m s^{-1}, respectively, calculated according to Eq. 11.

$$D_i = 0.5346 \exp \frac{65.9418}{R\ T_a} \quad (14)$$

$$D_i = 0.7437 \exp \frac{66.0839}{R\ T_a} \quad (15)$$

The activation energy for liquid diffusion in the temperature range from 40 to 70 °C during the drying of blackberry leaves

Table 3. Parameters of the Midilli model and effective diffusion coefficient (D_i x 10^{-11} m^2 s^{-1}) for the different conditions of the air used to dry blackberry leaves

Air temperature (°C)	0.4 m s^{-1}					0.8 m s^{-1}				
	a	k	n	b	D_i	a	k	n	b	D_i
40	0.9659	0.2055	1.1448	-0.0079	0.4590	0.9719	0.3198	1.1442	-0.0102	0.7038
50	0.9696	0.7997	1.1888	-0.0050	1.5008	0.9801	0.8472	1.1018	-0.0150	1.5835
60	0.9501	1.4236	1.3217	-0.0071	2.4058	0.9779	1.7984	1.1927	-0.0509	3.1023
70	0.9774	2.8382	1.2413	-0.1044	4.5459	0.9921	4.0350	1.1512	-0.1894	6.6212

was approximately of 65.94 and 66.08 kJ mol^{-1} (Eqs. 14 and 15), for the drying air speeds of 0.4 and 0.8 m s^{-1}, respectively. According to Kashaninejad et al. (2007), the activation energy is a barrier that must be overcome for the diffusion process to be triggered in the product.

Activation energy values did not vary much as a function of the variation in drying air speed; these values are higher than those found by other researchers working with medicinal plants, such as *Cymbopogon citratus* leaves (53.76 kJ mol^{-1}) (Gomes et al., 2017), 'fruta-de-lobo' leaves (44.60 kJ mol^{-1}) (Prates et al., 2012), 'jenipapo' leaves (33.87 kJ mol^{-1}) (Silva et al., 2015).

Conclusions

1. Among the models fitted, Midilli was the only one with satisfactory fit to the observed data for the drying air conditions studied.

2. The increase in drying air temperature and speed caused a reduction in drying time, but the effect of increased air speed was more pronounced, regarding the drying time, at the lowest temperatures evaluated.

3. Effective diffusion coefficients increased with the increment in drying air temperature and speed, whereas the activation energy increased slightly with the increment in drying air speed.

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