



Study of the drying kinetics of black mulberry (*Morus nigra* L.) leaves

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Abstract

Black mulberry leaves have been used to promote health and prevent diseases due to their biological properties. Drying leaves allows greater stability during storage and marketing. However, studying this operation is important to ensure that the properties are maintained. Therefore, the objective of this study was to use mathematical modeling to define the model that best fits to the experimental data of the drying kinetics at temperatures of 40, 50, 60, and 70°C. Liquid diffusion, leaf surface area, the relationship between temperature increase and diffusion coefficient, and energy activation were also determined. Among the mathematical models studied, the Page model was the one that best described the drying process of black mulberry leaves. As for the diffusion coefficient, the values increase as the drying temperature increases. The activation energy was 65.418 kJ mol⁻¹, and it was observed that the lower the activation energy, the greater the water diffusivity in the product. The drying kinetics data of black mulberry leaves allowed a better understanding of this operation in a matrix with wide application potential. Drying black mulberry leaves at different temperatures altered their color. Considering the evaluations, temperatures of 40 and 50°C are the most indicated for drying black mulberry leaves.

Keywords: mathematical modeling, convective drying, *Morus nigra* L.

Practical application: Optimization of the drying of black mulberry leaves.

1 INTRODUCTION

Black mulberry is a plant species that originated in Asia, fruiting with greater intensity and abundance in Asia Minor and being fully acclimatized in Brazil (Cruz, 1979). According to Tutin et al. (1996), the genus *Morus* consists of approximately 24 species and 1 subspecies, with at least 100 varieties described. Black mulberry leaves have a beneficial effect in the treatment of respiratory diseases such as asthmatic bronchitis. These leaves are also used in the treatment of intestinal infections, intestinal cramps, intestinal spasms, diarrhea, and abdominal pain (Malaquinas & Costa, 2006).

Reports of folk medicine suggest the use of black mulberry leaves for women during the menopause period, and studies seek to prove their action on estrogen receptors (Franzotti et al., 2004). These benefits are associated with the composition of the leaves, consisting of compounds such as phenolics, which have antioxidant properties and contribute to reducing oxidative stress in cells (Dalmagro et al., 2018). Studies have also reported hypoglycemic, hypolipidemic, anti-inflammatory, and antiatherogenic properties (Hago et al., 2021; Sánchez-Salcedo et al., 2016). Dietary supplementation of *Morus nigra* L. leaves has been shown to partially reduce fat mass by increasing lipolysis in pig models (Fan et al., 2020). Given these benefits, conservation

methods must be applied to preserve black mulberry leaves and enable their use.

An important operation applied in food preservation is drying, which brings perishable food to a stable state (Azmir et al., 2019). It is one of the oldest operations and is most widely applied to food preservation (Llavata et al., 2020). For agricultural products, this technique is used to promote quality and stability, as it provides the opportunity to reduce the amount of water in the material, besides reducing the biological activity and the chemical and physical changes that occur during storage (Calín-Sánchez et al., 2020; Resende et al., 2018).

Drying consists of removing a large part of the volume of water from the product, until creating unfavorable conditions for the continuity of its metabolic activities and the development of microorganisms (Hariadi et al., 2023; Martinazzo et al., 2007). Controlled and proper application can result in the maintenance of nutrients, color, and fragrance in the dried leaves (Pantoja Espinosa et al., 2022). In an attempt to predict the phenomena that occur during the drying kinetics of agricultural products, several mathematical models have been used to describe these processes (Jiang et al., 2023; Resende et al., 2010). In view of the above, the study aimed to analyze the drying kinetics of black mulberry (*Morus nigra* L.) leaves and determine the effective diffusivity and activation energy under different drying conditions.

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2 MATERIAL AND METHODS

2.1 Obtaining black mulberry leaves

Black mulberry (*M. nigra* L.) leaves were collected in the rural area of the city of Quirinópolis (18.482 S, 50.421 W), Goiás, Brazil, in October 2016. The experiment was conducted at the Laboratory of Post-Harvest of Plant Products of the Instituto Federal de Educação, Ciência e Tecnologia de Goiás - Rio Verde Campus.

2.2 Drying study

Leaves were manually harvested with an initial moisture content of approximately 2.2 (decimal, d.b.) and dried in a forced air circulation oven at four different temperatures, 40, 50, 60, and 70°C, and the experiment was carried out in quadruplicate. The gravimetric method was applied in the experiment, with periodical measurement of the masses of the trays on a digital scale with 0.001 g resolution. The leaves were dried until reaching equilibrium moisture contents of 0.08, 0.06, 0.04, and 0.02 (d.b.) for temperatures of 40, 50, 60, and 70°C, respectively. The moisture content ratio was determined using Equation 1.

$$RX = \frac{X - X_e}{X_i - X_e} \quad (1)$$

where:

RX: the moisture content ratio of the product (dimensionless);

X: the moisture content of the product (w.b.);

X_i : the initial moisture content of the product (w.b.);

X_e : the equilibrium moisture content of the product (d.b.).

A total of 11 mathematical models frequently used to represent the drying of agricultural products (Table 1) were fitted to

Table 1. Mathematical models that were used to predict the drying curve of black mulberry leaves.

Models' Designations	Models	Equations
Page	$RX = \exp(-k \cdot t^n)$	(2)
Henderson & Pabis	$RX = a \cdot \exp(-k \cdot t)$	(3)
Midilli	$RX = a \cdot \exp(-k \cdot t^n) + b \cdot t$	(4)
Diffusion Approximation	$RX = a \cdot \exp(-k \cdot t) + (1-a) \cdot \exp(-k \cdot b \cdot t)$	(5)
Two-Term Exponential	$RX = a \cdot \exp(-k \cdot t) + (1-a) \exp(-k \cdot a \cdot t)$	(6)
Logarithmic	$RX = a \cdot \exp(-k \cdot t) + c$	(7)
Thompson	$RX = \exp\left(\left(-a \cdot (a^2 + 4 \cdot b \cdot t)^{0.5}\right) / 2 \cdot b\right)$	(8)
Newton	$RX = \exp(-k \cdot t)$	(9)
Verma	$RX = a \cdot \exp(-k \cdot t) + (1-a) \exp(-k_1 \cdot t)$	(10)
Wang & Singh	$RX = 1 + a \cdot t + b \cdot t^2$	(11)

t: drying time; h, k, k_1 , and k_2 : drying constants; h^{-1} , a, b, c, and n: model parameters.

the obtained experimental data by nonlinear regression analysis using the Gauss–Newton method.

The criteria for fitting the models to the data were the magnitude of the mean relative error (P), standard deviation of the estimate (SE), and coefficient of determination (R^2), at a 5% significance level, according to Equations 12 and 13, respectively:

$$P = \frac{100}{n} \sum \frac{|Y - \hat{Y}|}{Y} \quad (12)$$

$$SE = \sqrt{\frac{\sum (Y - \hat{Y})^2}{DF}} \quad (13)$$

where:

Y: the experimental value;

\hat{Y} : the value estimated by the model;

N: the number of experimental observations;

DF: the degrees of freedom of the model (number of experimental observations minus the number of model parameters).

To select a single model to describe the drying of black mulberry leaves under each condition, the models that obtained the best fits were subjected to the Akaike Information Criterion (AIC) and Schwarz's Bayesian Information Criterion (BIC). Lower AIC and BIC values indicate a better fit of the model, and BIC is the most rigorous criterion (Wolfinger, 1993). According to Gomes et al. (2018), these criteria can be additionally included in the selection of drying models. The information criteria were determined by Equations 14 and 15.

$$AIC = -2 \log L + 2p \quad (14)$$

$$BIC = -2 \log L + p \ln(N-r) \quad (15)$$

where:

p: the number of model parameters;

N: the total number of observations;

r: the rank of the X matrix (fixed effects incidence matrix);

L: the maximum likelihood.

Liquid diffusion was described using the model, which considers the geometric shape of a flat plate with an eight-term approximation, using Equation 16:

$$RX = \frac{X - X_e}{X_i - X_e} = \left(\frac{8}{\pi^2}\right) \sum_{n=0}^{\infty} \frac{1}{(2n+1)^2} \exp\left[-\frac{(2n+1)^2 \cdot \pi \cdot D \cdot t}{4} \cdot \left(\frac{S}{V}\right)\right] \quad (16)$$

where:

RX: the moisture content ratio of the product (dimensionless);

n: the number of terms;

D: the effective diffusivity ($\text{m}^2 \text{s}^{-1}$);

S: the leaf surface area (m^2);

V: the leaf volume (m^3).

The leaf surface area was determined using the ImageJ program. A total of 15 black mulberry leaves were used for the measurements, and their thickness was measured in 5 different regions using a digital caliper with a 0.01 mm resolution. With the mean data of thickness and leaf surface area, it was possible to calculate the volume according to Equation 17:

$$V = S \cdot LT \quad (17)$$

where:

LT: leaf thickness.

The Arrhenius equation was used to determine the relationship between temperature increase and effective diffusion coefficient (Equation 18):

$$D = D_o \cdot \exp\left(\frac{-E_a}{R \cdot T_{\text{abs}}}\right) \quad (18)$$

where:

D_o : the pre-exponential factor;

E_a : the activation energy (kJ mol^{-1});

R: the universal gas constant ($8.134 \text{ kJ kmol}^{-1} \text{ K}^{-1}$);

T_{abs} : the absolute temperature (K).

The activation energy was determined by linearizing the coefficients of the Arrhenius equation and applying the logarithm as follows (Equation 19):

$$\text{Ln}D = \text{Ln}D_o - \frac{E_a}{R} \cdot \frac{1}{T_{\text{abs}}} \quad (19)$$

where:

D: the effective diffusion coefficient ($\text{m}^2 \text{s}^{-1}$);

D_o : the pre-exponential factor ($\text{m}^2 \text{s}^{-1}$);

E_a : the activation energy (kJ mol^{-1});

R: the universal gas constant ($8.134 \text{ kJ kmol}^{-1} \text{ K}^{-1}$);

T_{abs} : the absolute temperature (K).

2.3 Thermodynamic properties of drying

The thermodynamic properties of the drying process of black mulberry leaves were obtained by the method described by Jideani and Mpotokwana (2009) (Equations 20–22):

$$\Delta H = E_a - R \cdot T \quad (20)$$

$$\Delta S = R \cdot \left(\ln D_o - \ln \frac{k_B}{h_p} \right) - \ln T_{\text{abs}} \quad (21)$$

$$\Delta G = \Delta H - T_{\text{abs}} \cdot \Delta S \quad (22)$$

where:

ΔH : the enthalpy (J mol^{-1});

ΔS : the entropy (J mol^{-1});

ΔG : the Gibbs free energy (J mol^{-1});

k_B : Boltzmann constant ($1.38 \times 10^{-23} \text{ J K}^{-1}$);

h_p : Planck constant ($6.626 \times 10^{-34} \text{ J s}^{-1}$).

2.4 Color analysis

The color of black mulberry leaves was evaluated by the direct reading of reflectance of the coordinates “L,” “a,” and “b,” using the ColorFlex EZ spectrophotometer with the Hunter color system, with “L” relative to white and black, “a” relative to red and green, and “b” relative to yellow and blue, according to Afonso Júnior and Corrêa (2003).

For better characterization, the leaves were evaluated at two different points on the adaxial and abaxial sides, and the mean for each leaf was subsequently calculated. Then, the coordinates “L,” “a,” and “b” were determined, and the values of chroma (Chr), hue angle ($^{\circ}\text{h}$), and total color difference (ΔE) were calculated according to Equations 23 to 25, respectively.

$$\text{Chr} = \left[(a^2 + b^2)^{\frac{1}{2}} \right] \quad (23)$$

$$^{\circ}\text{h} = \left[\arctang\left(\frac{b}{a}\right) \right] \quad (24)$$

$$\Delta E = \sqrt{\Delta L^2 + \Delta a^2 + \Delta b^2} \quad (25)$$

where:

Chr: chroma;

$^{\circ}\text{h}$: hue angle;

ΔE : total color difference;

L, a, and b: coordinates determined by the spectrophotometer.

The experiment was conducted in a completely randomized design with 15 replicates. The data were analyzed by analysis of variance and the Tukey's means test at a 5% significance level using the SISVAR® statistical software.

3 RESULTS AND DISCUSSION

Black mulberry (*M. nigra* L.) leaves were dried with an initial moisture content of 2.2 (d.b.) until reaching a final moisture content of 0.05 (d.b.) (Figure 1), requiring times of 23.0,

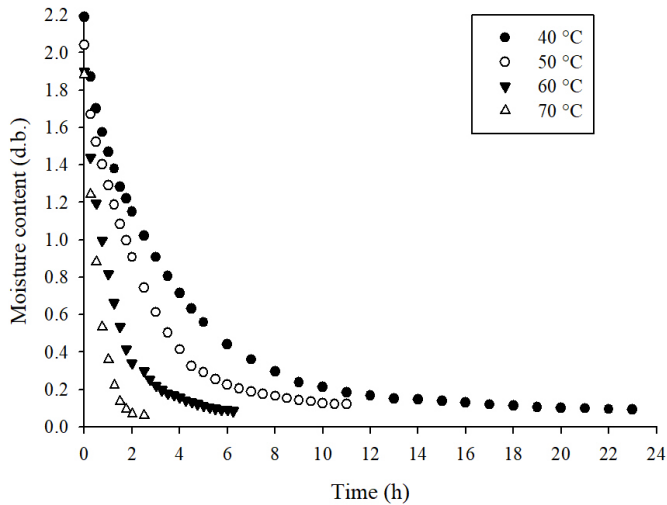


Figure 1. The moisture content of black mulberry (*Morus nigra* L.) leaves over the time of drying at different temperatures.

11.0, 6.25, and 2.5 h at temperatures of 40, 50, 60, and 70°C, respectively. It can be observed that temperature is inversely proportional to the time spent drying; that is, the higher the temperature, the shorter the drying time. The same has been described in other studies with medicinal plants (Evin, 2012; Prates et al., 2012; Radünz et al., 2011; Reis et al., 2012; Rocha et al., 2012).

Table 2 describes the values of the statistical parameters, the standard deviation of the estimate (SE), the mean relative error (P), and the coefficient of determination (R^2), which were used as criteria to evaluate the fit of the models to the experimental data obtained in the drying of black mulberry leaves at different temperatures.

All models showed coefficients of determination (R^2) higher than 0.96 (decimal); the higher the value of R^2 , the more satisfactory the representation by the model (Kashaninejad et al., 2007). However, for Madamba et al. (1996) and Prates et al. (2012), individual analysis of R^2 is not a good criterion for selecting nonlinear models to describe the drying process; so, for this reason, other parameters were taken into account to select the models in the present study.

In general, all models showed low SE values, except for the Wang & Singh model at a temperature of 40°C, so the values were adequate for the good fit of the models to the experimental data. According to Mohapatra and Rao (2005), models with mean relative error values greater than 10% are not adequate for describing the drying phenomenon. Among the models studied, only the Page and Midilli models obtained mean relative error (P) values below 10% for all temperatures.

Table 2. Statistical parameters for models used to describe the drying of black mulberry leaves.

Models	40°C			50°C		
	R ² (decimal)	SE (decimal)	P (%)	R ² (decimal)	SE (decimal)	P (%)
Page	0.9980	0.0829	6.20327	0.9958	0.1246	8.1633
Henderson & Pabis	0.9960	0.1731	12.2241	0.9925	0.0839	6.5984
Midilli	0.9988	0.0996	6.07212	0.9983	0.0433	3.2634
Diffusion Approximation	0.9974	0.2583	14.3986	0.9929	0.1402	9.3320
Two-Term Exponential	0.9957	0.2109	14.5943	0.9905	0.9991	7.9343
Logarithmic	0.9970	0.2701	14.4784	0.9932	0.1230	8.4982
Thompson	0.9957	0.2111	14.6028	0.9905	0.9992	7.9356
Newton	0.9957	0.2076	14.5944	0.9905	0.0979	7.9342
Verma	0.9974	0.2583	14.3986	0.9929	0.1402	9.3319
Wang & Singh	0.9862	1.1901	50.7602	0.9968	0.1037	6.8984
Models	60°C			70°C		
	R ² (decimal)	SE (decimal)	P (%)	R ² (decimal)	SE (decimal)	P (%)
Page	0.9962	0.1050	7.4962	0.9978	0.1929	8.8225
Henderson & Pabis	0.9962	0.0809	6.0362	0.9756	0.4226	26.5662
Midilli	0.9977	0.0638	4.5336	0.9982	0.1292	6.7342
Diffusion Approximation	0.9956	0.0829	5.9592	0.9866	0.4825	22.7561
Two-Term Exponential	0.9958	0.1057	7.5735	0.9689	0.5000	31.2063
Logarithmic	0.9963	0.0529	4.0677	0.9874	0.4686	22.3861
Thompson	0.9956	0.0692	5.1149	0.9689	0.5001	31.2122
Newton	0.9955	0.6678	5.1220	0.9689	0.4714	31.2066
Verma	0.9956	0.0829	5.9591	0.9689	0.5344	31.1995
Wang & Singh	0.9885	0.2543	15.624	0.9942	0.1701	10.1031

Table 3 presents the values of the AIC and BIC parameters, which have been used to select mathematical models to describe the post-harvest processes of plant products (Ferreira Junior et al., 2018; Gomes et al., 2018; Jorge et al., 2021; Quequeto et al., 2019).

For the AIC and BIC criteria, the Midilli model had lower values, indicating a better fit of the estimated data to the experimental conditions (Table 3). Table 3 also presents the values of the parameters of the Midilli and Page models, and those of the Page model were all significant by the t-test. Based on the above, the Page model was selected to describe the drying phenomenon of black mulberry leaves, and it was also selected by Radünz et al. (2011) when they evaluated the drying kinetics of carqueja (*Baccharis trimera*).

Figure 2 shows the adequate behavior of the data obtained in the drying of black mulberry leaves when subjected to Page's equation, demonstrating that it is a suitable model for this purpose.

Figure 3 shows the values of the effective diffusion coefficient for the different drying temperatures, and it is possible to observe that these values increase as the drying temperature increases. This is because water viscosity decreases with increasing drying temperature; the lower the viscosity, the lower the resistance of the flowing fluid, favoring the flow of the fluid through the capillaries of the leaf (Goneli et al., 2007). According to Rizvi (1995), the effective diffusion coefficient is directly related to drying air temperature but also depends on the composition of the materials, among others. The values found here ranged from 2.67×10^{-12} to 2.26×10^{-11} , being close to those cited by Madamba et al. (1996), who stated that the values found in the drying of agricultural products range from $10^{-9} \text{ m}^2 \text{ s}^{-1}$ to $10^{-11} \text{ m}^2 \text{ s}^{-1}$.

The Arrhenius equation represents the dependence of the effective diffusion coefficient of black mulberry leaves on the drying air temperature, which is illustrated in Figure 3. Such dependence has been found by several researchers (Martinazzo et al., 2007; Resende et al., 2007). For the drying of black mulberry leaves, the activation energy value found here was $65.418 \text{ kJ mol}^{-1}$,

which is consistent with the range described by Zogzas et al. (1996), who stated that the activation energy for agricultural products is between 12.7 and 110 kJ mol^{-1} .

Table 4 presents the values of enthalpy, entropy, and Gibbs free energy for the different drying conditions of black mulberry leaves. As the drying temperature increases, enthalpy and entropy decrease, while Gibbs free energy increases.

Color is an important indicator of the quality of the dried samples, such as black mulberry leaves. When dried, the leaves generally have a coiled appearance and a dark green to brownish-green color (Arslan & Ozcan, 2008). The results of the evaluation of the color of black mulberry leaves are presented in Table 5. Drying of black mulberry leaves at different temperatures altered their color, as can be observed by the significant differences ($p < 0.05$) in the parameters L , a^* , and b^* compared to fresh leaves. Significant differences were also observed in Chr and $^{\circ}\text{h}$ when different drying temperatures were used.

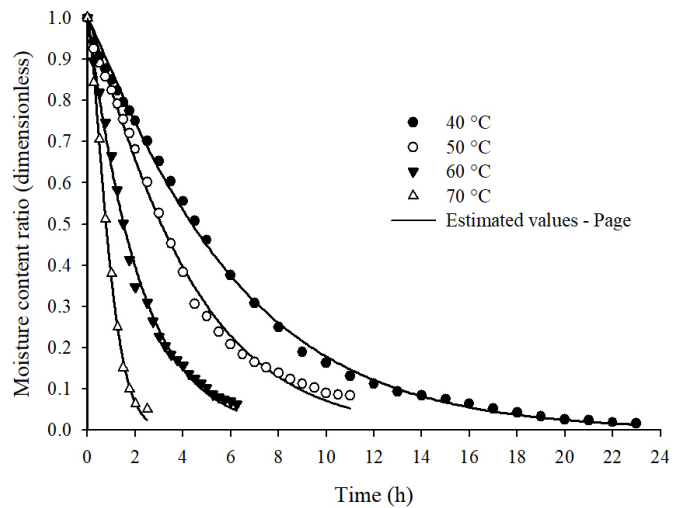


Figure 2. Moisture content ratio of black mulberry (*M. nigra* L.) leaves estimated by the Page model for drying under different temperature conditions.

Table 3. Akaike Information Criterion (AIC) and Schwarz's Bayesian Information Criterion (BIC) for the models that best fitted the drying data of black mulberry (*Morus nigra* L.) leaves and the parameters of these models.

Models	Temperatures (°C)							
	40		50		60		70	
	AIC	BIC	AIC	BIC	AIC	BIC	AIC	BIC
Midilli	-191.90	-184.42	-148.62	-142.15	-131.65	-128.55	-73.56	-70.36
Page	-177.65	-173.16	-128.50	-124.61	-125.45	-121.80	-63.97	-62.05
Models	Coefficients							
	40		50		60		70	
	a	b	a	b	a	b	a	b
Midilli	0.96335**	0.00513**	0.96277**	0.150056**	0.998201**	0.44758 ^{ns}	0.986201**	1.02687 ^{ns}
	0.000587*	1.20757**	0.00662**	1.35491 ^{ns}	0.00729**	1.13748**	0.00981 ^{ns}	1.50801**
Page	0.13628**	1.1018**	0.19131**	1.13962**	0.44922**	1.04427**	1.0005**	1.38752**

*Significant at $p \leq 0.05$ by t-test; **significant at $p \leq 0.01$ by t-test; ^{ns}not significant by t-test.

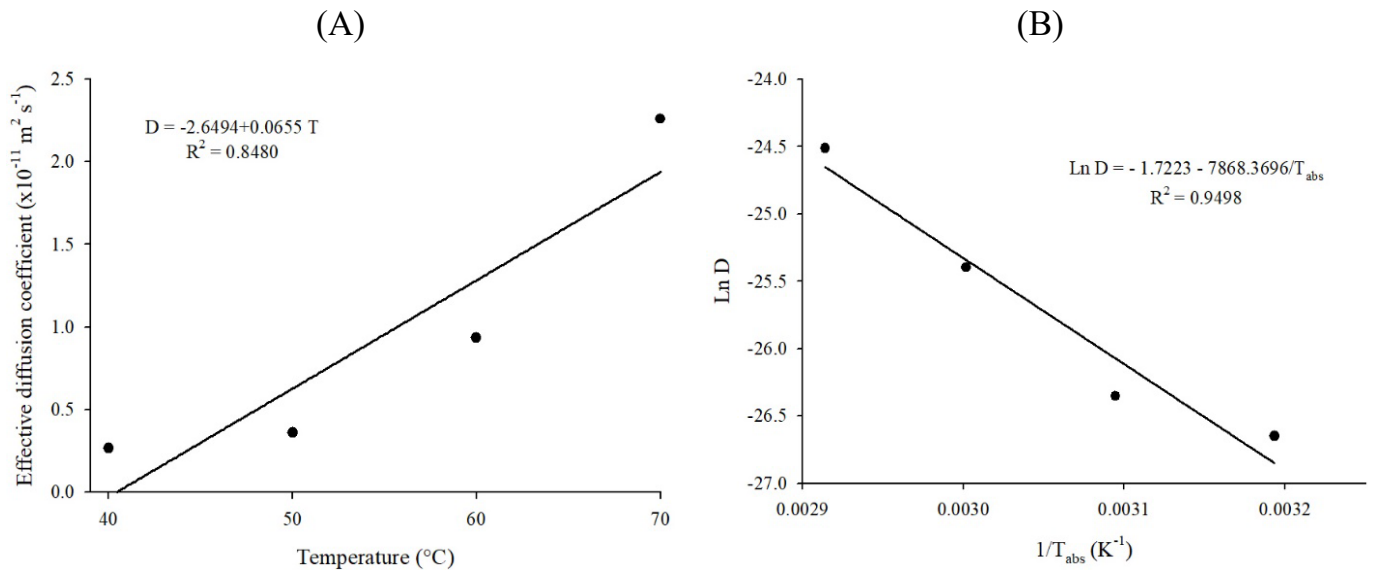


Figure 3. (A) Effective diffusion coefficient for the drying of black mulberry (*M. nigra* L.) leaves at different temperatures. (B) Arrhenius representation for the effective diffusion coefficient as a function of the inverse of the absolute temperature of the air in the drying of black mulberry (*M. nigra* L.) leaves.

Table 4. Values of enthalpy (ΔH , kJ mol⁻¹), entropy (ΔS , kJ mol⁻¹ K⁻¹), and Gibbs free energy (ΔG , kJ mol⁻¹) for different drying air conditions in the drying of black mulberry (*M. nigra* L.) leaves.

Temperatures (°C)	Thermodynamic Properties		
	ΔH	ΔS	ΔG
40	64,878.54	-231.00	137,214.73
50	64,861.32	-231.26	139,591.92
60	64,844.09	-231.51	141,971.67
70	64,826.87	-231.76	144,353.93

Table 5. Means of the parameters of the color analysis of black mulberry (*M. nigra* L.) leaves.

Treatments	L	a*	b*	a*/b*	Chr	°h	ΔE
Fresh leaves	30.38 a	-4.72a	12.92a	-0.36	13.79a	290.51a	0.00
40	27.44bc	-3.92a	15.92b	-0.25	16.44b	284.29a	4.51
50	29.05ab	-4.43a	17.11b	-0.26	17.79b	290.94a	4.53
60	29.24ab	-3.71a	17.19b	-0.21	17.63b	282.60a	4.58
70	26.26c	-0.97b	13.19a	-0.07	13.33a	340.27b	5.60

*Equal letters in the columns do not differ statistically from each other by the Tukey's test ($p > 0.05$) at a 5% probability level.

When higher temperatures were used, a reduction was observed in lightness (L), indicating a darker color. These results follow the same trend observed in studies on the drying of leaves of rosemary (Arslan & Ozcan, 2008), basil (Lima-Corrêa et al., 2017), agarwood (Alwi et al., 2022), and denaian thyme (Rahimalek & Goli, 2013), which suggest that lower temperatures are more suitable for drying the leaves. The increase in the value of the parameter a* at a temperature of 70°C indicates a loss of green color in the dry leaf, resulting from chlorophyll degradation (Arslan & Ozcan, 2012), confirming that lower temperatures are more appropriate for drying. According to Bušić et al. (2014) and Rocha et al. (2012), the degradation of

chlorophyll pigments is caused by enzymatic or non-enzymatic browning reactions (such as the Maillard reaction), which lead to the formation of dark pigments. The value of parameter b* was affected when temperatures of 40, 50, and 60 °C were used, at which the yellow color of the samples increased. The value of the a*/b* ratio is also considered one of the criteria that determine the quality of dry products, and lower values are desirable (Arslan & Ozcan, 2008; Doymaz et al., 2006). According to this ratio, temperatures of 40 and 50°C are the most indicated for drying black mulberry leaves.

The Chr value differed significantly ($p < 0.05$) for leaves dried at 40, 50, and 60°C when compared to the control and to those dried at 70°C, indicating higher purity of tone and intensity at intermediate temperatures, as reported by Gasparin et al. (2014). The hue angle (°h) provides a measure of how the color is perceived by the observer. An angle of 0° represents full red color, 90° full yellow color, 180° full green color, and 270° full blue color (McGuire, 1992). As can be seen in Table 5, only the sample dried at 70°C differed ($p < 0.05$) from the fresh sample and the others. In the evaluation of total color difference (ΔE), an increase was observed with the increase in drying temperature, which is consistent with the results observed for the parameters L, a*, and b*. However, for all samples, the values are within the range of 3.0–6.0, which is classified as easily distinguishable in human perception according to DIN 6174 (DIN, 1979).

4 CONCLUSION

Page's mathematical model was the one that best described the drying of black mulberry leaves according to the selection criteria. The results showed that temperature directly affects the drying time and color of the leaves. The effective diffusion coefficient tended to increase with increasing temperature, and the activation energy represented by the Arrhenius equation showed that, in drying processes, the lower the activation energy, the

greater the water diffusivity in the product. Enthalpy and entropy decrease while Gibbs free energy increases with increasing drying temperature. Drying black mulberry leaves at different temperatures altered their color. Considering the evaluations, temperatures of 40 and 50°C are the most indicated for drying of black mulberry leaves.

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