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Thermodynamic properties and drying kinetics of *Bauhinia forficata* Link leaves

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ABSTRACT

The aim of this study was to determine the effective diffusion coefficient and the thermodynamic properties of *Bauhinia forficata* Link leaves, considering two forms of thickness measurements and to describe the process by fitting mathematical models. The leaves were collected, taken to the laboratory and prepared to start the drying process in which four temperatures (40, 50, 60 and 70 °C) were applied. After the drying process, the effective diffusion coefficient was determined through the theory of diffusion in liquid, allowing to obtain the values of the activation energy, enthalpy, entropy and Gibbs free energy. The description of the drying process was performed by setting the thirteen mathematical models used to represent constant drying of agricultural products. The Valcam model was selected to represent the drying kinetics *B. forficata* Link. Increased temperature promotes: decreasing enthalpy and entropy; increasing Gibbs free energy and effective diffusion coefficient. The effective diffusion coefficient is higher when the rib thickness is considered; thus, it is recommended to standardize and/or specify the points of measurement of leaf thickness.

Palavras-chave:
plantas medicinais
coeficiente de difusão efetivo
temperatura

Propriedades termodinâmicas e cinética de secagem de folhas de Bauhinia forficata Link

RESUMO

Objetivou-se, neste trabalho, determinar o coeficiente de difusão efetivo e as propriedades termodinâmicas das folhas de *Bauhinia forficata* Link, considerando-se duas formas de medição de espessura, bem como descrever o processo de secagem por meio do ajuste de modelos matemáticos. As folhas foram coletadas, levadas ao laboratório e preparadas para dar início ao processo de secagem em quatro temperaturas (40, 50, 60 e 70 °C). Após a secagem determinou-se o coeficiente de difusão efetivo por meio da teoria da difusão líquida permitindo a obtenção dos valores da energia de ativação, entalpia, entropia e energia livre de Gibbs. Já a descrição do processo de secagem foi realizada por meio do ajuste de treze modelos matemáticos constantemente utilizados para representação de secagem de produtos agrícolas. O modelo de Valcam foi selecionado para representar a cinética de secagem de folhas de *B. forficata* Link. O aumento da temperatura promove: decréscimo de entalpia e entropia; aumento da energia livre de Gibbs e do coeficiente de difusão efetivo. O coeficiente de difusão efetivo é maior quando se considera a espessura da nervura, recomendando-se a padronização e/ou especificação dos pontos de medição da espessura da folha.

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Introduction

Medicinal plants have been used in the treatment of diseases since the past generations, which characterizes the millennial use of these products, combined with the popular knowledge and experience (Feijó et al., 2012). Among the more than 300 known species from the *Bauhinia* genus (Lusa & Bona, 2009), the species *Bauhinia forficata* Link, popularly known in Brazil as 'pata-de-vaca', is constantly used in popular medicine, standing out in the treatment of diabetes mellitus (Rodrigues et al., 2012).

As most agricultural products, some medicinal plants need to undergo a drying process, but each agricultural product has a different behavior during this process, since, besides the characteristics of the drying air, its physical properties and chemical composition also have great influence. Because of that, various authors (Martinazzo et al., 2007; Prates et al., 2012; Rocha et al., 2012) use the technique of statistical modeling to predict such behavior.

Along the drying process, it is interesting not only to describe the drying kinetics, but also to observe the thermodynamic properties. They provide important information on the water properties and also on the energy necessary in the process (Corrêa et al., 2010).

This study aimed to determine the effective diffusion coefficient and the thermodynamic properties of leaves of 'pata-de-vaca' (*Bauhinia forficata* Link) considering two thicknesses, as well as describe the drying process through the fit of mathematical models.

Material and Methods

The leaves of 'pata-de-vaca' (*B. forficata* Link) were collected in October 2015 in the Medicinal Plants Garden of the Faculty of Agricultural Sciences - FCA, of the Federal University of Grande Dourados.

The initial and equilibrium moisture contents of the samples were determined using the gravimetric method proposed by ASABE (2010) and forced-air oven at 103 ± 1 °C, for 24 h, in four replicates.

The initial and equilibrium moisture contents, for each temperature, were 1.61 ± 0.08 and 0.05 ± 0.008; 1.86 ± 0.09 and 0.04 ± 0.002; 1.81 ± 0.07 and 0.04 ± 0.008; 1.78 ± 0.07 and 0.04 ± 0.002, for the temperatures of 40, 50, 60 and 70 °C, respectively. The equilibrium moisture content was considered when there was no variation in the mass of the product in three consecutive weighings in intervals of 2 h.

The experiment was conducted using an experimental dryer with four trays equipped with a system that precisely controls the air flow and drying air temperature. The experimental dryer has, as heating source, a set of electrical resistances and a Sirocco fan, with 1-hp motor. Temperature is controlled through a universal process controller working with Proportional-Integral-Derivative (PID) control, while the air flow is selected by a frequency inverter connected to the fan motor. Drying air speed was monitored with the aid of a rotating vane anemometer and maintained around 0.4 m s^{-1}.

The drying temperatures adopted in the dryer were: 40, 50, 60 and 70 °C, under controlled conditions, with relative humidity values of 20.55, 12.74, 9.23 and 4.96%, respectively, obtained through basic principles of psychrometry. At all temperatures, drying was interrupted when the leaves reached 0.11 ± 0.005 decimal (b.s), according to Goneli et al. (2014).

The moisture content ratio of *B. forficata* Link leaves at all temperatures was determined through Eq. 1.

$$RX = \frac{U - U_e}{U_i - U_e} \tag{1}$$

where:

RX - moisture content ratio of the product, dimensionless;
U - moisture content at a certain time, decimal (b.s);
Ue - equilibrium moisture content, decimal (b.s); and,
Ui - initial moisture content, decimal (b.s).

The data of moisture content ratio of *B. forficata* Link leaves were fitted to the thirteen mathematical models presented in Eqs. 2 to 14:

- Diffusion approximation

$$RX = a \cdot \exp(-k \cdot t) + (1 - a)\exp(-k \cdot b \cdot t) \tag{2}$$

- Two terms

$$RX = a \cdot \exp(-k_0 \cdot t) + b \cdot \exp(-k_1 \cdot t) \tag{3}$$

- Two-term exponential

$$RX = a \cdot \exp(-k \cdot t) + (1 - a)\exp(-k \cdot a \cdot t) \tag{4}$$

- Modified Henderson-Pabis

$$RX = a \cdot \exp(-k \cdot t) + b \cdot \exp(-k_0 \cdot t) + c \cdot \exp(-k_1 \cdot t) \tag{5}$$

- Henderson-Pabis

$$RX = a \cdot \exp(-k \cdot t) \tag{6}$$

- Logarithmic

$$RX = a \cdot \exp(-k \cdot t) + c \tag{7}$$

- Midilli

$$RX = a \cdot \exp(-k \cdot t^n) + b \cdot t \tag{8}$$

- Newton

$$RX = \exp(-k \cdot t) \tag{9}$$

- Page

$$RX = \exp(-k \cdot t^n) \tag{10}$$

- Thompson

$$RX = \exp\left\{\left[-a - \left(-a^2 + 4 \cdot b \cdot t\right)^{0.5}\right]\left(2 \cdot b\right)^{-1}\right\} \quad (11)$$

- Valcam

$$RX = a + b \cdot t + c \cdot t^{1.5} + d \cdot t^2 \quad (12)$$

- Verma

$$RX = -a \cdot \exp\left(-k \cdot t\right) + \left(1 - a\right)\exp\left(-k_1 \cdot t\right) \quad (13)$$

- Wang-Singh

$$RX = 1 + \left(a \cdot t\right) + \left(b \cdot t^2\right) \quad (14)$$

where:

t - time of drying, h;
k, k_0, k_1 - constants of drying, h^{-1}; and,
a, b, c, d, n - coefficients of the models.

The effective diffusion coefficient at the various drying temperatures was determined using Eq. 15, based on the theory of liquid diffusion, which considers the geometric form of the product as close to a flat plate with approximation of eight terms.

$$RX = \frac{U - U_e}{U_i - U_e} = \frac{8}{\pi^2}\sum_{n=0}^{\infty}\frac{1}{\left(2n+1\right)^2}\exp\left[\left(2n+1\right)^2 \pi^2 D_i \left(\frac{\theta}{4L}\right)^2\right] \quad (15)$$

where:

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

The thickness (L) of the *B. forficata* Link leaves was measured with a digital micrometer with resolution of 0.001 mm, using 40 fresh leaves, which were the replicates, 10 for each drying temperature. The thickness of the leaves was determined in two ways: four points on each side of the midrib, totaling eight points on the entire leaf area (Figure 1A) and fifteen points on the entire leaf area; eight outside the ribs and seven on the ribs (Figure 1B). In both cases, the contraction of the thickness was disregarded.

Then, the mean thickness of the *B. forficata* Link leaves was calculated considering the points outside the rib (OSR), whose mean value was 0.250 ± 0.07 mm, and those outside and on the ribs (ONR), whose mean value was 0.583 ± 0.12 mm. The higher standard deviation of this mean results from the large variation in the thickness of the ribs.

The Arrhenius equation, described in Eq. 16, was used to evaluate the behavior of the effective diffusion coefficient in relation to the different temperatures applied during the drying process for both situations: OSR and ONR.

$$D_i = D_0 \exp\left(\frac{E_a}{RT_a}\right) \quad (16)$$

where:

D_0 - 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.

After selecting the model to represent the effective diffusivity, an equality hypothesis test of the models was performed using the method of Regazzi (2003). This method allows the analysis of equivalence between the models and aims to establish a single equation to describe the studied phenomenon.

The parameters of the linear model used to describe the effect of the thickness of *B. forficata* Link leaves on the effective

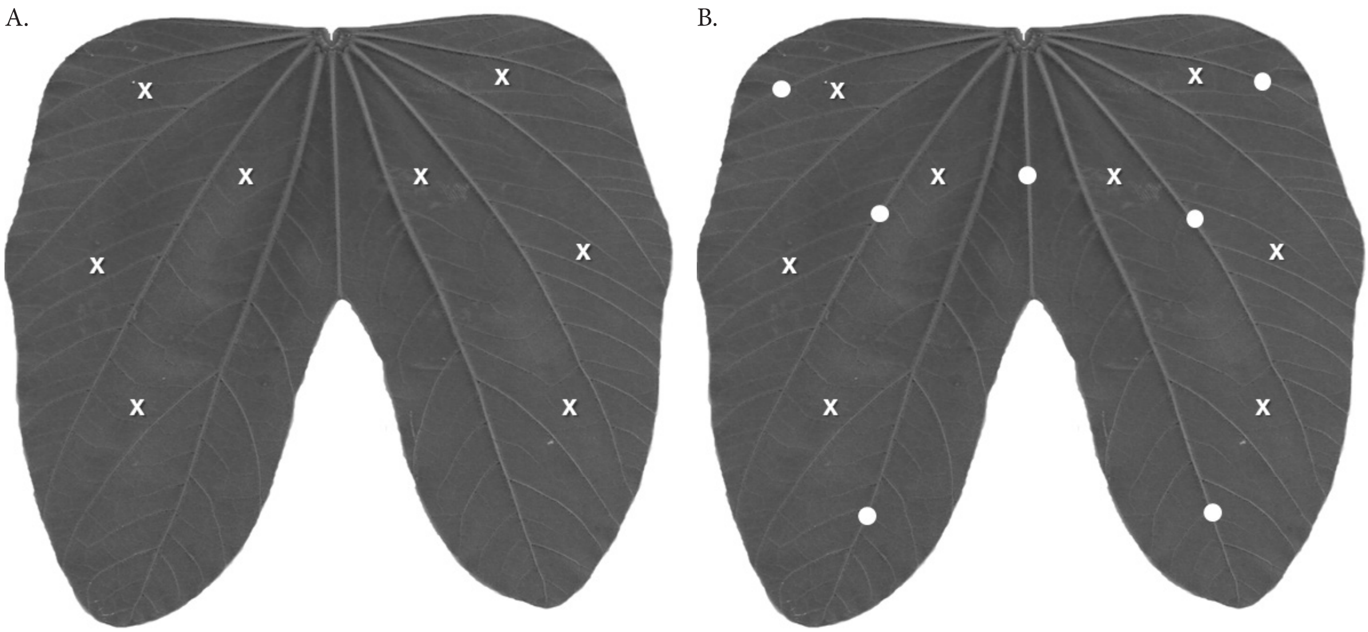

Figure 1. Points of thickness measurements on *B. forficata* Link leaves outside the rib (OSR) (A), and outside and on the ribs (ONR) (B)

diffusivity (a_i and b_i) were compared to verify their equality. The formulated hypotheses were:

H_o - $a_1 = a_2$ and $b_1 = b_2$; and,
H_a - there is at least one inequality between the parameters.

According to Regazzi (2003), to obtain the forms of the complete model of the equations with restrictions, dummy variables (D) were considered: $D_i = 1$ if the observation y_{ij} belongs to the group i, and $D_i = 0$ in the opposite case.

The decision rule was based on the chi-square test (χ^2), according to Eq. 17.

$$\chi^2_{calculado} = -N \ln\left(\frac{RSS_\Omega}{RSS_{wi}}\right) \quad (17)$$

where:

N - number of observations;
RSS_Ω - residual sum of squares of the complete model; and,
RSS_{Wi} - residual sum of squares of the restricted parameter space.

The tabulated value of (χ^2) is a function of the significance level α and the number of degrees of freedom, described in Eq. 18.

$$\nu = P_\Omega - P_{Wi} \quad (18)$$

where:

ν - degrees of freedom of the model;
P_Ω - number of parameters of the complete model; and,
P_{Wi} - number of parameters of the model with restriction.

The diffusion models were compared using the two previously mentioned values of thickness (0.250 and 0.583 mm).

The thermodynamic properties associated with the drying process were determined according to the method proposed by Jideani & Mpotokwana (2009), presented in Eqs. 19, 20 and 21, respectively, specific enthalpy, specific entropy and Gibbs free energy.

$$h = E_a - RT_a \quad (19)$$

$$s = R\left(\ln D_0 - \ln\frac{k_B}{h_p} - \ln T_a\right) \quad (20)$$

$$G = h - T_a s \quad (21)$$

where:

h - specific enthalpy, J mol^{-1};
s - specific entropy, J mol^{-1} K^{-1};
G - Gibbs free energy, J mol^{-1};
K_B - constant of Boltzmann, 1.38 x 10^{-23} J K^{-1}; and,
h_p - constant of Planck, 6.626 x 10^{-34} J s^{-1}.

The degree of fit of each model was analyzed using the magnitudes of the determination coefficient (R^2), relative mean error (P) and standard deviation of the estimate (SE), calculated according to Eqs. 22 and 23.

$$P = \frac{100}{N}\sum\frac{|RX_{obs} - RX_{est}|}{RX_{obs}} \quad (22)$$

$$SE = \sqrt{\sum\left(RX_{obs} - RX_{est}\right)^2 / v} \quad (23)$$

where:

RX_{obs} - moisture content ratio observed experimentally;
RX_{est} - moisture content ratio estimated by the model; and,
v - degrees of freedom of the model.

The experimental data from the drying kinetics of *B. forficata* Link leaves were analyzed and subjected to nonlinear regression analysis, through the Gauss-Newton method, using the software Statistic 8.0.

Results and Discussion

Only the Wang-Singh model showed determination coefficients (R^2) lower than 0.95, which, according to Kashaninejad et al. (2007), is the minimum value to obtain a satisfactory representation of models of the drying process (Table 1). However, the determination coefficient is not a correct parameter for this type of characterization when it is individually analyzed (Madamba et al., 1996). According to Siqueira et al. (2012), the lower the SE values, the better the fit of the models to the experimental data. In this case, it is possible to claim that the capacity of a model to precisely describe certain physical process is inversely proportional to the value of the standard deviation of the mean (Draper & Smith, 1998).

The acceptable values of P must be lower than 10% (Aguerre et al., 1989; Mohapatra & Rao, 2005). Therefore, the models Modified Henderson-Pabis, Logarithmic, Valcam and Verma are the only ones to meet this requirement, besides exhibiting low SE and high R^2 values (Table 1), at all drying air temperatures.

The model selected to represent the *B. forficata* Link drying curves was the Valcam model, for presenting a simplified form and lower number of coefficients, thus being easily used in drying simulation processes.

For *B. forficata* Link leaves to achieve the moisture content of approximately 0.11 ± 0.01 decimal b.s., 1.80, 1.72, 0.87 and 0.45 h of drying were necessary for the temperatures of 40, 50, 60 and 70 ºC, respectively (Figure 2).

The values of the effective diffusion coefficient calculated without considering the thickness of the leaf ribs (0.250 mm) and the effective diffusion coefficient calculated considering the points outside and on the leaf ribs (0.583 mm) increased as the drying air temperature increased (Table 2). Martinazzo et al. (2007) and Prates et al. (2012) observed the same behavior for lemon grass and 'fruta-de-lobo' leaves.

The effective diffusion coefficient serves as an indication of the speed of water outlet. With the increase in drying air temperature and consequent increment in the difference of partial pressure of water vapor between the drying air and

Table 1. Standard deviation of the estimate (SE), relative mean error (P) and determination coefficient (R^2) for the thirteen models analyzed during the drying of *B. forficata* Link leaves, under various temperature conditions (°C)

Models	40 °C			50 °C			60 °C			70 °C		
	SE	P	R²	SE	P	R²	SE	P	R²	SE	P	R²
(2)	0.0470	6.019	0.965	0.0208	10.602	0.994	0.0142	7.354	0.998	0.0183	8.217	0.997
(3)	0.0302	3.714	0.986	0.0214	10.601	0.994	0.0164	5.740	0.998	0.0190	8.210	0.998
(4)	0.0458	6.020	0.965	0.0432	10.180	0.974	0.0149	5.784	0.998	0.0206	14.678	0.996
(5)	0.0035	0.700	0.999	0.0079	6.7000	0.999	0.0165	7.739	0.998	0.0139	6.025	0.999
(6)	0.0312	6.715	0.984	0.0330	8.330	0.985	0.0149	5.739	0.998	0.0187	11.704	0.997
(7)	0.0301	3.429	0.986	0.0339	8.301	0.985	0.0145	7.983	0.998	0.0177	7.679	0.998
(8)	0.0141	4.218	0.997	0.0299	16.224	0.989	0.0153	6.751	0.998	0.0164	10.159	0.998
(9)	0.0447	6.020	0.965	0.0421	10.181	0.975	0.0144	5.784	0.998	0.0178	11.363	0.997
(10)	0.0355	9.617	0.979	0.0322	13.697	0.986	0.0142	5.234	0.998	0.0189	11.294	0.997
(11)	0.0420	9.954	0.971	0.0383	14.509	0.980	0.0149	5.789	0.998	0.0189	11.369	0.997
(12)	0.0257	4.862	0.990	0.0299	6.098	0.990	0.0120	6.566	0.999	0.0155	6.779	0.998
(13)	0.0471	6.000	0.970	0.0444	9.782	0.975	0.0142	6.149	0.998	0.0198	9.506	0.997
(14)	0.0577	10.180	0.950	0.0830	46.639	0.906	0.0390	28.012	0.985	0.0424	21.021	0.984

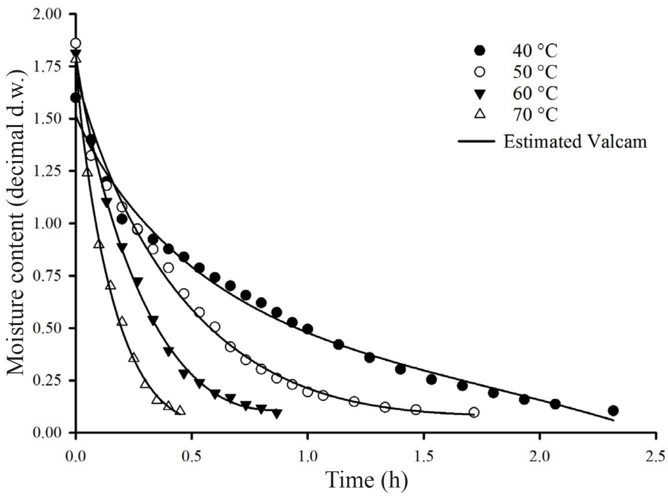

Figure 2. Moisture content obtained experimentally and estimated by the Valcam model, at the different drying temperatures of *B. forficata* Link leaves

the product, the effective diffusion coefficient becomes higher. Such behavior can be related to the viscosity of the water, which decreases with the temperature; oscillations in the behavior of this property leads to alterations in water diffusion, favoring the movement of water through the capillaries of the leaves (Goneli et al., 2014).

In addition, the coefficients "a", "b", "c" and "d" of the Valcam model showed high degree of significance for all drying conditions (Table 2).

The values of OSR effective diffusion coefficient varied from 6.4236 x 10^{-12} to 3.9491 x 10^{-11} $m^2 s^{-1}$, a behavior similar to that observed in lemon grass leaves (Martinazzo et al., 2007) and basil leaves (Reis et al., 2012). Both authors obtained effective diffusion coefficients ranging from 10^{-12} to 10^{-11} $m^2 s^{-1}$. On the other hand, the ONR effective diffusion coefficients remained in the range between 1.7829 x 10^{-11} and 1.0961 x 10^{-10} $m^2 s^{-1}$.

Considering that the effective diffusion coefficient varies according to temperature, concentration and vibration frequency of water molecules and volume of the product, the highest values observed for the ONR effective diffusion coefficient are due to the higher value of thickness, since the higher the thickness and, consequently, the volume of the leaf, the larger is the vibration field of the water molecules.

According to Zogzas et al. (1996), the values of the effective diffusion coefficient vary from 10^{-11} to 10^{-9} $m^2 s^{-1}$ for food products, and the effective diffusion coefficient calculated for the *B. forficata* Link leaves using ONR data is within this variation range.

According to the model identity test, the tabulated values of chi-square (5.991) were lower than the calculated values of chi-square (35.963), for the effective diffusion coefficient of *B. forficata* Link leaves. Thus, the H_o formulated hypothesis is rejected, denoting that the linear models applied to represent this phenomenon differ statistically, which indicates that the use of only one single model to represent the variation in the effective diffusion coefficient as a function of temperature is not representative.

It is possible to claim that, when leaf ribs are considered in the calculation of effective diffusivity, there is a significant variation of these values. Thus, it is recommended to standardize leaf thickness and/or to specify the points to be used to obtain the leaf thickness.

The representation of Arrhenius, which indicates the dependence of the effective diffusion coefficient in relation to the temperature, showed linear behavior for *B. forficata* Link leaves (OSR and ONR). Eqs. 24 and 25 show the coefficient of the fitted equation for the effective diffusion coefficients of the leaves (Eq. 16).

$$D_{OSR} = 0.0046 \exp\left(\frac{52.956}{RTa}\right) \tag{24}$$

Table 2. Estimated values of the parameters of the Valcam model and effective diffusion coefficient considering (ONR) and disregarding (OSR) the thickness of the ribs of *B. forficata* Link leaves

Temperature (°C)	Coefficients				D ($m^2 s^{-1}$)	
	a	b	c	d	OSR	ONR
40	0.93522**	-1.58374**	1.41794**	-0.44049*	6.4236×10^{-12}	1.7829×10^{-11}
50	0.92648**	-2.75860**	2.64556**	-0.71753**	1.3869×10^{-11}	3.8493×10^{-11}
60	0.99534**	-4.60885**	5.09702**	-1.42667**	2.2194×10^{-11}	6.1599×10^{-11}
70	0.99253**	-8.60268**	13.88675**	-6.33544*	3.9491×10^{-11}	1.0961×10^{-10}

*Significant at 0.05; **Significant at 0.10

Table 3. Thermodynamic properties of the drying process of *B. forficata* Link leaves: specific enthalpy (h), specific entropy (s) and Gibbs free energy (G)

Analyzed variables	40 °C		50 °C		60 °C		70 °C	
	OSR	ONR	OSR	ONR	OSR	ONR	OSR	ONR
h (kJ mol^{-1})	50353.2	50353.2	50270.1	50270.1	50186.9	50186.9	50103.8	50103.8
s (kJ mol^{-1} K^{-1})	-290.0	-275.9	-290.3	-276.2	-290.5	-276.4	-290.8	-276.7
G (kJ mol^{-1})	141188.9	136780.0	144090.8	139541.1	146995.3	142304.8	149902	145071.0

OSR - points outside the rib, ONR - points on the rib

$$D_{ONR} = 0.0128 \exp\left(\frac{52.956}{RTa}\right) \qquad (25)$$

The activation energy was equal to 52.95 kJ mol^{-1}, regardless of the leaf thickness. This value differs from that found by Rocha et al. (2012), in the drying of thyme (77.16 kJ mol^{-1}) and Goneli et al. (2014), for aroeira leaves (74.96 kJ mol^{-1}). This difference can be related to the chemical composition of the leaf, which makes even more important the standardization of thickness.

The activation energy has already been defined as the fundamental energy to break the barrier found by water molecules when they migrate to the surface of the product along the drying process (Sharma & Prasad, 2004). In general, products with high moisture content will have lower activation energy, since the higher the activation energy, the lower the speed at which the water will be removed (Siqueira et al., 2012).

Based on the thermodynamic properties (Table 3), the specific enthalpy decreases with the increase of temperature, i.e., the higher the temperature, the lower the demand of energy necessary for the drying process. The specific entropy showed the same behavior, since it is related to the disorder in the state of water molecules (Goneli et al., 2010).

Gibbs free energy increase with the increment in temperature, as observed by Martins et al. (2015). This behavior is expected, because it establishes the non-spontaneity or spontaneity of the sorption process (McMinn et al., 2005), that is, it is necessary to introduce energy from outside of the product for the process to occur. In this case, external energy is heated air.

The positive values of Gibbs free energy evidence that the drying process for *B. forficata* Link leaves does not occur spontaneously and needs a source of energy to make the sorption sites more available.

Conclusions

1. Among the 13 tested models, the Valcam, Modified Henderson-Pabis, Logarithmic and Verma models can be used to represent the drying kinetics of *B. forficata* Link leaves.

2. The effective diffusion coefficient increases with the increment in drying air temperature.

3. The activation energy was equal to 52.95 kJ mol^{-1}, obtained through the Arrhenius equation, which establishes the dependence of the diffusivity in relation to the temperature.

4. Specific enthalpy and specific entropy decrease with the increment of temperature, while Gibbs free energy increases.

5. It is recommended to standardize and/or specify the points of measurement of leaf thickness.

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