

Fitting of mathematical models in the drying of araticum (*Annona crassiflora*) seeds

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Abstract

Araticum (*Annona crassiflora*) seeds have relevant characteristics for industrialization due to the contents of phytosterols, carotenoids, phenolic compounds, fatty acids, and organic acids, among other components responsible for biological properties. The objective of this study was to fit different mathematical models to the experimental data, use criteria to choose the best model, evaluate the effective diffusion coefficient, and obtain the activation energy and thermodynamic properties of araticum seeds at different drying temperatures. Seeds of araticum fruits were dried at temperatures of 40, 50, 60, and 70°C. The drying time of araticum seed decreases with increasing temperature. Among the best models fitted, the Midilli model was recommended to predict the drying curves of araticum seeds under different drying conditions. The linear model represented the effective diffusion coefficient as a function of the drying temperature. Enthalpy and entropy tend to reduce, and Gibbs free energy increases as the drying temperature increases.

Keywords: mathematical modeling; Midilli; Akaike information criterion; bayesian information criterion.

Practical Application: To add value to araticum seeds, increasing their consumption and reducing waste.

1. Introduction

Araticum (*Annona crassiflora*) is a plant native to Tropical America and is found in several states in Brazil. Depending on the region, it is called 'pinha', 'ata', 'marolo', 'condessa', 'bruto', and 'cabeça-de-negro', among others (Fachinello & Nachtigal, 2010). It has high potential to be economically exploited because it is also an undomesticated, cross-pollinated plant (Pimenta et al., 2014). Araticum seeds represent around 10–19% of the whole fruit that are commonly discarded (Braga Filho et al., 2014). They are obovoid, flattened, and 13.4–22.7 mm long, 9.0–13.6 mm wide, and 6.4–11.2 thick. The characteristics of integument are glabrous, light brown, opaque, with a smooth texture, and a bony consistency. The tegmen has fibrous layers that spread within the endosperm, resulting in ruminations. Their surface is rough and irregular, and they have conspicuous boundaries arranged around the hilum and the micropyle. The hilum is basal and presents an irregular shape (between circular and oval). The endosperm is thick, abundant, and whitish-yellow, while the embryo is basal, crude, hyaline, and gelatinous (about 2 mm long) (Pimenta, 2014). Regarding their composition, studies have shown that araticum seeds are sources of phytosterols, carotenoids, phenolic compounds, fatty acids, and organic acids, among other components responsible for their biological properties (e.g., neuro- and cardio-protection, nematicidal activity, antioxidant activity, anti-inflammatory, antiproliferative,

and antimycobacterial) (Arruda & Pastore, 2019). It is also a promising source of oil (28.84–34.58%) containing the desired characteristics for the food and pharmaceutical industries, including significant concentrations of monounsaturated fatty acids (Egydio & Santos, 2011; Luzia & Jorge, 2013). Thus, it is important to transform this by-product into a value-added raw material. The drying of seeds is the initial step in this process.

The drying operation is extremely important in the technology that allows the production of high-quality food products, enabling the preservation of physical and chemical properties and reducing the moisture content to safe levels for storage so that the product can be used in periods when there is no production of the fruit (Resende et al., 2018). The drying kinetics are represented by statistical modeling under different processing conditions (Almeida et al., 2022). Mathematical modeling is used to satisfactorily represent the drying kinetics of various products. The quality of fit of the models to the experimental data can be evaluated with different statistical indices. However, some parameters adopted have limitations, making it necessary to adopt additional criteria for the selection of models to reinforce and endorse decision-making (Gomes et al., 2018). In this context, the Akaike Information Criterion (AIC) and the Bayesian Information Criterion (BIC) consist of evaluating the models according to the principle of parsimony since the number of parameters in the models varies.

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Therefore, the objective of this study was to fit different mathematical models to the experimental data, use criteria to choose the best model, evaluate the effective diffusion coefficient, and obtain the activation energy and thermodynamic properties of araticum seeds at different drying temperatures.

2. Material and Methods

2.1. Obtaining of araticum fruits

Araticum (*Annona crassiflora*) fruits were obtained from a farm located in the rural area of the municipality of Montividiu, State of Goiás, Brazil, from the 2017 season of the Midwest region of Goiás. The fruits were harvested directly from the plant, separated according to the degree of maturity in sacks, and sent to the Laboratory of Post-Harvest of Plant Products of the Federal Institute of Goiás — Rio Verde Campus.

As soon as the fruits reached an adequate degree of maturity, the peduncles were removed, washed in running water, and sanitized by immersion in 150 ppm L⁻¹ sodium hypochlorite for 10 min, then rinsed to remove residual chlorine. The fruits were subjected to pulping to separate the seeds.

2.2. Drying study

The seeds were dried at temperatures of 40, 50, 60, and 70°C with an internal relative humidity of 34.0, 18.1, 11.4, and 7.0%, respectively. For the determination of the drying curves and fits of the models, an initial moisture content of 0.37 (dry basis, d.b.) and a final moisture content of 0.04±0.02 (d.b.) were respectively established for the mesocarp, and the moisture contents of the product were determined in an oven at 105±3°C, until reaching constant mass in three replicates (AOAC, 2012).

The seeds were homogeneously distributed and dried in trays without perforations, containing 100 g of product in a completely randomized design in three replicates. To obtain hygroscopic equilibrium, three repetitions containing 10 g were maintained under the drying conditions indicated above and periodically weighed until their mass remained constant. The equilibrium moisture content was 0.019, 0.01, 0.008, and 0.005 (d.b.) for temperatures of 40, 50, 60, and 70°C, respectively.

2.3. Mathematical drying modeling

The moisture content ratios of the seeds during drying were determined using the Equation 1:

$$RX = \frac{X^* - X_c^*}{X_i^* - X_c^*} \quad (1)$$

where:

RX: the moisture content ratio, dimensionless;

X^{*}: the moisture content of the product (d.b.);

X_i^{*}: the initial moisture content of the product (d.b.);

X_c^{*}: the equilibrium moisture content of the product (d.b.).

For drying representation, the empirical mathematical models frequently used were fitted, as described in Table 1.

The mathematical models were fitted to the experimental drying data by nonlinear regression analysis through the Gauss-Newton method using the statistical program. The values reported in the literature for the modeling of other agricultural products were adopted as a criterion for the initial approximations of the coefficients of the models. The degree of fit for each

Table 1. Mathematical models used to predict the drying of araticum seeds.

Model designation	Model	Referencee
$RX = 1 + a \cdot t + b \cdot t^2$	Wang & Sing	(Equation 2)
$RX = a \cdot \exp(-k \cdot t) + (1 - a) \exp(-k_1 \cdot t)$	Verma	(Equation 3)
$RX = \exp\left(-a - (a^2 + 4 \cdot b \cdot t)^{0.5}\right) 2 \cdot b$	Thompson	(Equation 4)
$RX = \exp(-k \cdot t^n)$	Page	(Equation 5)
$RX = \exp(-k \cdot t)$	Newton	(Equation 6)
$RX = a \cdot \exp(-k \cdot t^n) + b \cdot t$	Midilli	(Equation 7)
$RX = a \cdot \exp(-k \cdot t) + c$	Logarithmic	(Equation 8)
$RX = a \cdot \exp(-k \cdot t)$	Henderson & Pabis	(Equation 9)
$RX = a \cdot \exp(-k \cdot t) + (1 - a) \exp(-k \cdot a \cdot t)$	Two-Term Exponential	(Equation 10)
$RX = a \cdot \exp(-k_0 \cdot t) + b \cdot \exp(-k_1 \cdot t)$	Two Terms	(Equation 11)
$RX = a \cdot \exp(-k \cdot t) + (1 - a) \cdot \exp(-k \cdot b \cdot t)$	Approximation of diffusion	(Equation 12)

t: the drying time; h; k, k₀, k₁: the drying constants, h⁻¹; a, b, c, n: the coefficients of the models; RX: the moisture content ratio, dimensionless.

drying temperature was determined considering the significance of the regression coefficients by the t-test, adopting the level of 5% significance, the magnitude of the coefficient of determination (R^2), the values of mean relative error (P) (Equation 13) and mean estimated error (SE) (Equation 14), the χ^2 test (Equation 15) at the 5% significance level, and the confidence interval at 95% ($p < 0.05$), according to Mohapatra and Rao (2005).

$$P = \frac{100}{n} \sum \frac{Y - \hat{Y}}{Y} \quad (13)$$

$$SE = \sqrt{\frac{\sum (Y - \hat{Y})^2}{DF}} \quad (14)$$

$$\chi^2 = \frac{\sum (Y - \hat{Y})^2}{DF} \quad (15)$$

where:

P: the mean relative error (%);

n: the number of experimental observations;

Y: the value observed experimentally;

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

SE: the mean estimated error;

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

To select a single model to describe the drying process of the araticum seeds under each condition, the models that obtained the best fits were subjected to the Akaike Information Criterion (AIC) and the Schwarz's Bayesian Information Criterion (BIC). Lower values of AIC and BIC 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 16 and 17.

$$AIC = -2\log L + 2p \quad (16)$$

$$BIC = -2\log L + p \ln(N - r) \quad (17)$$

Where:

p: the number of parameters of the model;

N: the total number of observations;

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

L: the maximum likelihood.

2.4. Effective diffusion coefficient and activation energy

The liquid diffusion of the seeds was described using the model with the analytical solution for the geometric shape of an infinite cylinder with an eight-term approximation (Equation 18):

$$RX = \frac{X - X_e}{X_i - X_e} = \sum_{n=1}^{\infty} \frac{4}{\lambda_n^2} \exp\left[-\frac{\lambda_n^2 \cdot D \cdot t}{4} \cdot \left(\frac{2}{r}\right)^2\right] \quad (18)$$

Where:

RX: the moisture content ratio (dimensionless);

λ_n : the roots of the zero-order Bessel equation;

n: the number of terms;

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

t: the drying time (s);

r: the equivalent radius (m).

The equivalent radius of the seeds was determined by the Equation 19:

$$r = \sqrt[3]{\frac{3 \cdot V_s}{4 \cdot \pi}} \quad (19)$$

Where:

V_s : the average volume of the seeds (m^3).

The volume of each seed (V_s) was obtained by measuring the three orthogonal axes (length, width, and thickness) in 15 units at the end of drying, using a digital caliper with a resolution of 0.01 mm, according to the Equation 20:

$$V_s = \frac{\pi \cdot A \cdot B \cdot C}{6} \quad (20)$$

Where:

A: the length (m);

B: the width (m);

C: the thickness (m).

The mean values obtained for length, width, and thickness were 0.0161, 0.010, and 0.0069 m, respectively. The relationship between the effective diffusion coefficient and the increase in drying air temperature was described using the Arrhenius equation (Equation 21).

$$D = D_o \cdot \exp\left(\frac{-E_a}{R \cdot T_{abs}}\right) \quad (21)$$

Where:

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

D_0 : is the pre-exponential factor ($\text{m}^2 \text{s}^{-1}$);

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

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

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

2.5. Thermodynamic drying properties

The thermodynamic properties of the drying process of araticum seeds were obtained by the method described by Jideani and Mpotokwana (2009) (Equations 22, 23 and 24):

$$\Delta H = E_a - R \cdot T \quad (22)$$

$$\Delta S = R \cdot \left(\ln k - \ln \frac{k_B}{h_p} \right) - \ln T_{\text{abs}} \quad (23)$$

$$\Delta G = \Delta H - T_{\text{abs}} \cdot \Delta S \quad (24)$$

Where:

ΔH : the enthalpy (J mol^{-1});

ΔS : the entropy (J mol^{-1});

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

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

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

3. Results and Discussion

The drying times required for araticum seeds to reach approximately 0.04 (d.b.) were 21, 15, 13, and 12 h at temperatures of 40, 50, 60, and 70°C, respectively, and the average initial moisture content was 0.37 (d.b.) and 27.06 (w.b.) (Figure 1). It can be noted that the time spent is inversely proportional to the drying temperature; that is, the higher the temperature, the shorter the time in which the product is subjected to drying, and this behavior has been found by several researchers (Freitas et al., 2018; Martins et al., 2020; Quequeto et al., 2019; Silva et al., 2018; Smaniotta et al., 2017).

Table 2 shows that all models for both drying conditions had low values of mean estimated error (SE), which indicates a good fit of the model. According to Draper and Smith (1998), the mean estimated error (SE) indicates the ability of a model to faithfully describe a given physical process, and the lower its magnitude, the better the quality of fit to the observed data.

All models, except Wang & Singh, Verma, Newton, and Henderson & Pabis, had coefficients of determination (R^2) greater than 0.99 under all drying conditions (Table 2). The Midilli model was the one that obtained the highest coefficients of determination. However, Mohapatra and Rao (2005) report that

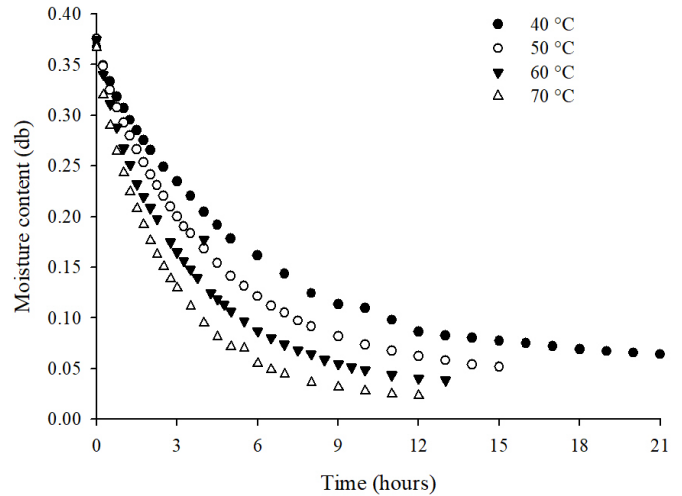


Figure 1. Moisture content of araticum seeds during drying at different temperatures.

the use of the coefficient of determination as the only evaluation criterion for selecting drying models does not constitute a good parameter to represent the drying phenomenon.

Regarding the mean relative error (P), values lower than 10% were found for drying conditions at temperatures of 40, 50, 60, and 70°C, for the Thompson, Page, Midilli, Logarithmic, Two-Term Exponential, Two Terms, and Approximation of Diffusion models. Mohapatra and Rao (2005) indicate that the mean relative error determines a good fit of the model to the drying conditions whether being used for the recommendation or not of a model. The values of the mean relative error reflect the deviation of the observed curve from the curve estimated by the model (Kashaninejad et al., 2007). Therefore, in this case, the deviation can be considered acceptable.

Also in Table 2, regarding the values of the χ^2 test obtained for the different models fitted to the drying curves of araticum seeds, it can be observed that all models had low values, and the lower this value, the better the fit to the conditions (Gunhan et al., 2005). Among the models that had the best statistical parameters previously calculated and presented in Table 2 (Thompson, Page, Midilli, Logarithmic, Two-Term Exponential, Two Terms, and Approximation of Diffusion), the Akaike Information Criterion (AIC) and Schwarz's Bayesian Information Criterion (BIC) (Table 3) were also considered additional parameters to select the best model.

Considering the lowest values of AIC and BIC (Table 3), the Midilli model showed the best fit to the experimental data. Thus, this model was selected to represent the drying kinetics of araticum seeds. Table 3 shows the coefficients of the Midilli model fitted to the experimental data used to predict the drying curves for each condition. It can be observed that all coefficients, for all temperatures studied, had a high level of significance ($p < 0.01$) and can be satisfactorily used.

Figure 2A shows the moisture content ratio obtained experimentally and estimated by the Midilli model for araticum seeds during the drying period at different temperatures.

Table 2. Statistical parameters for the models fitted to the drying of araticum seeds.

Models	40°C				50°C			
	SE	P	R ²	χ ²	SE	P	R ²	χ ²
Wang & Sing	0.0551	16.231	0.9652	0.003035	0.0552	15.837	0.9573	0.003045
Verma	0.0098	2.051	0.9989	0.000096	0.0365	12.064	0.9819	0.001334
Thompson	0.0127	4.315	0.9981	0.000162	0.0079	2.242	0.9991	0.000062
Page	0.0179	6.357	0.9963	0.000320	0.0107	3.704	0.9984	0.000114
Newton	0.0426	15.219	0.9784	0.001818	0.0353	12.064	0.9819	0.001245
Midilli	0.0062	1.483	0.9984	0.000038	0.0035	0.584	0.9998	0.000012
Logarithmic	0.0074	1.490	0.9994	0.000055	0.0070	0.938	0.9904	0.000049
Henderson & Pabis	0.0339	11.360	0.9994	0.001147	0.0070	8.350	0.9904	0.000049
Two-Term Exponential	0.0200	7.201	0.9954	0.000398	0.0125	4.123	0.9978	0.000155
Two Terms	0.0076	1.478	0.9994	0.000057	0.0063	0.968	0.9995	0.000040
Approximation of Diffusion	0.0098	2.051	0.9989	0.000096	0.0086	1.778	0.9990	0.000074
	60°C				70°C			
	SE	P	R ²	χ ²	SE	P	R ²	χ ²
Wang & Sing	0.0697	23.920	0.9288	0.004861	0.0850	42.784	0.9093	0.007231
Verma	0.0417	14.204	0.9753	0.001741	0.0304	16.700	0.9889	0.000922
Thompson	0.0233	3.620	0.9921	0.000542	0.0103	4.069	0.9987	0.000106
Page	0.0222	4.104	0.9928	0.000491	0.0080	5.157	0.9992	0.000065
Newton	0.0404	14.203	0.9753	0.001633	0.0291	16.700	0.9889	0.000848
Midilli	0.0227	3.151	0.9930	0.000514	0.0062	2.391	0.9996	0.000038
Logarithmic	0.0255	2.808	0.9908	0.000649	0.0115	2.759	0.9984	0.000131
Henderson & Pabis	0.0320	10.016	0.9850	0.001024	0.0211	11.906	0.9944	0.000447
Two-Term Exponential	0.0232	5.817	0.9921	0.000540	0.0107	6.397	0.9986	0.000114
Two Terms	0.0233	4.978	0.9926	0.000541	0.0100	4.063	0.9988	0.000100
Approximation of Diffusion	0.0229	5.012	0.9926	0.000524	0.0109	6.434	0.9980	0.000119

Table 3. Akaike Information Criterion (AIC) and Schwarz's Bayesian Information Criterion (BIC) for the models that fitted best to the drying data of araticum seeds and the parameters of the selected model.

Model	Temperature (°C)							
	40		50		60		70	
	AIC	BIC	AIC	BIC	AIC	BIC	AIC	BIC
Thompson	-150.547	-143.065	-208.416	-204.114	-150.608	-146.118	-160.244	-156.470
Page	-157.542	-153.240	-189.649	-185.347	-153.857	-149.368	-173.055	-169.281
Midilli	-221.628	-214.458	-256.344	-249.174	-168.702	-161.219	-184.875	-178.585
Logarithmic	-211.078	-205.342	-214.863	-209.127	-143.753	-137.767	-153.800	-148.768
Two-Term Exponential	-150.769	-146.467	-179.935	-175.633	-136.308	-131.818	-158.242	-154.468
Two Terms	-209.118	-201.948	-220.264	-213.094	-165.200	-157.718	-159.941	-153.651
Approximation of Diffusion	-193.836	-188.101	-202.174	-196.438	-161.705	-155.719	-156.243	-151.213
	Coefficients							
	40		50		60		70	
Midilli	a=0.9924**		a=0.9932**		a=0.9825**		a=0.9911**	
	k=0.1818**		k=0.2489**		k=0.4127**		k=0.4202**	
	b=0.0041**		b=0.0038**		b=0.0049**		b=0.0018**	
	n=0.9316**		n=0.9024**		n=0.9025**		n=0.8823**	

**Significant at $p \leq 0.01$ by t-test.

Based on the correspondence between the experimental values and those estimated by the model, there was a satisfactory fit of the model to the data obtained during the drying of araticum seeds under all conditions, as confirmed by the AIC and BIC. Recently, several researchers have updated, based on the AIC and BIC criteria to select with greater accuracy, the best mathematical model to represent the drying curves

for different agricultural products (Bastos et al., 2019; Gomes et al., 2018; Pinheiro et al., 2020; Quequeto et al., 2019; Souza et al., 2019).

The Midilli model has been recommended with satisfaction by several researchers to predict the drying process of different agricultural products: leaves of *Solanum paniculatum* L. (Martins et al., 2020), pulp of yellow mombin (Freitas et al., 2018),

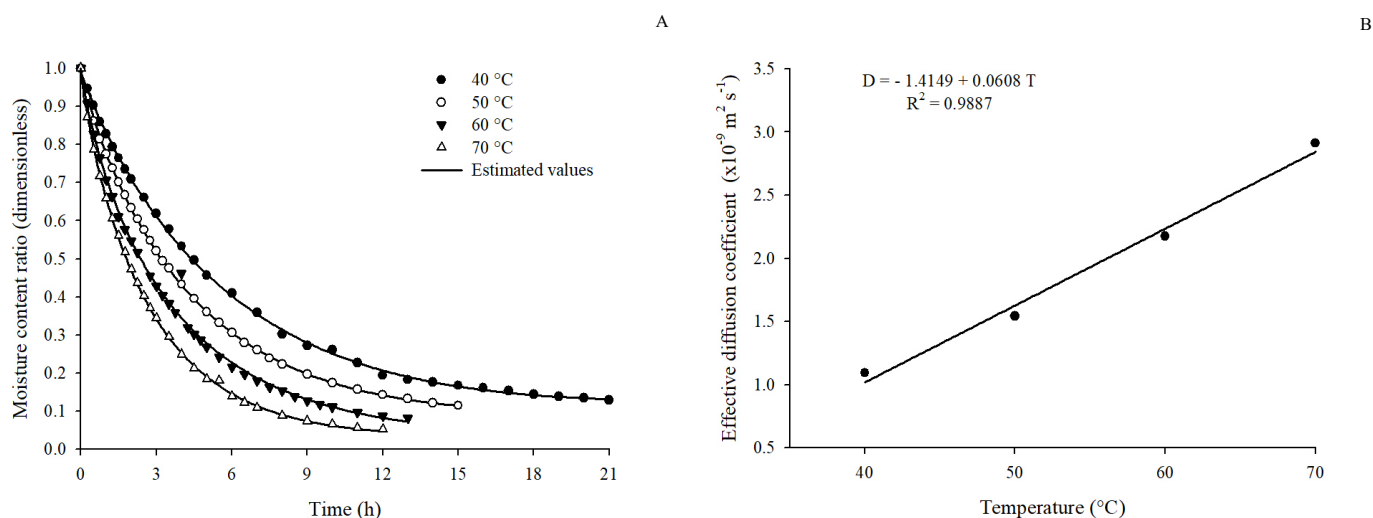


Figure 2. Moisture content ratio obtained experimentally and estimated by the Midilli model (A) and effective diffusion coefficient (B) for the drying of araticum seeds during the period of drying at different temperatures.

seeds of ‘Cabacinha’ pepper (Silva et al., 2018), and grains of sunflower (Smaniotto et al., 2017). The diffusion coefficients obtained were 1.094×10^{-9} , 1.543×10^{-9} , 2.173×10^{-9} , and $2.911 \times 10^{-9} \text{ m}^2 \text{ s}^{-1}$ for temperatures of 40, 50, 60, and 70°C, respectively (Figure 2B). It is verified that the effective diffusion coefficient increases with the increase in drying air temperature, as verified by several researchers (Baptestini et al., 2015; Morais et al., 2013; Quequeto et al., 2019; Resende et al., 2018). According to Madamba et al. (1996), the effective diffusion coefficient for the drying of agricultural products ranges from 10^{-9} to $10^{-11} \text{ m}^2 \text{ s}^{-1}$. Therefore, the calculated values for araticum seeds are within the established range.

The activation energy for the drying process of araticum seeds was $29.31 \text{ kJ mol}^{-1}$ for the temperature range studied. In the drying process, the lower the activation energy, the greater the diffusivity of water in the product (Morais et al., 2013). Activation energy is a barrier that must be overcome so that the diffusion process can be triggered in the product (Kashaninejad et al., 2007). In the literature, it is possible to find activation energy values for several agricultural products: $46.83 \text{ kJ mol}^{-1}$ for niger seeds (Silva et al., 2017); $30.76 \text{ kJ mol}^{-1}$ for sunflower grains (Smaniotto et al., 2017); and $38.94 \text{ kJ mol}^{-1}$ for adzuki beans (Resende et al., 2010). According to Zogzas et al. (1996), the activation energy for agricultural products ranges from 12.7 to 110 kJ mol^{-1} . Thus, the value obtained in the present study is within this range.

In the evaluation of thermodynamic properties, it can be noted that both enthalpy and entropy tend to decrease when temperature increases (Table 4). According to Oliveira et al. (2010), lower values of enthalpy indicate lower energy needed to remove the water bound to the product during drying. The present study showed lower values of enthalpy for higher drying temperatures, which indicates that less energy is required for drying to occur at higher temperatures.

According to Goneli et al. (2010), entropy is a thermodynamic property associated with the degree of disorder, being a

Table 4. Values of enthalpy (ΔH , J mol^{-1}), entropy (ΔS , $\text{J mol}^{-1} \text{ K}^{-1}$), and Gibbs free energy (ΔG , J mol^{-1}) for different drying air conditions in the drying of araticum seeds.

Temperature (°C)	Thermodynamic properties		
	ΔH	ΔS	ΔG
40	25,653.21	-148.22	72,069.45
50	25,536.43	-148.49	73,519.36
60	25,419.65	-148.74	74,971.84
70	25,302.87	-148.98	76,426.82

state function, and its values increase during a natural process in an isolated system. The fact that entropy values decrease dramatically when temperature increases is due to the greater excitation of water molecules within the product. Entropy values were equal to -148.22, -148.49, -148.74, and $-148.98 \text{ J mol}^{-1} \text{ K}^{-1}$ for temperatures of 40, 50, 60, and 70°C, respectively. Negative values of entropy were attributed to the existence of chemical adsorption and/or structural changes in the adsorbent (Moreira et al., 2008).

Nkolo Mezeè et al. (2008) report that Gibbs free energy is attributed to the work required to make sorption sites available. With the increase in temperature, Gibbs free energy increased, and its values were positive. According to Corrèa et al. (2010), this indicates that drying under the conditions of the present study was not spontaneous. The values found were 72,069.45, 73,519.36, 74,971.84, and $76,426.82 \text{ J mol}^{-1}$ for temperatures of 40, 50, 60, and 70°C, respectively.

4. Conclusion

Drying araticum seeds contributes to their preservation throughout storage and might collaborate with an increase of its consumption. Understanding drying conditions is essential, and the present study demonstrated that the drying time of araticum seeds decreases with increasing temperature. Among the best models fitted, the Midilli model was recommended to predict

the drying curves of araticum seeds under different drying conditions. The linear model represented the effective diffusion coefficient as a function of the drying temperature. Enthalpy and entropy tend to reduce, and Gibbs free energy increases as the drying temperature increases.

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