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Multivariate analysis in mathematical model selection to describe *Croton urucurana* Baill drying kinetics

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Abstract

The *Croton urucurana* Baill species is known in Brazil as "sangra d'água" and is popular due to its medicinal properties. For better processing of herbal medicines, it is essential that efficient drying and storage techniques are developed and that compounds are preserved. Therefore, this study aimed to select models through multivariate cluster analysis applying Akaike (AIC) and Bayesian information criteria (BIC) to describe *Croton urucurana* leaves drying kinetics at different temperatures (40-70 °C). The initial moisture content in *Croton urucurana* leaves was 1.791, 1.841, 2.196 and 2.144 kg water kg dry matter^{-1}, and 8.25, 7.75, 4.25 and 2 hours were required to reach hygroscopic equilibrium, with a final moisture content of 0.134, 0.105, 0.065 and 0.0601 kg water kg dry matter^{-1}, at 40, 50, 60 and 70 °C, respectively. The models with the greatest similarity to the experimental data were Diffusion Approximation; Cavalcanti Mata; Two-term; Two-term Exponential; Modified Henderson & Pabis; Logarithmic; Midilli; Page and Verma. The multivariate cluster technique associated with AIC and BIC criteria during model selection is a great applicability tool to help decision-making when evaluating the drying plant leaves. The Cavalcanti Mata mathematical model was selected to represent the drying kinetics.

Keywords: mathematical modeling; AIC and BIC; plant products; post-harvest.

Practical Application: Contribute to estimate the drying process of the *Croton urucurana* B. leaves in different temperatures.

1 Introduction

The Brazilian biological diversity has aroused the international scientific community's interest, as we search for new potential compounds to be the basis in new drug synthesis (Souza & Felfili, 2006). The *Croton urucurana* B. species is popularly known in Portuguese as "sangra d'água", and it is a tall plant that has red-colored sap and is predominantly found in riparian forests or floodplains (Rao et al., 2007). Its sap contains a wide variety of phytochemicals that have anti-hemorrhagic, anti-inflammatory, antiseptic and healing properties, as well as potential antifungal and entomological actions (Soldera et al., 2010; Carvalho et al., 2014).

The main feedstocks for obtaining plant products are the medicinal and aromatic plant aerial parts, which contain the largest phytochemical amounts, and secondary metabolism products constituting the plant's defense system (Koche et al., 2010; Silva et al., 2015). After harvesting, the water in plant tissues keeps the metabolic and enzymatic mechanism active, which may lead to modifications in the bioactive compounds' effectiveness present in medicinal plants (Maciel et al., 2002; Morais et al., 2013; Silva et al., 2015). Natural product processing requires efficient techniques for drying and storage of the plant biomass produced and for its chemical properties to be fully and effectively used (Tabaldi et al., 2012; Martins et al., 2015).

Drying is the most recommended process to ensure post-harvest quality and stability. It is defined as a single operation to remove water, or any other liquid contained in a solid, to a minimum moisture content level in which plant products can be stored for long periods, without losses or alterations in the characteristics obtained at harvest (Matias et al., 2010; Gadelha-Neto et al., 2013). Drying can also be defined, according to Brooker et al. (1992), as a process that involves the simultaneous transfer of energy in the form of heat and mass between the product and the drying air.

Drying system studies, dimensioning and commercial application viability determination can be conducted through mathematical simulations, satisfactorily representing product moisture loss during the drying process (Martinazzo et al., 2010). The physical description of mathematical models has great relevance in the drying equipment dimensioning and improvement, providing information and time estimates required to reach the ideal product moisture content at different drying curve points (Vilela & Artur, 2008; Andrade et al., 2010).

In some situations, more than one model can describe the same phenomenon, since there is not only one methodology to follow, and a good model should have a balance between adjustment quality and complexity, which is usually measured by the number of parameters in the model (Mazerolle, 2004; Emiliano,

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2013). When fitted to the same dataset, the adjustment quality evaluators are used to compare different nonlinear regression models and indicate which best represent the studied situation (Emiliano, 2013; Dias, 2014; Varanis et al., 2016).

Using a large evaluator number may transform the model choice into a complex activity, and since each evaluator has a specific characteristic, the same model may exhibit high or low performance depending on the evaluator (Silva et al., 2011).

Clustering by multivariate classification methods allows for grouping of models with similar results for all evaluators considered and indicates a model that best fits all or most of the studied conditions. The magnitude of the coefficient of determination, mean relative error, mean estimated error and chi-square test have been used (Goneli et al., 2016; Smaniotto et al., 2017; Guimarães et al., 2018; Resende et al., 2018; Xavier et al., 2018; Beigi & Ahmadi, 2019; Quequeto et al., 2019; Cavalcante et al., 2020; Aydar, 2021; Silva et al., 2021). Since these methods have some limitations, it is necessary to adopt other criteria to increase the accuracy in the model selection and further support decision-making. The Akaike Information Criterion (AIC) and Bayesian Information Criterion (BIC) consist of evaluating the models according to the parsimony principle, penalizing models according to the variation in the number of parameters (Gomes et al., 2018).

Thereby, the objective of this study was to select, through multivariate cluster analysis applying Akaike (AIC) and Bayesian information criteria (BIC), models to describe the drying kinetics of *Croton urucurana* B. leaves at temperatures of 40, 50, 60 and 70 °C.

2 Material and methods

2.1 Obtaining plant material

The experiment was carried out at Pós-Colheita de Produtos Vegetais Laboratory in Instituto Federal de Educação, Ciência e Tecnologia Goiano - Campus Rio Verde. The first steps were leaves removal and plant material selection. Raw material consisted of leaves detached from the middle third of *Croton urucurana* B. plants, collected between 7:30 and 8:00 a.m. in January 2017 from the Santo Antônio da Barra – GO, Brazil (17°33’05.7”S and 50°36’19.5”W). The exsiccates were registered at the Instituto Federal Goiano Herbarium – Campus Rio Verde, under the number 602.

2.2 Drying study

Drying was completed in a forced air circulation oven, Marconi MA35 (Piracicaba, São Paulo, Brazil), at air temperatures of 40, 50, 60 and 70 °C, and air circulation of 2.0 ± 0.2 m/s, where the average relative humidity values were 33.74, 20.01, 12.37 and 7.95%, respectively. The trays containing the plant product with an approximately 0.15-cm thin layer were removed and weighed periodically. To construct the drying curves, the material was weighed every 15 minutes until hygroscopic equilibrium and constant mass. In the final moisture content determination, the material was placed in metal capsules and dried in a forced air circulation oven at 103 °C for 24 hours (American Society of Agricultural Engineers, 2000). The external environment temperature and relative humidity were monitored by a datalogger, *LogBox-DA Novus* model (Canoas, Rio Grande do Sul, Brazil), during the drying period at each temperature, and the relative humidity inside the dryer was obtained by the basic psychrometry principles with the GRAPSI program (Melo et al., 2004).

2.3 Mathematical modeling of drying

Moisture content ratios in *Croton urucurana* B. leaves during drying were determined using the following expression (Equation 1):

$$MR = \frac{X - X_e}{X_i - X_e} \tag{1}$$

where MR: Moisture Ratio (dimensionless); X: product moisture content (kg water kg dry matter^{-1}); X_i: product initial moisture content (kg water kg dry matter^{-1}); and X_e: product equilibrium moisture content (kg water kg dry matter^{-1}).

Fourteen mathematical models (Table 1) were tested to represent the *Croton urucurana* B. leaves drying process. The mathematical models were set for the experimental drying data through nonlinear regression analysis using the Gauss-Newton method. The mathematical model parameters were estimated using the STATISTICA 7.0® program (Equations 2-15).

For the multivariate analysis, the models estimated the moisture content ratio values. The cluster optimal number selection was made according to hierarchical clustering methods through the level of change in the dendrograms. The best model cluster determination was performed according to the clusters optimal number defined by the Duda & Hart (1973) index, based on the similarities of the experimental values in relation to the values obtained by the tested models. The cluster analysis was used to identify the models whose estimated theorical data were closest to the experimental data, and the dissimilarity measure adopted was the Euclidean distance. The multivariate analysis was carried out using the *FactoMineR* package (Lê et al., 2008), present in R® *software*.

The magnitude of the coefficient of determination (R^2), relative error (P%), estimated mean error (SE), and chi-square test (χ^2) were used to compare and select the mathematical models that best represented the *Croton urucurana* B. leaves drying. Akaike (AIC) and Bayesian information criteria (BIC), which use the parsimony principle in the best model selection, were also used (Akaike, 1974; Schwarz 1978). All of these selection criteria are presented in Equations 16-20:

$$P = \frac{100}{N} \Sigma \frac{|Y - \hat{Y}|}{Y} \tag{16}$$

$$SE = \sqrt{\Sigma \frac{|Y - \hat{Y}|^2}{DF}} \tag{17}$$

$$\chi^2 = \frac{\Sigma \left(Y - \hat{Y}\right)^2}{DF} \tag{18}$$

$$AIC = -2 \, . \, \log L + 2p \tag{19}$$

Table 1. Mathematical models used to predict the plant product drying.

Model	Model description	
Diffusion Approximation (Kassem, 1998)	$MR = a\exp(-k\,t)+(1-a)\exp(-k\,b\,t)$	(2)
Cavalcanti Mata (Cavalcanti Mata et al., 2006)	$MR = a_1\exp\left(-k_1\left[t\right]^{n_1}\right)+a_2\exp\left(-k_1\left[t\right]^{n_2}\right)+a_3$	(3)
Two-term (Henderson, 1974)	$MR = a\exp(-k_0\,t)+b\exp(-k_1\,t)$	(4)
Two-term Exponential (Sharaf-Eldeen et al., 1980)	$MR = a\exp(-k\,t)+(1-a)\exp(-k\,a\,t)$	(5)
Modified Henderson & Pabis (Karathanos, 1999)	$MR = a\exp(-k_0\,t)+b\exp(-k_1\,t)+c\exp(-k_2\,t)$	(6)
Henderson & Pabis (Henderson & Pabis, 1961)	$MR = a\exp(-k\,t)$	(7)
Logarithmic (Yagcioglu et al., 1999)	$MR = a\exp(-k\,t)+c$	(8)
Logistic (Chandra & Singh, 1995)	$MR = \frac{a_0}{1+a\exp(k\,t)}$	(9)
Midilli (Midilli et al., 2002)	$MR = a\exp\left(-k\,t^n\right)+b\,t$	(10)
Newton (Lewis, 1921)	$MR = \exp(-k\,t)$	(11)
Page (Page, 1949)	$MR = \exp\left(-k\,t^n\right)$	(12)
Modified Page (Overhults et al., 1973)	$MR = \exp(-k\,t)^n$	(13)
Verma (Verma et al., 1985)	$MR = a\exp(-k\,t)+(1-a)\exp(-g\,t)$	(14)
Wang & Sing (Wang & Singh, 1978)	$MR = 1+a\,t+b\,t^2$	(15)

t = drying time, h; k, k_o, k_1, k_2; g = drying constants in h^{-1}; a, a_0, a_1, a_2, a_3, b, c, n, n_1, n_2 = model parameters.

$$BIC = -2\,.\,\log L + p\,.\,\ln(N - r) \tag{20}$$

where, R^2: coefficient of determination; p: parameter number; Y: observed values; $\hat{Y}$: model estimated values; DF: residual degrees of freedom (number of observations minus the number of parameters of the models); L: Maximum likelihood; N: number of experimental observations; r: rank of the matrix X (incidence matrix for fixed effects).

Data were submitted to ANOVA and when significant effects were observed, the data were compared with each other by Tukey test at 5% probability ($p \le 0.05$).

3 Results and discussion

3.1 Moisture content during drying

The initial moisture content of *Croton urucurana* B. leaves was 1.791, 1.841, 2.196 and 2.144 kg water kg dry matter^{-1} for the temperatures of 40, 50, 60 and 70 °C, respectively (Figure 1). To reach hygroscopic equilibrium, the times required were 8.25, 7.75, 4.25 and 2 hours with 0.134, 0.105, 0.065 and 0.0601 kg water kg dry matter^{-1} moisture content at temperatures of 40, 50, 60 and 70 °C, respectively. An increase in the drying air temperature generates a vapor pressure gradient between air and the leaves surface, provoking a reduction in *Croton urucurana* B. leaves drying time as the drying temperature increases (Castiglioni et al., 2013; Soares et al., 2016).

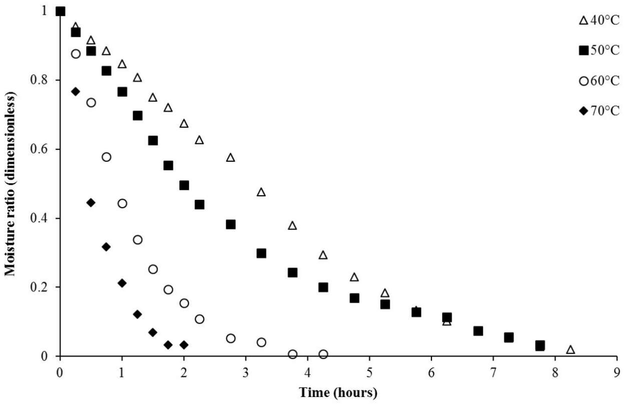

Figure 1. *Croton urucurana* B. leaves moisture ratio during drying in a forced air circulation oven at temperatures of 40, 50, 60 and 70 °C as a function of time.

A reduction in drying time has also been observed in studies evaluating an increase in drying air temperature on water removal time from *Cymbopogon citratus* (Martinazzo et al., 2010; Gomes et al., 2017), *Ziziphus joazeiro* (Sousa et al., 2015) and *Genipa americana* (Silva et al., 2015) leaves. At high temperatures, this process occurs rapidly, reducing the material's surface water amount and causing the products to adapt to the drying conditions. In the decreasing rate period, the moisture would be proportional to the instantaneous difference between the product moisture content and the equilibrium moisture content (Akpinar et al., 2003).

3.2 Mathematical modeling

The experimental moisture content ratio values of *Croton urucurana* B. leaves during drying and the estimated values by the models converged to the formation of five, eight, six and eight groups for 40, 50, 60 and 70 °C, respectively (Figure 2).

From the fourteen models proposed to describe the *Croton urucurana* B. leaves drying kinetics, only nine showed greater similarity to the experimental data and presented shorter

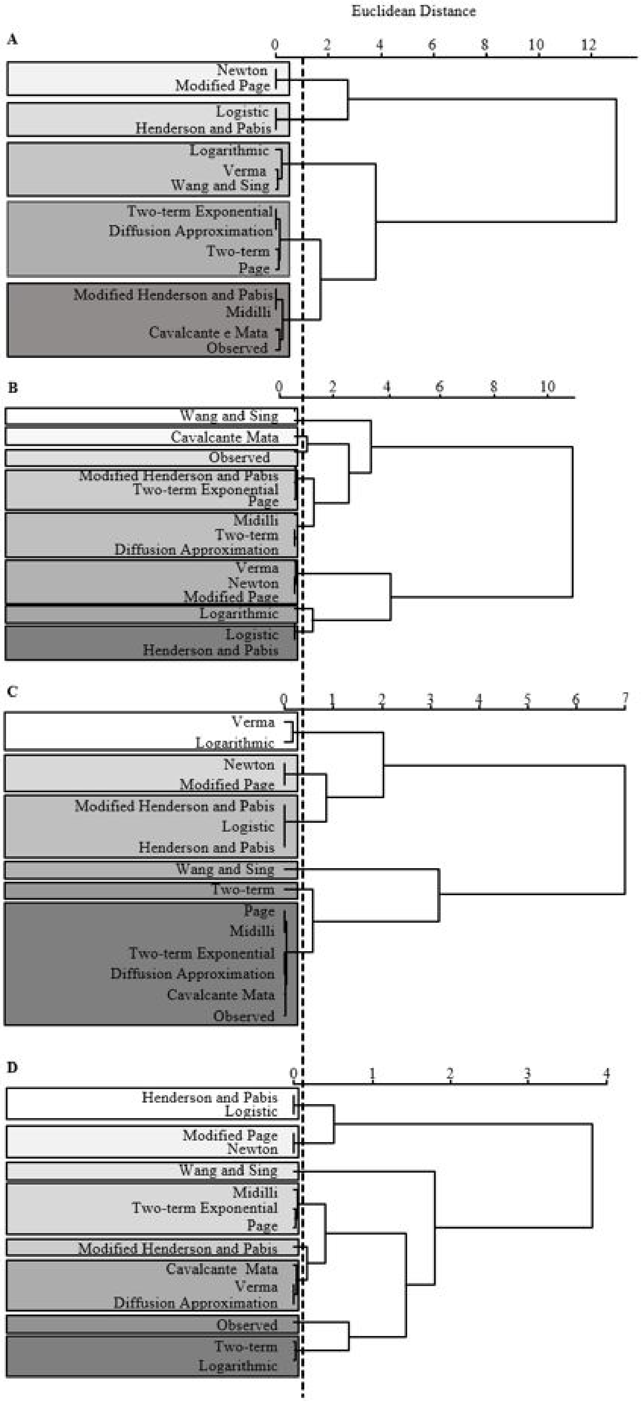

Figure 2. Model clustering fitting to the observed and estimated data describing the *Croton urucurana* B. leaves drying kinetics in a forced air circulation oven at A) 40 °C, B) 50 °C, C) 60 °C and D) 70 °C.

distances between the experimental values and the mathematical estimated values.

At 40 °C (Figure 2A), the Modified Henderson & Pabis, Midilli and Cavalcanti Mata models, belonging to group 5, had the highest similarity to the observed values. A value of 0.045 was observed with the Cavalcanti Mata model, which was the shortest distance between estimated and experimental data.

Drying at 50 °C the groups (Figure 2B) in which the Cavalcanti Mata, Modified Henderson & Pabis, Two-term Exponential and Page models were the most similar to the observed values. The shortest Euclidean distance between estimated and experimental data, 0.035, was found with the Cavalcanti Mata model.

Higher model numbers that showed greater similarity to the experimental values were found for 60 and 70 °C. When the drying air temperature is increased, the relative humidity is lower and water removal from agricultural products occurs faster (Silva et al., 2017). These changes influence the number of points sampled and lead to a rise in model number with similarity to the sampled values, that is, the greater the number of points collected during the experiment, the greater the similarity with the values presented by the models.

For the 60 °C drying air temperature (Figure 2C) the Page, Midilli, Two-term Exponential, Diffusion Approximation and Cavalcanti Mata models were the most similar to the observed values. The shortest distance between the models and the experimental data was observed for the Cavalcanti Mata model, with values of 0.023.

At groups formed for 70 ºC (Figure 2D) the Cavalcanti Mata, Verma, Diffusion Approximation, Two-term and Logarithmic models were the most similar to the observed values. The shortest distances between the models and the experimental data were observed for Cavalcanti Mata (0.056), and Verma and Diffusion Approximation (0.046) models.

Thus, the models with greatest similarity to the experimental data, considering all *Croton urucurana* B. leaves drying temperatures were the Diffusion Approximation, Cavalcanti Mata, Two-term, Two-term Exponential, Modified Henderson & Pabis, Logarithmic, Midilli, Page and Verma.

Evaluating the models' coefficients of determination (R^2), they showed greater similarity to the experimental data and were above 99.12% (Table 2). According to Kashaninejad et al. (2007), models with coefficients of determination above 98% can satisfactorily represent the drying phenomenon. Nevertheless, Mohapatra & Rao (2005) report that the coefficient of determination as single criterion of evaluation to select drying models is not a good parameter to represent the drying phenomenon.

P values indicate the observed values' deviation from the model estimated curve, and values lower than 10% are recommended for model selection (Mohapatra & Rao, 2005). Among the models with the shortest distance from the experimental values, the Cavalcanti Mata model was the only one with a P value lower than 10% for all drying temperatures (Table 2).

Through the SE and χ^2 results, it is observed that all nine models obtained values close to zero for all temperatures, in which, the lower the SE and χ^2 values, the smaller the discrepancy between the experimental and estimated values by the models (Siqueira et al., 2012).

Estimated value analysis using AIC and BIC information criteria showed that the Cavalcanti Mata model had the lowest values among all models tested (Table 3). The AIC and BIC information criteria assist model selection by penalizing the difference between the equation terms, where the classical estimation procedure would not be adequate (Akaike, 1974; Schwarz 1978). These information criteria have been used for model selection when describing 'jambu' leaves (Gomes et al., 2018), *Piper aduncum* leaves (Quequeto et al., 2019), 'Prata' and 'D'água' banana fruit (Furtado et al., 2019) and 'jabuticaba' fruit (López-Vidaña et al., 2015) drying kinetics. Thus, the Cavalcanti Mata model was the best model for representing the *Croton urucurana* B. leaves drying kinetics at 40, 50, 60 and 70 ºC (Figure 3).

Therefore, it is possible to describe most thin-layer drying processes as a function of temperature and initial moisture content. Therefore, it is possible to describe most thin-layer drying processes with as function of temperature and initial

Table 2. Evaluators to determine model adjustment quality through the coefficient of determination (R^2), relative error (P, %), estimated mean error (SE) and chi-square (χ^2) calculated for nine models grouped according to the maximum similarity method, using the observed MR values as reference, to represent the *Croton urucurana* B. leaves drying kinetics at temperatures of 40, 50, 60 and 70 ºC, as a function of time.

Models	40 °C				50 °C				60 °C				70 °C			
	R^2	P(%)	SE	χ^2	R^2	P(%)	SE	χ^2	R^2	P(%)	SE	χ^2	R^2	P(%)	SE	χ^2
Diffusion Approximation	99.40	21.43	0.000	0.001	99.86	6.18	0.000	0.000	99.96	12.47	0.000	0.000	99.77	9.45	0.000	0.000
Cavalcanti Mata	99.94	3.75	0.000	0.000	99.94	3.30	0.000	0.000	99.96	7.55	0.000	0.000	99.67	9.33	0.001	0.001
Two-term	99.49	19.58	0.000	0.001	99.86	6.16	0.000	0.000	99.71	35.18	0.000	0.000	99.38	11.74	0.001	0.001
Two-term Exponential	99.24	25.39	0.000	0.001	99.75	6.67	0.000	0.000	99.96	10.21	0.000	0.000	99.61	8.76	0.000	0.001
Modified Henderson & Pabis	99.85	6.95	0.000	0.000	99.74	6.50	0.000	0.000	98.98	53.87	0.001	0.002	99.83	10.02	0.000	0.001
Logarithmic	99.30	22.48	0.000	0.001	99.61	5.72	0.000	0.000	99.35	31.50	0.000	0.001	99.33	13.30	0.001	0.001
Midilli	99.83	5.51	0.000	0.000	99.81	8.09	0.000	0.000	99.94	10.60	0.000	0.000	99.63	8.39	0.000	0.001
Page	99.62	12.29	0.000	0.000	99.77	6.36	0.000	0.000	99.94	1.59	0.000	0.000	99.61	8.66	0.000	0.001
Verma	99.16	25.47	0.001	0.001	99.12	12.21	0.001	0.001	99.24	38.17	0.000	0.001	99.77	9.45	0.000	0.000

t = drying time, h; k, k_o, k_1, k_2; g = drying constants in h^{-1}; a, a_0, a_1, a_2, a_3, b, c, n, n_1, n_2 = model parameters.

Table 3. Akaike (AIC) and Bayesian Information Criteria (BIC) for the nine models grouped according to the maximum similarity method, using the observed MR values as reference, to represent *Croton urucurana* B. leaves drying kinetics.

Models	40 °C		50 °C		60 °C		70 °C	
	AIC	BIC	AIC	BIC	AIC	BIC	AIC	BIC
Diffusion Approximation	-84.45	-80.08	-91.53	-87.55	-53.10	-50.55	-31.32	-30.53
Cavalcanti Mata	-137.83	-130.20	-124.76	-117.79	-96.24	-91.77	-96.24	-91.77
Two-term Exponential	-75.14	-71.86	-83.27	-80.28	-47.26	-45.35	-33.60	-33.01
Logarithmic	-86.81	-82.44	-92.16	-88.17	-54.62	-52.06	-54.62	-52.06
Midilli	-116.49	-111.03	-110.05	-105.07	-87.63	-84.44	-87.63	-84.44
Page	-102.34	-99.06	-104.70	-101.71	-90.52	-88.61	-38.68	-38.09
Verma	-75.71	-72.44	-83.28	-80.29	-47.27	-45.35	-31.21	-30.62

Table 4. Cavalcanti Mata model parameters adjusted to *Croton urucurana* B. leaves drying in a forced air circulation oven at 40, 50, 60 and 70 °C.

Parameter	Temperature (°C)			
	40	50	60	70
a_1	-0.2653**	0.2900**	0.0643 ^{ns}	-0.0400**
k_1	0.1652**	0.2320**	0.7973**	1.6435**
n_1	2.4233**	2.2213**	4.0458 ^{ns}	175.1356**
a_2	1.2651**	0.8690**	0.9410**	1.0386**
n_2	1.4740**	0.9038**	1.2148**	1.2497**
a_3	-0.0096 ^{ns}	-0.1589 ^{ns}	-0.0020 ^{ns}	0.0062**

^{ns}Not significant by Tukey's test; **Significant at 0.01 probability level by Tukey's test.

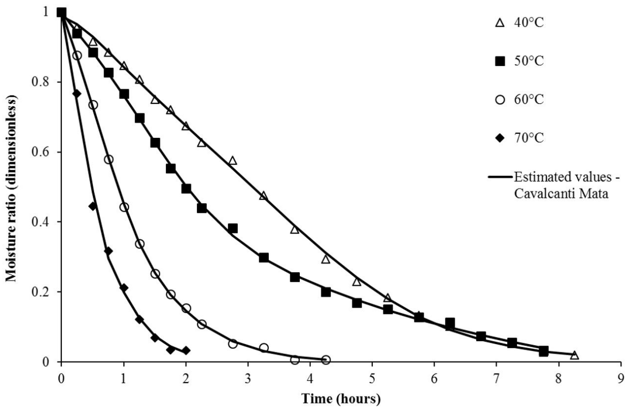

Figure 3. Moisture ratios obtained experimentally and estimated by the Cavalcanti Mata model for *Croton urucurana* B. leaves drying in a forced air circulation oven, at 40, 50, 60 and 70 °C, as a function of time.

moisture content. The Cavalcanti Mata model is able to describe adequately the parameters of drying proposed, enabling the visualization of the three drying periods (constant, decreasing and equilibrium moisture content). The model maintained higher similarity with the moisture content ratio values when compared to the experimental data for the *Croton urucurana* B. leaves drying.

An increase in the k_1 values for the Cavalcanti Mata model was observed with the drying air temperature changes, while for the other parameters, there was no clear trend as a function of temperature (Table 4). According to Goneli et al. (2009), the magnitude of the drying constant (k), represents the effect of external drying conditions and tends to increase with the elevation of the drying air temperature.

The increment in the k_1 coefficient with increasing temperature indicates that the water viscosity decreases, and the water found inside the leaves can easily migrate when compared to lower drying temperatures. Hence, the higher the k_1 parameter magnitude, the higher is the effective diffusivity in the drying process (Martins et al., 2015). The increment in drying air temperature increased vibration at the water molecule level, reducing the fluid viscosity and favoring its movement through the *Croton urucurana* B. leaves (Alves et al., 2017), accelerating the drying process.

4 Conclusions

The application of multivariate clustering techniques to select models is a great applicability tool to evaluate the drying of *Croton urucurana* B. leaves, just as the AIC and BIC information criteria can be used to assist in the decision-making, when more than one model overlaps each other. The Cavalcanti Mata mathematical model was selected to represent the drying kinetics of *Croton urucurana* B. leaves.

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