

Ethanol pretreatment in taioba leaves during vacuum drying

Pré-tratamento com etanol em folhas de taioba durante secagem à vácuo

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ABSTRACT

Non-conventional vegetables are those with limited distribution, restricted to certain regions. As a globalization and food industrialization result, its cultivation and consumption has decreased. The taioba [*Xanthosoma sagittifolium* (L.) Schott] is an example of this vegetable category. The drying process increases the food stability, but it can negatively alter the nutritional value and the final quality of the product. Ethanol pretreatment reduces the drying time and can assist in the preservation of the nutritional characteristics. The aim of this work was to evaluate the effect of vacuum drying and ethanol pretreatment on the drying behavior, mathematical modeling, and final quality of taioba leaves. Higher temperatures and ethanol pretreatment lead to a shorter drying time. Thin-layer equations were evaluated for their ability to predict the drying kinetics, of which the Logarithmic and Midilli & Kuçuk equations performed best. A significant difference ($p \leq 0.05$) was observed in the ascorbic acid content, antioxidant activity and total phenolic compounds in the different treatments. There was preservation in ascorbic acid content in treatments in which ethanol was applied; moreover, lower total phenolic compounds and antioxidant activity were observed when ethanol was used. There was no significant difference ($p > 0.05$) in pH values and titratable acidity.

Index terms: Food dehydration; non-conventional vegetables; *Xanthosoma sagittifolium* (L.) Schott.

RESUMO

Vegetais não convencionais são aqueles que apresentam distribuição limitada, restrita a determinadas regiões. Como resultado da globalização e da industrialização de alimentos, seu cultivo e consumo foram reduzidos. A taioba [*Xanthosoma sagittifolium* (L.) Schott] é um exemplo desta categoria de vegetais. O processo de secagem aumenta a estabilidade do alimento, mas pode alterar negativamente o valor nutricional e a qualidade final do produto. O pré-tratamento com etanol reduz o tempo de secagem e pode auxiliar na preservação das características nutricionais. O objetivo deste trabalho foi avaliar o efeito da secagem a vácuo e do pré-tratamento com etanol na cinética de secagem, modelagem matemática e qualidade final de folhas de taioba. Maior temperatura e uso de etanol proporcionaram menor tempo de secagem. Equações empíricas foram empregadas para avaliação do comportamento da secagem, das quais as equações Logarítmica e Midilli & Kuçuk apresentaram melhores desempenhos. Foi observada diferença significativa ($p \leq 0,05$) no teor de ácido ascórbico, atividade antioxidante e compostos fenólicos totais nos diferentes tratamentos. Houve preservação do teor de ácido ascórbico nos tratamentos em que o etanol foi aplicado. Menores teores de compostos fenólicos totais e atividade antioxidante foram observados quando o etanol foi empregado. Não houve diferença significativa ($p > 0,05$) nos valores de pH e acidez titulável.

Termos para indexação: Desidratação de alimentos; vegetais não convencionais; *Xanthosoma sagittifolium* (L.) Schott.

INTRODUCTION

Due to biodiversity wealth, many plant species are underexploited, but these products can be used as an alternative nutrient source, complementing feeding and assisting populations with their subsistence. Non-conventional vegetables present limited distribution, restricted to locations close to their production. These

vegetables are not organized in a productive chain, instead conventional vegetables (such as lettuce, potatoes, and tomatoes), and present reduced commercial interest (Almeida et al., 2014; Ziegler et al., 2020).

The taioba [*Xanthosoma sagittifolium* (L.) Schott] is an example of a non-conventional vegetable which presents edible leaves and tubers and is widely distributed in regions of South America, Africa, and Asia. The taioba

leaves present potential nutritional and functional benefits, but they are underutilized (Jackix et al., 2013; Ukom; Nwanagba; Okereke, 2020). Oliveira et al. (2013) found higher levels of potassium, phosphorus, calcium and magnesium in taioba leaves, compared with conventional vegetables (chicory, cabbage and watercress).

Drying techniques are widely employed as a food preservation method. The convective drying is the most used for industrial purposes, but this process presents some drawbacks, including low energy efficiency, long heat exposure, and changes in structural, nutritional, functional and sensorial product characteristics. Edible leaves are considered heat-sensitive materials and must be dried in the vacuum drying system - VD at low temperatures (< 50 °C), for a reduction in drying time process, oxygen suppression and properties maintenance (Babu et al., 2018; Oliveira et al., 2021; Szadzińska et al., 2018).

Comparing different drying methods (shade drying, freeze drying, oven drying and vacuum drying), Ebadi et al. (2015) concluded that VD at 60 °C could be considered an alternative approach for lemon verbena (*Lippia citriodora* Kunth) drying. The VD of coriander leaves (*Coriandrum sativum* L.) was studied by Thirugnanasambandham and Sivakumar (2016). Those authors evaluated the effects of vacuum, loading rate and temperature and concluded that the VD was an effective method to achieve desired final moisture content.

The ethanol pretreatment is related to a reduction in drying process time (Souza et al., 2018), diffusivity increase (Corrêa et al., 2012), and the maintenance of nutritional and sensory characteristics (Araújo et al., 2020).

The aims of this work were to (i) investigate the effect of VD and ethanol pretreatment (at different temperatures) on the drying behavior of a non-conventional vegetable: taioba; (ii) evaluate the suitability of diffusional and empirical equations for predicting the process; and (iii) to examine the final quality of the dried leaves (ascorbic acid retention, total phenolic content, antioxidant capacity, pH and titratable acid).

MATERIAL AND METHODS

Sample preparation

Edible taioba leaves [*Xanthosoma sagittifolium* (L.) Schott] were obtained from a local producer (Lavras, Brazil, 21° 14' 45" S; 44° 59' 59" E). The selected leaves present similar visual aspects (size, shape, color) and absence of physical injuries. Prior to the drying experiments, the leaves were stored at 4 ± 1 °C. The

samples were characterized with respect to the initial moisture content (Association of Official Agricultural Chemists - AOAC, 2016) and water activity (a_w) (Aqualab, 3-TE model, Decagon Devices Inc., Pullman, WA, USA). The initial moisture content was 5.711 ± 0.103 kg water/kg dry basis (d.b.) and the a_w was 0.984 ± 0.002, demonstrating its perishability.

The leaves were washed in tap water, gently dried with absorbent paper, and sliced in slabs (4.0 × 10⁻² m length, 2.0 × 10⁻² m width). The pretreated samples were sprayed with ethanol (4.0 × 10⁻⁴ L) on each leaf side (Corrêa et al., 2012).

Vacuum drying experiments

The vacuum drying of pretreated and untreated samples was conducted in a temperature-controlled oven (Solab SL104/40, Piracicaba, Brazil) coupled with a vacuum pump. The experiments were performed at two different temperatures, generally employed for leaves drying (40 and 50 °C). A vacuum pressure of 10 kPa was applied. During the vacuum drying, the mass of the samples was monitored using a digital balance. All the drying experiments were carried out in triplicate, and for each assay, approximately 25 slices were used.

Drying kinetics and mathematical modeling

Drying behavior

The experimental results obtained were fitted using drying equations. The moisture ratio (MR) of the samples was calculated using the Equation 1.

$$MR = \frac{X_t - X_e}{X_0 - X_e} \quad (1)$$

where MR is the moisture ratio [dimensionless], X_t is the moisture content at a specific time [kg water/kg], X_0 is the initial moisture content [kg water/kg] and X_e is the moisture content under equilibrium conditions [kg water/kg].

The drying rate (DR) of the taioba leaves was calculated using Equation 2.

$$DR = \frac{X_{t+dt} - X_t}{dt} \quad (2)$$

where DR is drying rate [kg water/ (kg × min)], t is time [min] and dt is time increase [min].

Effective diffusivity determination

The effective diffusivity (D_{eff}) was obtained according to the analytical solution of Fick's second law in unsteady state (Equation 3):

$$\frac{\partial X_t}{\partial t} = D_{\text{eff}} \nabla^2 X_t \quad (3)$$

where D_{eff} is the effective diffusivity [m^2/s].

The solution to Equation 3 is obtained using the Fourier series, following some assumptions:

Uniform initial moisture content $X_{(z,0)} = X_0$;

Moisture concentration symmetry $\left. \frac{\partial X_t}{\partial z} \right|_{z=0} = 0$;

Equilibrium content at the surface, $X_{(L,t)} = X_e$;

The samples are infinite slabs (length and width much greater than thickness);

Isothermal process;

Shrinkage and external resistance to mass transfer are neglected;

Considering a brief process and unidirectional moisture diffusion, the D_{eff} is calculated according to Equation 4.

$$MR = \left(\frac{8}{\pi^2} \sum_{i=0}^{\infty} \frac{1}{(2i+1)^2} \exp\left(- (2i+1)^2 \pi^2 D_{\text{eff}} \frac{t}{4L^2}\right) \right) \quad (4)$$

where L is the characteristic length (half of the thickness), which corresponds to 2.842×10^{-4} m.

Empirical equations

The drying curve was fitted with some empirical equations derived from Fick's diffusion model. The empirical equations applied to agricultural products are shown in Table 1.

Quality analyses

The following analyses were performed in either fresh or dried taioba leaves. All the analyses were performed in triplicate.

Total phenolic content

The total phenolic content (TPC) was quantified in the Folin-Ciocalteu assay (Singleton; Rossi, 1965) with certain modifications. The absorbance was measured by UV-visible spectrophotometer (Cary 50, Varian, Australia) at 750 nm. Gallic acid was used as the standard, and the results were expressed as gallic acid equivalents (GAE) in mg/g (d.b.).

Antioxidant activity

The DPPH (2,2-difenil-1-picril-hidrazil) radical-scavenging capacity of the leaf extracts was evaluated according to Brand-Williams et al. (1995) with slight modifications. The DPPH method is an electron transfer based assay, based on the sequester the stable radical DPPH•, which measures the antioxidant capacity (Apak et al., 2013).

Table 1: Empirical equations applied to the drying curves.

Model	Equations	References
Henderson & Pabis	$MR = a \exp(-kt)$	Henderson and Pabis (1961)
Logarithmic	$MR = a \exp(-kt) + b$	Akpinar et al. (2003)
Midilli & Kuçuk	$MR = a \exp(-kt^n) + bt$	Midilli et al. (2002)
Page	$MR = \exp(-kt^n)$	
Parabolic	$MR = a + bt + ct^2$	Sharma and Prasad (2004)
Two terms	$MR = a \exp(-k_0 t) + b \exp(-k_1 t)$	Madhyanon et al. (2009)
Wang & Singh	$MR = 1 + at + bt^2$	Wang and Singh (1978)

Where: MR is the moisture ratio, t is the drying time [s] and a, b, c, n and k are empirical constants and coefficients in drying equations.

Ascorbic acid

The ascorbic acid (AA) content was determined according to Strohecker and Henning (1967) by the colorimetric method using 2,4-dinitrophenyl hydrazine. Briefly, the AA was extracted with 0.5 % oxalic acid, filtered, and dosed in the extract, using AA as a standard. The absorbance was measured by a UV-visible spectrophotometer (Cary 50, Varian, Australia) at 520 nm. The results were expressed in mg/100 g (d.b.).

pH and titratable acidity

The pH was obtained in a pHmeter (Digimed, DMpH-2 model, São Paulo, Brazil) and the titratable acidity (TA) determination was performed by titration with an NaOH solution, according to AOAC (2016). The TA was expressed as mg of citric acid (CA)/g (d. b.)

Statistical analyses

Statistical evaluation of the mathematical modeling

The results were analyzed using Statistica software (Statistica 8.0, Statsoft Inc., Tulsa, UK). The equations parameters were estimated using a non-linear regression procedure. The coefficient of determination (R^2), root mean square error (RMSE), reduced chi-square (χ^2), and percent mean deviation modulus (P%) were used to determine the quality of the adjustment. Higher R^2 and lower RMSE and P% values indicated better adjustment (Babu et al., 2018). These parameters can be calculated using Equations (5, 6 and 7):

$$RMSE = \sqrt{\frac{1}{N} \sum (Y - \hat{Y})^2} \quad (5)$$

$$\chi^2 = \frac{\sum (Y - \hat{Y})^2}{DF} \quad (6)$$

$$P(\%) = \frac{100}{N - DF} \sum \left| \frac{Y - \hat{Y}}{Y} \right| \quad (7)$$

where Y and \hat{Y} denote experimental and predicted values respectively, DF is the residual degrees of freedom (number of experimental observations minus the number of model parameters) and N is number of experimental observations.

Statistical evaluation of the quality analysis

The quality analysis results were evaluated by one-way ANOVA at the 95% probability level. The means were compared using the Tukey test, in case of significant effects ($p < 0.05$). These analyses were also performed using the software Statistica 8.0 (Statsoft Inc., Tulsa, UK).

RESULTS AND DISCUSSION

Drying kinetic

Figure 1 shows the drying curves of untreated and treated taioba leaves during the vacuum drying. The time for the samples to reach a final moisture content of 0.873 ± 0.003 kg water/ kg d.b. ranged from 720 min (50 °C, ethanol) to 1500 min (40 °C, untreated).

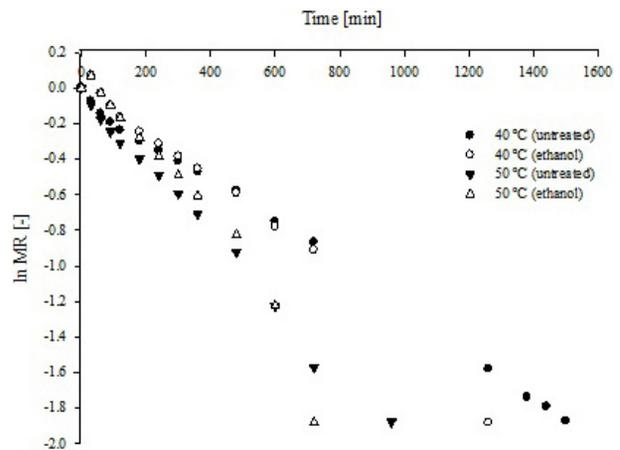


Figure 1: Natural logarithm of the dimensionless moisture versus time in different vacuum drying treatments.

Lower drying time was observed in the treatments at 50 °C (Figure 1). The internal resistance to moisture removal reduces as the drying temperature increases. Such a fact is related to the increase in the water molecules mobility. The external resistance also reduces with the temperature, due to the increase in the driving force. According to Figure 1, the drying time decreased by about 40 % increasing the drying air temperature from 40 to 50 °C. The enhancement of vapor pressure in the sample intensifies the moisture evaporation from the solid inner to the surface, with the temperature increase (Aral; Beşe, 2016; Cano-Lamadrid et al., 2018; Oliveira et al., 2021).

Some authors, as Elhussein and Şahin (2018) and Thirugnanasambandham and Sivakumar (2016), have

concluded that high temperatures lead to a reduction in the total drying time during the vacuum drying of olive and *Coriandrum* leaves, respectively.

The ethanol treatment reduced the drying time, for both temperatures (Figure 1). The pretreatments could reduce the drying time by about 20 %. This fact occurred due to the Marangoni effect. This effect is due to the existence of a surface tension gradient at the interface between the liquids (water + ethanol). The ethanol presents a lower surface tension (and higher volatility) than pure water, and in this solution, a rather strong convective motion may be produced, which results in a surface shear stress, facilitating the water removal (Gambaryan-Roisman, 2015).

Similar reports were presented by Silva, Celeghini and Silva (2018) during the drying of treated and untreated guaco leaves (*Mikania laevigata* Schultz Bip. ex Baker). In this study, comparing the treatments with and without ethanol, a reduction in drying time ranging from 16 to 20 %, at 50 and 60 °C, respectively, was observed. The influence of ethanol pretreatment was evaluated by Souza et al. (2018) during the vacuum and convective drying of okara (soy coproduct processing). The ethanol reduced the drying time by 20 to 40 %, saving energy.

The Figure 2 shows the drying rates of taioba leaves during the different treatments.

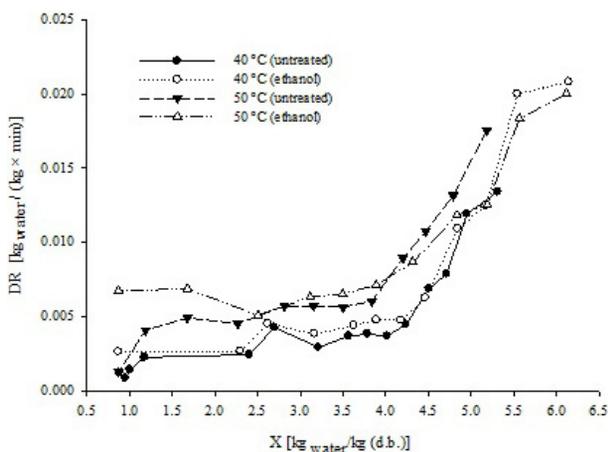


Figure 2: The drying rate of taioba leaves versus moisture content in different vacuum drying treatments.

It can be seen from Figure 2 that the different treatments were found in the falling rate period, which implies that the diffusion mass transfer controls the drying process. The absence of a constant drying rate period has been reported by several authors during agricultural food drying (Babu et al., 2018; Lyu et al., 2017; Purkayastha et al., 2013).

At the end of the process when moisture content was low, the drying rate under all drying conditions reduced (Figure 2), and it was higher in the leaves pretreated with ethanol [$> 0.002 \text{ kg water}/(\text{kg} \times \text{min})$]. In such situation, the water molecules form a eutectic solution with the ethanol, which accelerates the water evaporation, by reducing the vapor pressure, as can be noted at the process beginning. The higher DR during the initial period of the process is related to the increased water migration and evaporation, and less external resistance (Esturk, 2012; Mghazli et al., 2017; Yilmaz; Alibas, 2017).

As can be observed in Figures 1 and 2, the leaves dried at 50 °C present higher drying rates (lower drying periods). The drying temperature increase promoted an increase in the drying rate, thus the drying time was reduced. Shi, Zheng and Zhao (2013) pointed out that the water molecules move faster with the temperature increase, due to the enhancement in the heat transfer rate. During leaf drying, Doymaz (2009) and Therdthai and Zhou (2009) observed that the increase in the air temperature improved the drying rate of spinach and mint, respectively.

Mathematical model

Several mathematical models were used for modeling the drying kinetics. Table 2 presents the effective diffusivity (D_{eff}) values.

The D_{eff} values ranged from 4.418×10^{-11} to $8.008 \times 10^{-11} \text{ m}^2/\text{s}$ and the R^2 values ranged from 0.9058 to 0.9899. The RMSE and χ^2 values were under 1.184×10^{-1} and 1.525×10^{-2} , respectively.

The D_{eff} values vary with the agricultural material and the experimental conditions, hindering the comparison of exact values, but the results presented in Table 2 indicate an analogous magnitude order of edible leaves subjected to drying processes (Elhoussein; Şahin, 2018). During the drying of bay laurel leaves, Ghnimi, Hassini and Bagane (2016) observed D_{eff} varying from 1.21×10^{-11} to $5.27 \times 10^{-11} \text{ m}^2/\text{s}$ in the temperature range of 45–75 °C and during the drying of rosemary leaves, Mghazli et al. (2017) observed D_{eff} varying from 1.21×10^{-11} to $5.27 \times 10^{-11} \text{ m}^2/\text{s}$ in the temperature range of 50–80 °C.

According to Table 2, the pretreatment with ethanol reduced the accuracy of this diffusive model ($R^2 < 0.93$). This fact occurred due to the interaction between the water and the ethanol, which affects the initial and boundary assumptions employed for the analytical development of this model (such as constant initial moisture distribution and homogeneous D_{eff}) during the drying process, beyond the Marangoni's effect (Junqueira et al., 2021; Simpson et al., 2015).

Table 2: Effective diffusivities (D_{eff}) and statistical parameters during the VD of taioba leaves.

Treatment	$D_{\text{eff}} \times 10^{11}$ [m/s ²]	R ²	RMSE $\times 10^2$	$\chi^2 \times 10^3$
40 °C (untreated)	5.054	0.9899	3.909	1.630
40 °C (ethanol)	4.418	0.9393	8.758	8.309
50 °C (untreated)	8.008	0.9858	4.342	2.042
50 °C (ethanol)	6.335	0.9058	11.845	15.254

The highest D_{eff} values were obtained at the highest temperature (50 °C). This behavior was expected and is consistent with the process physics, since D_{eff} encompasses all phenomena that can intervene in the water migration in the system: the higher temperature, the lower internal resistance (Golestani; Raisi; Aroujalian, 2013). Elhussein and Şahin (2018) studied different methods for olive leaves drying and noted that the D_{eff} ranged from 1.1×10^{-10} m²/s (at 50 °C) to 6.2×10^{-10} m²/s (at 90 °C) in the VD method.

Table 3 summarizes the empirical equation parameters with their comparison criteria (R², RMSE, χ^2 and P%) for their suitability. In general, all the empirical equations were found highly appropriate for describing the taioba leaves drying behavior in different treatments. The R² value were higher than 0.988, and the RMSE, χ^2 and P% were lower than 0.04, 0.003 and 8.82, respectively. The P% value analysis was conducted and according to Kaushal and Sharma (2016), P% < 5 indicates an excellent fit, while P% > 10 is indicative of an inadequate fit.

Therefore, depending on the relatively high P% values (> 5), Henderson & Pabis, Page, Two terms and Wang & Singh equations were the least compatible models, even though under some conditions, these equations adequately described the experimental drying.

The suitability of Logarithmic, Midilli & Kuçuk and Parabolic equations for portraying the convective drying behavior of agricultural products is extensively presented in the literature (Kucuk et al., 2014). Mbegbu, Nwajinka and Amaefule (2021) reported that the Logarithmic model showed good fit for scent and lemon basil leaves in the vacuum drying processes. Such a model was used for describing the drying behavior of thyme leaves during convective drying at different temperatures (Turan; Firatligil, 2019).

Alara et al. (2018) concluded that the Midilli & Kuçuk model could be used in the prediction of both open sun and shade drying behavior of *Vernonia amygdalina* Del. leaves.

Quality analysis

The effect of ethanol pretreatment and temperature during the vacuum drying on total phenolic content (TPC), total antioxidant activity (TAA), ascorbic acid (AA), pH and titratable acidity (TA) was evaluated. The results are shown in Figures 3 and 4 and Table 4. Different treatments caused significant changes in TPC and AA, but no differences were observed for pH and TA.

The TPC of the fresh leaves was 5.01 ± 0.15 mg GAE/g (d. b.), and according to Figure 3, all treatments exhibited TPC reduction. A higher TPC retention (p < 0.05) was observed in the untreated treatments (without ethanol pretreatment). In those treatments, the retention ranged from 57.11 % (50 °C, untreated) to 62.34 % (40 °C, untreated). The TPC retention is desirable, since phenolics present a wide range of biological activity, including antioxidant properties (Del Rio et al., 2013).

The phenolic compounds are within the vacuole (apart from the enzymes and oxygen). During the drying process, irreversible changes in the cellular structure are observed, and a decompartmentation occurs, favoring degradative reactions, since those damages trigger the release of enzymes (mainly polyphenol oxidase and peroxidase) and decreases the TPC. Nevertheless, the vacuum limits the oxygen concentration (for oxidative reactions), the phenolic compounds are heat sensitive, and the exposure to the heat and light could complete the phenolics damage (Erbay; Icier, 2009; Zielinska; Zielinska; Markowski, 2018).

Our results are also in agreement with those reported by Nóbrega et al. (2014), who observed that the TPC of acerola residue was significantly reduced after convective drying.

The TAA seems to be related to the phenolic presence, and their properties may change because of their oxidation state, and it was determined by the DPPH• method. According to Figure 4, a significant difference between treatments (p > 0.05) was observed.

Table 3: Adjustment parameters and statistics results obtained from different thin-layer drying equations.

Equation	Constant	R ²	RMSE	χ ²	P (%)
40 °C (untreated)					
Henderson & Pabis	a= 0.95; k= 1.91×10 ⁻⁵	0.9972	0.0206	0.0005	2.796
Logarithmic	a= 0.96; k= 1.93×10 ⁻⁵ ; c= -1.21×10 ⁻²	0.9972	0.0206	0.0005	2.438
Midilli & Kuçuk	a= 1.00; k= 6.81×10 ⁻⁴ ; n=0.64 ; b= -2.51×10 ⁻⁶	0.9994	0.0093	0.0001	2.181
Page	k= 9.00×10 ⁻⁵ ; n= 0.86	0.9966	0.0227	0.0006	5.199
Parabolic	a= 0.93; b= -1.59×10 ⁻⁵ ; c= 8.17×10 ⁻¹¹	0.9962	0.0240	0.0007	3.022
Two terms	a= 4.30×10 ⁻⁵ ; b= 0.95; k ₀ = 6.24×10 ⁻⁵ ; k ₁ = 1.95×10 ⁻⁵	0.9972	0.0204	0.0005	2.354
Wang & Singh	a= -1.96×10 ⁻⁵ ; b= 1.16×10 ⁻¹⁰	0.9888	0.0412	0.0019	6.479
40 °C (ethanol)					
Henderson & Pabis	a= 1.04; k= 2.31×10 ⁻⁵	0.9930	0.0301	0.0011	4.092
Logarithmic	a= 1.08; k= 2.14×10 ⁻⁵ ; c= -4.85×10 ⁻²	0.9932	0.0297	0.0011	3.378
Midilli & Kuçuk	a= 1.05; k= 5.17×10 ⁻⁵ ; n= 0.91; b= 1.11×10 ⁻⁶	0.9935	0.0289	0.0012	2.642
Page	k= 7.83×10 ⁻⁶ ; n= 1.10	0.9913	0.0337	0.0013	3.341
Parabolic	a= 1.02; b= -2.00×10 ⁻⁵ ; c= 1.15×10 ⁻¹⁰	0.9916	0.0329	0.0014	3.384
Two terms	a= 2.81; b = -1.77; k ₀ = 2.60×10 ⁻⁵ ; k ₁ = 2.78×10 ⁻⁵	0.9930	0.0301	0.0013	3.970
Wang & Singh	a= -1.86×10 ⁻⁵ ; b= 9.99×10 ⁻¹¹	0.9905	0.0349	0.0014	2.983
50 °C (untreated)					
Henderson and Pabis	a= 0.96; k= 3.25×10 ⁻⁵	0.9971	0.0194	0.0004	3.458
Logarithmic	a= 0.97; k= 3.22×10 ⁻⁵ ; c= -5.86×10 ⁻³	0.9971	0.0194	0.0005	3.502
Midilli and Kuçuk	a= 0.99; k= 2.38×10 ⁻⁴ ; n= 0.79; b= 2.12×10 ⁻⁶	0.9983	0.0149	0.0003	3.928
Page	k= 9.98×10 ⁻⁵ ; n= 0.89	0.9970	0.0197	0.0004	4.318
Parabolic	a= 0.95; b= 2.61×10 ⁻⁵ ; c= 2.15×10 ⁻¹⁰	0.9962	0.0223	0.0006	3.045
Two terms	a= -0.17; b = 1.13; k ₀ = 3.25×10 ⁻⁵ ; k ₁ = 3.25×10 ⁻⁵	0.9971	0.0194	0.0005	3.458
Wang and Singh	a= -3.03×10 ⁻⁵ ; b= 2.78×10 ⁻¹⁰	0.9905	0.0355	0.0015	5.511
50 °C (ethanol)					
Henderson and Pabis	a= 1.08; k= 3.41×10 ⁻⁵	0.9879	0.0432	0.0028	8.827
Logarithmic	a= 1.95; k= 1.38×10 ⁻⁵ ; c= -0.89	0.9945	0.0291	0.0011	3.747
Midilli and Kuçuk	a= 1.04; k= 1.31×10 ⁻⁵ ; n= 1.05; b= 5.23×10 ⁻⁶	0.9946	0.0289	0.0016	3.867
Page	k= 6.68×10 ⁻⁷ ; n= 1.38	0.9919	0.0353	0.0015	5.542
Parabolic	a= 1.05; b= 2.61×10 ⁻⁵ ; c= 1.36×10 ⁻¹⁰	0.9943	0.0295	0.0012	3.870
Two terms	a= 7.34; b = -6.29; k ₀ = 5.87×10 ⁻⁵ ; k ₁ = 6.52×10 ⁻⁵	0.9928	0.0333	0.0017	6.099
Wang and Singh	a= -2.13×10 ⁻⁵ ; b= 4.28×10 ⁻¹¹	0.9910	0.0372	0.0016	3.217

Where: a, b, c, n and k are empirical constants and coefficients in drying equations.

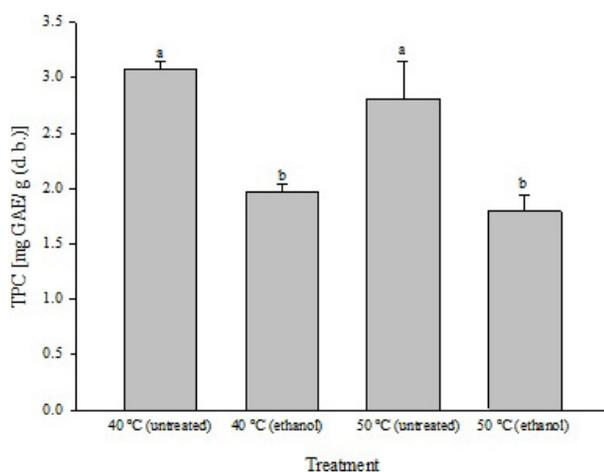


Figure 3: Total phenolic content (TPC) of taioba leaves in different vacuum drying treatments. Means followed by different letters show significant differences ($p \leq 0.05$), according to Tukey's test.

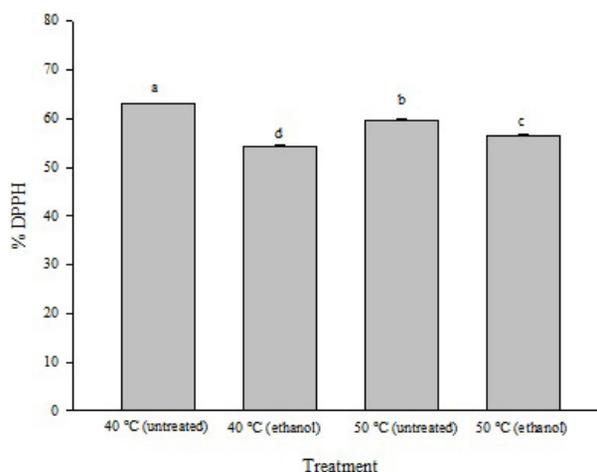


Figure 4: Total antioxidant activity (TAA) of taioba leaves in different vacuum drying treatments. Means followed by different letters show significant differences ($p \leq 0.05$), according to Tukey's test.

Regardless of the temperature, the ethanol treatment leads to a lower DPPH• radical scavenging activity, when compared to the untreated. According to Rojas and Augusto (2018), the use of ethanol can produce or intensify of microchannels, which in processes as drying, could cause the loss of food matrix compounds. The best result should be attributed to the treatment submitted to 40 °C, untreated ($63.12 \% \pm 0.01$), followed by samples submitted to 50 °C, untreated ($59.76 \% \pm 0.08$). Such a result reinforces that compounds with antioxidant character may exhibit thermosensitivity and significant degradation with increased temperature (Lutz; Hernández; Henríquez, 2015).

The results showed that the drying method had a significant effect on AA content ($p \leq 0.05$), according to Table 4. The highest AA content [0.860 ± 0.002 mg/ g (d.b.)] occurred with VD at 40 °C with ethanol

pretreatment, and the lowest AA content was obtained by [0.239 ± 0.002 mg/ g (d.b.)] VD at 50 °C without ethanol pretreatment. All the treatments presented lower AA content, compared with fresh taioba leaves [4.924 ± 0.007 mg/ g (d.b.)]. Such a reduction was expected, as this vitamin is heat-sensitive (Junqueira et al., 2017).

The temperature increase enhances the drying evaporation, reducing the total drying time (Figure 1), although, a degradation of heat-sensitive compounds is observed, indicating quality loss (Saini et al., 2014). During the pequi drying under different conditions, Mendonça et al. (2017) found that samples dried at low temperatures, had high AA retention. The ethanol pretreatment assisted the AA preservation (Table 4). This technique increases the moisture evaporation, reducing the long exposure to temperature conditions (Araújo et al., 2020).

Table 4: Chemical characteristics of dried taioba leaves.

Treatment	AA [mg/ g (d. b.)]	pH [-]	TA [mg CA/g (d. b.)]
40 °C (untreated)	0.689 ± 0.003 b	6.13 ± 0.04 a	0.174 ± 0.026 a
40 °C (ethanol)	0.860 ± 0.002 a	6.11 ± 0.03 a	0.152 ± 0.013 a
50 °C (untreated)	0.239 ± 0.002 c	6.09 ± 0.02 a	0.182 ± 0.022 a
50 °C (ethanol)	0.683 ± 0.004 b	6.10 ± 0.04 a	0.175 ± 0.013 a
CV (%)	1.60	0.93	1.54

Average value \pm standard deviation. Mean followed by different letters in the column differs significantly ($p \leq 0.05$), according to Tukey's test.

The pH of the fresh taioba leaves was 5.87 ± 0.01 . According to Table 4, an increase in pH was observed and it was not significantly different among the samples ($p > 0.05$). The pH values ranged from 6.09 ± 0.02 to 6.13 ± 0.04 . This result might be due to the organic acids in the leaves, moreover the drying process may provoke the acidic compounds evaporation (Sagrin; Chong, 2013). Similar reports were found by Shitanda and Wanjala (2006) during the evaluation of different drying techniques of jute leaves and by Sagrin and Chong (2013) during the drying of banana leaves in different temperatures.

According to Table 4, no significant difference ($p > 0.05$) was observed in the TA values among the treatments. The TA of the fresh leaves was 0.365 ± 0.097 mg CA/g (d. b.). In general, the exposure to high temperatures during the drying leads to organic acid degradation, inducing the oxidation of those compounds. The TA values ranged from 0.152 ± 0.013 to 0.182 ± 0.022 mg CA/ g (d.b.). No difference was observed in that parameter, and this occurred probably due to the oxygen suppression during the vacuum processes (Oliveira et al., 2021).

CONCLUSIONS

Higher temperature and ethanol pretreatment promoted a shortened drying process. The D_{eff} ranged from 4.4×10^{-11} to 8.0×10^{-11} m²/s, and the Logarithmic and Midilli & Kuçuk equations performed best adjustments. The treatments presented a significant difference ($p \leq 0.05$) in the TPC, TAA and AA. There was no significant difference ($p > 0.05$) in pH values and titratable acidity. Preservation in the AA content and a reduction in the TPC and TAA were observed in pretreated samples.

AUTHORS CONTRIBUTION

Conceptual idea: Junqueira, J. R. J.; Corrêa, J. L. G.; Methodology design: Junqueira, J. R. J.; Resende, N. S.; Data collection: Junqueira, J. R. J.; Mendonça, K. S.; Data analysis and interpretation: Junqueira, J. R. J.; Corrêa, J. L. G.; Balbinoti, T. C. V.; Writing and editing: Junqueira, J. R. J.; Corrêa, J. L. G.; Gatti, I. P.

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