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ORIGINAL ARTICLE

Effects of different drying conditions on bioactive potential of Brazilian olive leaf

Efeitos de diferentes condições de secagem no potencial bioativo da folha de oliveira de origem brasileira

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Abstract

Olive leaf is a residue in olive oil and fruits production, which is considered with bioactive potential due to the high antioxidant activity attributed mainly to the phenolic compounds. The research aimed to investigate the Brazilian olive leaf drying, and also study its influence on the bioactive potential of the leaf. The desorption isotherms of olive leaves were determined and experimental curves were fitted to GAB, BET and Peleg models. Convective drying in a fixed bed dryer was used in different conditions of air temperature (50 °C and 70 °C) and air speed (0.9 $m\ s^{-1}$ and 1.5 $m\ s^{-1}$). Drying curves were obtained for each experimental test. The bioactive potential was reflected in the determination of total phenolic content, antioxidant activity and color parameters. Among the moisture equilibrium predictions between the GAB, BET and Peleg models, the first showed a better predictable capability. The results showed that in the drying operation, the increase in air temperature and speed influenced the increase in the drying rate and the reduction of time. The values of the effective diffusivity of the olive leaves varied between $2.61 \times 10^{-9}\ m^2\ s^{-1}$ and $10.12 \times 10^{-9}\ m^2\ s^{-1}$. The samples dried until 10% of moisture (wet-basis) showed a good antioxidant activity, higher than 85%, and maintenance of the phenolic compounds regarding the fresh leaves around 70%. Bleaching and yellowing of leaves after drying were observed for all studied conditions and a decrease in green color at 70 °C and 1.5 $m\ s^{-1}$, in this condition the L*a*b* color parameters were 35.39, -5.00 and 42.66, respectively. This study demonstrated that the proper drying condition was at 70 °C and 1.5 $m\ s^{-1}$ for olive leaf drying and these conditions were important to maintain the original characteristics of the leaves and to spend less time in operation. Thus, this paper describe a viable drying process to take advantage of the olive leaf for the benefit of the environment and human health.

Keywords: *Olea europaea* L.; Convective drying; Fixed bed dryer; Total phenolic content; Antioxidant activity; Desorption isotherms; Dehydration; Equilibrium moisture content.

Resumo

A folha de oliveira, um resíduo da produção do azeite e de seus frutos, é considerada com potencial bioativo devido à elevada atividade antioxidante, atribuída principalmente aos compostos fenólicos. O objetivo desta pesquisa foi investigar a secagem da folha da oliveira de origem brasileira, estudando sua influência no potencial bioativo da folha. Para tal, as isotermas de dessorção das folhas de oliveira foram determinadas. Curvas experimentais foram ajustadas aos modelos GAB, BET e Peleg. A secagem por convecção em leito fixo foi utilizada em diferentes condições de temperatura do ar (50 °C e 70 °C) e velocidade do ar (0,9 m s^{-1} e 1,5 m s^{-1}). Curvas de secagem foram obtidas para cada teste experimental. O potencial bioativo é refletido na determinação dos parâmetros de fenólicos totais, atividade antioxidante e cor. Dentre as previsões de equilíbrio de umidade, entre os modelos GAB, BET e Peleg, o primeiro tem uma capacidade de melhor previsibilidade. Os resultados mostraram que, na operação de secagem, o aumento da temperatura e da velocidade do ar acarretou aumento da taxa de secagem e redução do tempo de secagem. Os valores de difusividade efetiva das folhas de oliveira variaram entre 2,61 × 10^{-9} m^2 s^{-1} e 10,12 × 10^{-9} m^2 s^{-1}. As amostras secas até 10% de umidade (base úmida) apresentaram boa atividade antioxidante, superior a 85%, e manutenção dos compostos fenólicos em relação às folhas frescas em torno de 70%. Observaram-se o branqueamento e o amarelecimento das folhas após a secagem para todas as condições estudadas e a diminuição da coloração verde a 70 ºC e 1,5 m s^{-1}. Nessa condição, os parâmetros de cor $L^*a^*b^*$ foram 35,39, -5,00 e 42,66, respectivamente. Este estudo demonstrou que a condição de secagem adequada é a 70 ºC e 1,5 m s^{-1} para a secagem da folha da oliveira e mostra-se importante por manter as características originais das folhas e dispender menos tempo de operação. Assim, este trabalho descreve um processo de secagem viável para aproveitar a folha da oliveira em benefício do meio ambiente e da saúde humana.

Palavras-chave: *Olea europaea* L.; Secagem convectiva; Leito fixo; Fenólico; Atividade antioxidante; Isotermas de dessorção; Desidratação; Teor de umidade de equilíbrio.

Highlights

- Olive leaf is a residue of the production of olive oil and fruits;
- This residue is important due to its high antioxidant activity attributed mainly to its content of phenolic compounds, taking advantage of this product by a variety of industries such as food, pharmaceutical and cosmetics;
- To be used by the industry, the leaves need to be dried after collection for their best preservation;
- The present work provides valuable results for the industry that intends to use this raw material, showing the influence of the conditions of the convective drying process on the variation of bioactive compounds.

1 Introduction

Olive (*Olea europaea* L.) is a plant from the *Oleaceae* family, including plant species distributed in tropical and temperate regions, nevertheless this is also a consolidated species in the Mediterranean region (Giacometti et al., 2018; Putnik et al., 2017). However, this species has been expanding into other territories, especially in Brazil. The Brazilian lands proved to be good for the cultivation of the olive tree, and nowadays producing olive oils awarded worldwide. This demonstrates the social and economic importance of this crop and the emerging opportunity of using any of its by-products from olive crops (Cavalheiro et al., 2015; Rosa et al., 2021). According to an economic point of view, the interest for olive trees is linked to the production of olives and olive oil, but during this production, by-products are generated, thus being equally valuable, in liquid and solid fractions; whereas the solid fractions are related to the leaves and pomace and the liquid fraction is the mill wastewater, which can be valued by composing innovative products, considered to be a rich source of natural products such as high phenolic compounds. In addition, the reuse of by-products reduces the pollution caused by them, as well as making olive mill plants more sustainable (Elkacmi et al., 2017; Nunes et al., 2016).

Among these by-products, the olive leaves stand out, which are generated during the processing of the fruit, at harvest and in pruning, in large quantity, about 25 kg of leaves and branches, per tree, from the pruning activities (Lamprou et al., 2020). The olive oil plant, where the leaves are separated from the olives by a blowing machine, can represent between 4% and 10% by weight of the olive that goes into processing. According to the latest Food Agriculture and Organization (FAO) report that surveyed the sector, 21.06 Mt of olive fruits are processed every year (Food Agriculture and Organization, 2020) leading to the generation of 0.84 Mt of olive leaves. Only in Spain, 0.2 million tons of olive leaves are generated per year (Lama-Muñoz et al., 2019; Romero et al., 2018; Selin et al., 2018). In Brazil, in the 2018/2019 harvest, with a production area of 1.500 hectares, 1.700.000 kg of olives were harvested and 198.664 liters of oil were produced, which represents a substantial amount of leaves generated as a by-product. The current production of oils in Brazil is still small and typical of a nascent industry, however, it already has significant international prominence and growth prospects (International Oliviculture Council, 2020; Costa, 2019).

The reuse of the olive leaf is important for waste management, thus contributing to the environment. The leaf has potential for conversion into useful products of high added-value, especially with regard to the benefits to human health due to its composition rich in bioactive compounds (Ahmad-Qasem et al., 2016; Erbay & Icier, 2009b). The investigation on the use of olive leaves, in the most diverse industrial sectors, is relevant. Some of its applications include the development of natural drugs, functional foods and natural food preservatives (Ahmad-Qasem et al., 2016; Martiny et al., 2020b). However, the presence of phenolic contents and antioxidant activity in olive leaves have been reported (Abaza et al., 2011; El & Karakaya, 2009; Lee & Lee, 2010), but very few studies are available on methods to preserve them. And when it comes to the olive tree of Brazilian geographical origin, there are no studies.

Studies indicated that the biological activity of the compounds present in olive leaves is associated with the treatment of diseases, such as fever, inflammation and hypertension (Kermanshah et al., 2020). In view of the current pandemic situation triggered by the Coronavirus Disease 2019 (COVID-19), a study has shown that oleuropein (phenolic compound) extracted from olive leaf can act as an antiviral agent (Coppa et al., 2017; Omar, 2010; Sun & Ostrikov, 2020). The olive leaves medicinal properties are mainly referred to as phenolic compounds, which constitute an important group of antioxidant compounds, formed by a large group of chemical substances. Phenolic compounds are considered secondary plant metabolism products, usually derived from defense reactions against environmental aggressions, with different chemical structures and activities (Martínez-Valverde et al., 2000). Olive leaves have substantial amounts of phenolic compounds such as oleuropein (the most common compound), verbascoside, luteolin-7-*O*-glucoside, hydroxytyrosol, vanillin and rutin in their composition, resulting in bioactive properties (Giacometti et al., 2018; Kiritsakis et al., 2017). In this sense, it is important to study techniques that assist in the preservation of these compounds in olive leaves and that enable their application. Among these techniques is the drying operation.

The drying process of vegetal products that are used by industry as raw material is important and must be applied after the harvest for a better preservation, since the reduction of moisture content prevents the action of deteriorating agents such as enzymes and microorganisms (Geankoplis, 1998; Martinazzo et al., 2010). Moreover, depending on the heat treatment, the drying of plant products can promote the undesirable reduction of constituents of interest or even changes in product characteristics (Erbay & Icier, 2010). In this sense, one of the challenges of the industry in the olive leaf drying is the knowledge of the best drying conditions to decrease losses of the bioactive compounds. Convective drying has been commonly used for dehydration of vegetal products over the years (Calín-Sánchez et al., 2020).

Convective drying uses hot air as a heat transfer agent and dewatering the products. Some advantages of this technique are: cheap method; hygiene promotion; uniformity; simplicity; convenient; affordability; and it improves drying process (Calín-Sánchez et al., 2020; Senadeera et al., 2020). The transfer of mass of water from the product to the medium is called desorption. The study of this phenomenon is extremely important for the industry, to avoid undesirable transformations due to water loss. The determination of equilibrium isotherms under different air conditions is a fundamental part of studies involving drying. Isotherms indicate the end point of the process at which the balance between the solid and gaseous phase is established and the minimum moisture content that a material will reach under certain conditions of temperature, pressure and relative humidity, thus aiming at optimal drying conditions. Based on the acquisition of equilibrium data, it is possible to obtain important information regarding the association of moisture with the solid and to identify the predominant vapor sorption mechanism from specific air conditions (relative humidity, temperature and pressure). In addition, many mathematical models use the difference between the material's actual moisture content and the equilibrium content as a driving force for mass transfer. All the necessary information regarding equilibrium moisture in the context of drying can be known from the moisture desorption isotherms (Calín-Sánchez et al., 2020; Hubinger et al., 2009; Senadeera et al., 2020).

This work aimed to study the drying process of Brazilian olive leaves using convective drying in fixed bed dryer in order to determine its impact on the bioactive values (total phenolic compounds and antioxidant activity) and color parameters, thus aiming to propose a method for the leaves preservation and their later use.

2 Materials and methods

2.1 Raw material

The olive leaf (*Olea europaea* L.) cultivar Arbequina were obtained from a private farm Estância Guarda Velha, located in Pinheiro Machado, in the state of Rio Grande do Sul (RS), in Brazil (31º30'04.0”S, 53º30'42.0”W).

2.2 Chemicals

Folin Ciocalteu’s, anhydrous sodium carbonate and 2,2-diphenyl-1-picrylhydrazyl (DPPH) were obtained from Sigma Aldrich (St. Louis, USA).

2.3 Desorption Isotherms

The desorption isotherms were performed to determine the equilibrium moisture content of the olive leaves under the conditions of drying experiments. The tests were performed in triplicate by the gravimetric static method using acid solutions with various concentrations to keep the relative humidity from 0.05 to 0.88. The isotherms were determined at temperatures of 50 ºC and 70 ºC (drying temperatures).

Three mathematic models were fitted to the desorption isotherms data obtained experimentally: BET (Equation 1); GAB (Equation 2); and Peleg (Equation 3). Non-linear regression was done using the experimental data of equilibrium isotherms of leaves, using the software Statistica 7.1, obtaining an estimation of the model’s parameters. The satisfactory data fit was evaluated by the determination coefficient (R^2) and mean relative error (P).

$$X_e = \frac{X_m . C_B . RH}{(1 - RH).(1 - RH + C_B . RH)} \tag{1}$$

where X_e corresponds to the equilibrium moisture content (d.b.), X_m to the monolayer moisture content, C_B to the constant associated with sorption in the first layer and RH with the relative humidity.

$$X_e = \frac{X_m . C_B . RH . K_S}{(1 - K_S . RH).(1 - K_S . RH + C_B . K_S . RH)} \tag{2}$$

where K_S corresponds to the constant related to multilayer sorption.

$$X_e = a.RH^b + c.RH^d \tag{3}$$

where a, b, c and d correspond to the empirical constants of the model.

Parallel air flow was used in a fixed bed dryer (EcoEducacional, Brazil), as shown in Figure 1. The air flow coming from the blower (Artek, 1CV, Brazil) (Figure 1a) was monitored by a psychrometer (Figure 1b) and was heated by an electrical heater (Figure 1c). Measurements of air speed were performed by an anemometer (Figure 1f) and controlled by a frequency inverter on the control panel (Figure 1d). In order to check the mass of leaves samples during the drying experiments, a digital balance (Figure 1e) was used, connected with a tray (thickness of 13.3 mm and a diameter of 139.2 mm) in the drying chamber (Figure 1g).

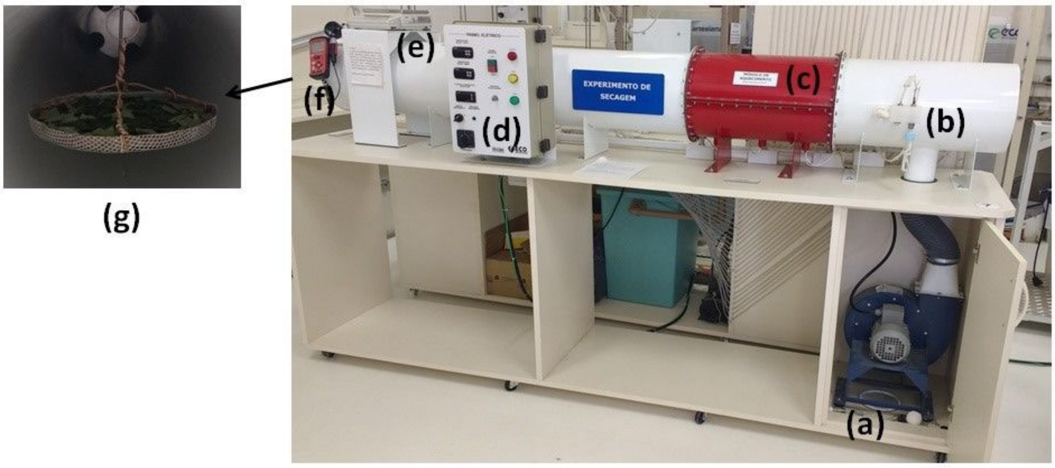

Figure 1. Fixed bed dryer and parallel air flow.

2.4 Drying experiments

Drying experiments were performed in four different conditions, varying the air temperature and the air speed drying (Table 1). For each drying assay was used 30 g of olive leaves. The leaves were cut to smaller sizes with a surface area of approximately 1 cm^2 and were dried until a final moisture content of 10% (wet-basis). The sample mass value was checked every 1 min in the first 120 min, and every 3 min after this period. The values of the dry-bulb and wet-bulb temperatures of the air before heating were checked every 5 min. The average relative humidity of the heated air was determined using a psychrometric chart and the equilibrium moisture content of the leaves was obtained from the desorption isotherms.

Table 1. Conditions of drying tests.

Test	T_{air} (°C)	v_{air} (m s^{-1})
1	50.0	0.90
2	70.0	0.90
3	50.0	1.50
4	70.0	1.50

T_{air} - air temperature; v_{air} - air velocity.

Drying curves were obtained for each test: free moisture dimensionless (Equation 4) varying with the time and drying rate, and also (Equation 5) varying with the moisture content in dry-basis (d.b.).

$$MD = \frac{X_t - X_e}{X_{to} - X_e} \tag{4}$$

where MD corresponds to the free moisture dimensionless, X_t to the moisture content at a certain instant (d.b.), X_{to} to the initial moisture (d.b.) and X_e corresponds to the equilibrium moisture content (d.b.).

$$R = -\frac{L_S}{A} \cdot \frac{dX}{dt} \tag{5}$$

where R is the drying rate, A is the area of the material being dried, L_s is the dry mass of the material, X is the moisture content (d.b.) and t is the time.

The theoretical model of Fick´s second law obtained by Crank (1975), considering a flat geometry, was used to represent the thin layer drying, as presented in Equation 6. By adjusting the model to the experimental data of the free moisture dimensionless as a function of time it was possible to obtain the effective diffusivity value for each drying condition.

$$MD = \frac{8}{\pi^2}\sum_{n=0}^{\infty}\frac{1}{(2n+1)^2}exp\left[-(2n+1)^2\frac{\pi^2 D_{ef}}{e_o^2}t\right] \tag{6}$$

where e_0 corresponds to the bed thickness, D_{ef} to the effective diffusivity and n to the number of terms in the series.

2.5 Analytical methodology

2.5.1 Determination of total phenolic contents

The total phenolic contents from Brazilian olive leaf were obtained according to the Folin-Ciocalteau colorimetric method proposed by Singleton & Rossi (1965). Briefly, 1 g of sample was used, crushed in an analytical mill, then selecting only the particles that had passed through a sieve with a mesh opening of 1 mm. The extraction was performed with 50 mL of methanol in a time of 30 min under agitation (90 rpm). After extraction, the mixture was filtered on qualitative filter paper (80 g m^{-2}). In 1 mL aliquots of the extract, 10 mL of distilled water and 1 mL of the Folin-Ciocalteau reagent were added. Afterwards, 8 mL of a sodium carbonate solution (7.5% m/v) were added. After 2 h, the absorbance of the solution was read at 760 nm in a UV spectrophotometer (Equilam, 755B) using a solution without the extract as blank. Total phenolic contents of extracts were expressed in milligrams of Gallic Acid Equivalent (GAE) per gram of dry matter. The analysis was performed in triplicate.

2.5.2 Evaluation of *in vitro* antioxidant activity

The antioxidant activity was obtained using the method proposed by Brand-Williams et al. (1995), which is consisted on the reduction of the radical 2,2-diphenyl-1-picrylhydrazyl (DPPH). Briefly, 100 µL of the extract was mixed to a 3.9 mL DPPH solution and after 30 min of reaction, the absorbance was read at 517 nm. The Antioxidant Activity (AA) was expressed in terms of the percentage of inhibition of the sample on the DPPH radical, according to Equation 7. The analysis was conducted in triplicate.

$$AA(\%) = \left(\frac{A_{control} - A_{sample}}{A_{control}}\right)\cdot 100 \tag{7}$$

where, $A_{control}$ is the absorbance of the blank solution (with water), and A_{sample} is the absorbance of the extract solution.

2.5.3 Color parameters

The color parameters of the olive leaves were determined with the crushed samples in analytical grind, using a colorimeter (Konica Minolta, D2008304, Japan) with a D65 illuminator and 10º angle, previously calibrated, with reference to the CIE Lab system. The parameters analyzed were L^*, which corresponds to the luminosity (black 0 / white 100), a^* (green - / red +) and b^* (blue - / yellow +). Through the variation of the color parameters with the drying, it was possible to calculate the total color variation (ΔE) associated to the process according to Equation 8 (Corrêa et al., 2012).

$$\Delta E = \sqrt{(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2} \quad (8)$$

where ΔE corresponds to the total color variation and L^*, a^* and b^* to the color parameters.

2.6 Morphological analysis

Olive leaves images were recorded using a stereomicroscope microscope (Motic, K Series, Canada), where it was possible to compare the surface of the dried and fresh samples.

2.7 Statistical Analysis

The total phenolic compounds, antioxidant activity and color parameters L^*, a^* and b^* were evaluated for the fresh and dried leaves. By using the Tukey's test, and considering confidence of 95% (*p-level* < 0.05), the statistically significant differences between the averages for fresh and dried samples were evaluated.

3 Results and discussion

The results of the desorption isotherms of the olive leaves are presented in Figure 2, where can be verified that the curves showed a sigmoidal behavior and, according to Brunauer et al. (1940), these curves could be classified as type II isotherms. Similar results were observed for the olive leaves by Bahloul et al. (2008) and microalgae Spirulina platensis by Oliveira et al. (2009).

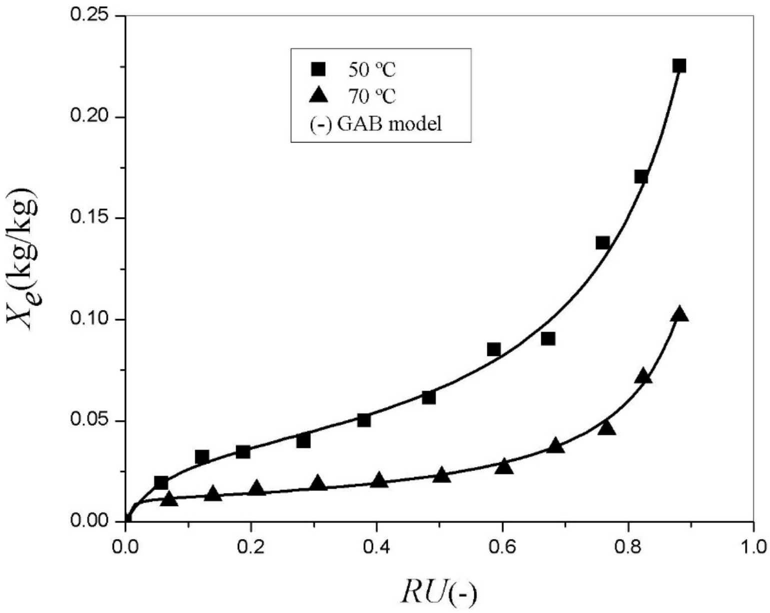

Figure 2. Desorption isotherms of the olive leaf with data adjusted to the GAB model. Xe - equilibrium moisture content (d.b.); *RH* - Relative Humidity.

Table 2 shows the parameters of the mathematical models (Equations 1 to 3) adjusted to the experimental data, as well as the values of P and R^2. The parameter X_m is the value of the moisture content in the monolayer formation and, according to Table 2, this parameter decreased with the increase of temperature, because of the increment in the state of agitation of the molecules. This parameter corresponds to the ideal moisture content for storage and control of possible deterioration reactions (Kaya & Kahyaoglu, 2007). The GAB model was chosen to represent the behavior of the experimental data and to obtain the equilibrium moisture content for drying tests, in addition to the good fitting, it has shown physical parameters associated with the structure of the material. Moreover, according to Hassini et al. (2015), the model is widely used to describe vegetable product isotherms.

Table 2. Parameters of the isotherms models adjusted to the experimental data.

Parameter	50 °C	70 °C
	BET	
X_m	0.033	0.012
C_B	24.25	46.28
R^2	0.989	0.987
P (%)	4.77	4.21
	GAB	
X_m	0.037	0.011
C_B	15.11	186.23
K_S	0.949	1.009
R^2	0.995	0.994
P (%)	5.41	6.03
	Peleg	
C	0.07	0.21
D	0.42	8.25
E	0.29	0.02
F	5.20	0.39
R^2	0.996	0.997
P (%)	5.17	2.98

BET - Brunauer, Emmett and Teller; GAB - Guggenheim, Anderson and de Boer; C, D, E and F - empirical constants; C_B - constant related to sorption in the first layer; K_S - constant related to multilayer sorption; P - mean relative error; R^2 - correlation coefficient; X_m - monolayer moisture content.

Table 3 shows the values of the air properties for each drying experiment. It can be observed that the values of the average relative humidity of the air after heating ($RH_{2average}$) decreased as the temperature of the drying air (T_{air}) increased, regardless of the value of the average relative humidity of the air before heating ($RH_{1average}$). This decrease causes an increase in the difference in moisture concentration between the drying air and the solid material, thus increasing the driving force of the process and facilitating the transfer of moisture. The average equilibrium moisture content for the tests decreased as the drying air temperature raised and the average air relative humidity decreased after heating, showing consistency with the physical process, as demonstrated in Figure 2.

Table 3. Air properties for each drying test.

Test	T_{air} (°C)	v_{air} (m.s^{-1})	$T_{DBaverage}$ (°C)†	$T_{WBaverage}$ (°C)†	$RH_{1average}$ (%)†	$RH_{2average}$ (%)†	$X_{e\ average}$ (kg kg^{-1})†
1	50.0	0.90	23.73 ± 0.24	19.59 ± 0.32	68.48 ± 2.33	16.28 ± 0.51	0.032 ± 5.10^{-4}
2	70.0	0.90	23.50 ± 0.05	19.98 ± 0.32	72.75 ± 2.28	6.76 ± 0.21	0.011 ± 5.10^{-5}
3	50.0	1.50	23.32 ± 0.18	21.26 ± 0.18	83.43 ± 0.66	19.38 ± 0.24	0.035 ± 2.10^{-4}
4	70.0	1.50	22.93 ± 0.23	20.15 ± 0.07	77.80 ± 1.72	6.98 ± 0.06	0.011 ± 1.10^{-5}

†Average values (n=3) ± standard deviation; $RH_{1average}$ - average relative humidity of the air before heating; $RH_{2average}$ - average relative humidity of the air after heating; T_{air} - air temperature; $T_{DBaverage}$ - average dry bulb temperature; $T_{WBaverage}$ - average wet bulb temperature; v_{air} - air velocity; $X_{e\ average}$ - average equilibrium moisture content (d.b.).

The drying curves are presented in Figure 3, where it can be observed the effect of the variables of the process, temperature and air velocity on the drying time. When the temperature and the drying air velocity increased, a decrease in the drying time was observed. This behavior was also verified in convective fixed bed dryer of guaco leaves (Silva, 2014) and olive leaves (Erbay & Icier, 2009b). The physical consideration related to the influence of the increase in temperature is regarding the decrease of the relative humidity of the air, increasing the humidity gradient between the leaf and the drying air. In addition, the higher temperature difference between the leaf and the drying air contributes to an increase in the rate of heat transfer. In relation to the increase in air velocity, its effect is mainly related to the increase of the convective portion related to heat transfer and mass transfer between the leaf and the drying air (Oliveira, 2015). It can also be seen that the temperature showed a higher reduction in the drying time than the air velocity; this fact is consistent with the physics of the phenomenon since the internal resistance to mass transfer is superior to the influence of the external resistance caused by the convective.

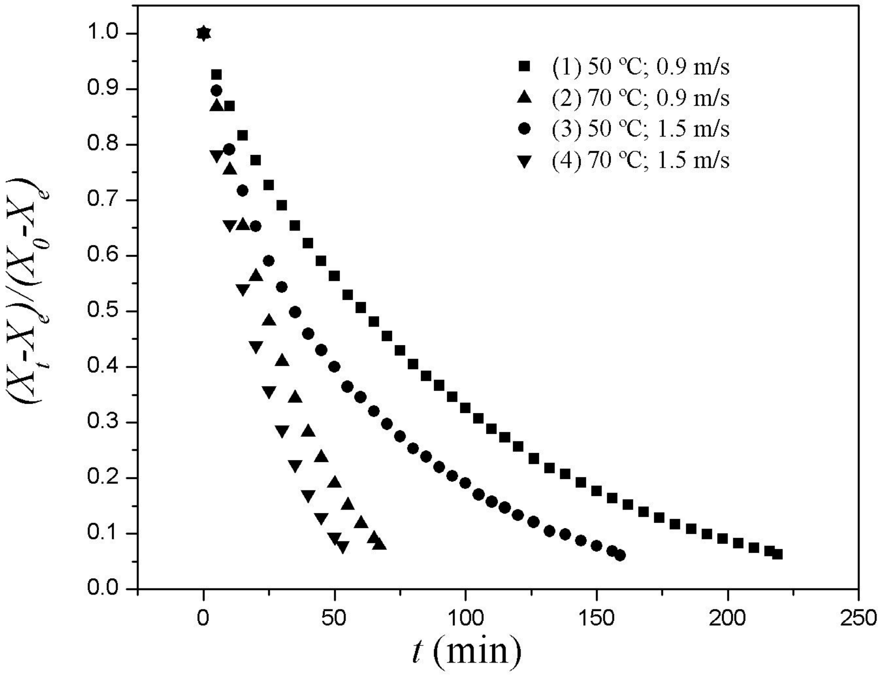

Figure 3. Free moisture dimensionless as a function of time. t - time; X_e - equilibrium moisture content (d.b.); X_t - moisture content at a certain instant (d.b.); X_o - initial moisture content (d.b.).

Figure 4 shows the curves of drying rate as a function of the moisture content (d.b.). It can be noticed that the presence of constant drying rate was not verified, being this consistent with the literature, since in many fibrous vegetable materials, moisture is retained as part of the structure of the solid, not being present in quantities sufficiently high in the pores or surface (Foust et al., 1982). Furthermore, the mass transfer resistances are essentially inside the product, causing the evaporation rate at the surface to be higher than the rate of replacement of moisture from inside to the surface area of the material (Park et al., 2007). The lack of a constant rate period in convective fixed bed dryer of leaves was also verified by Erbay & Icier (2009a) for olive leaves and by Martinazzo et al. (2010) for lemongrass leaves.

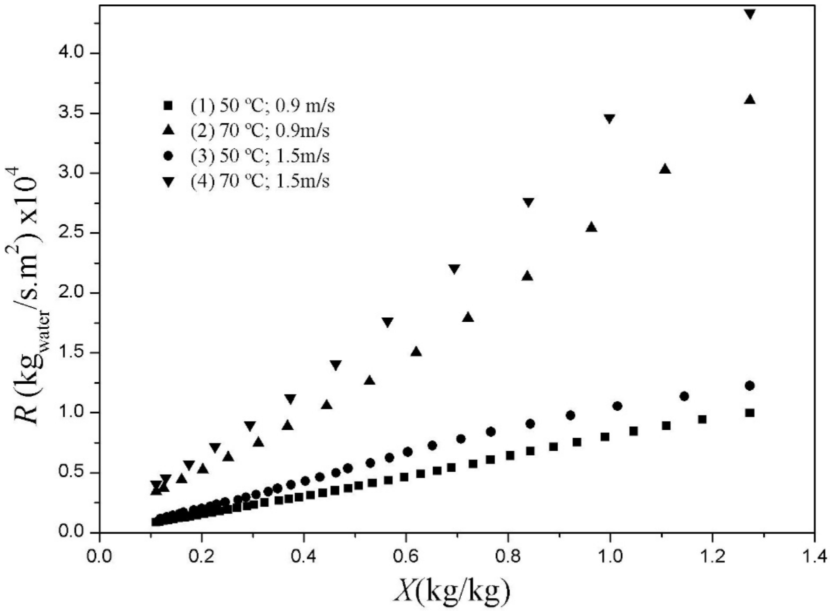

Figure 4. Drying rate as a function of moisture content. R - drying rate; X - moisture content (d.b.).

Table 4 presents the effective diffusivity values for each test, obtained from the adjustment of the model of Fick´s second law (Equation 6), as well as the R^2 values for each condition. The effective diffusivity values increased by increasing the temperature and the air velocity, being such behavior consistent with the process physics. The effective diffusivity at 70 ºC was 2.6 times higher than 50 ºC, demonstrating the influence of temperature on the internal migration of moisture content.

Table 4. Effective diffusivity obtained from Fick's second law model.

Test	T_{air}(ºC)	v_{air}(m s^{-1})	D_{ef}(m^2 s^{-1}) (.10^9)	R^2 (%)
1	50.0	0.90	2.61	93.00
2	70.0	0.90	7.32	90.70
3	50.0	1.50	4.24	96.22
4	70.0	1.50	10.11	93.97

D_{ef} - effective diffusivity; T_{air} - air temperature; v_{air} - air velocity; R^2 - correlation coefficient.

The results for the effective diffusivity were consistent with the values in the literature for the drying of plant products (10^{-11} to 10^{-9} m^2 s^{-1}) (Madamba et al., 1996). Erbay & Icier (2009b) reported for drying of olive leaves with temperature between 50 ºC and 70 ºC and air velocity between 0.5 m s^{-1} and 1.5 m s^{-1} values for effective diffusivity in the range of 1.05.10^{-9} to 4.97.10^{-9} m^2 s^{-1}. According to Nourhène et al. (2008), the drying of olive leaves with temperatures between 40 ºC and 60 ºC showed values for effective diffusivity between 2.95.10^{-10} m^2 s^{-1} and 3.6.10^{-9} m^2 s^{-1}.

Table 5 shows the values of Total Phenolic Compounds (*TPC*) and Antioxidant Activity (*AA*) for the fresh and dried samples. There were losses of *TPC* for all conditions of drying around 31%. According to Podsedek (2007), these compounds are sensitive to heat treatment. Boudhrioua et al. (2009) reported that the drying process promotes the rupture of the cell wall which may lead to the release of enzymes that oxidize the phenolic compounds, as well as promoting its decrease, being the polyphenoloxidase (PPO) enzyme one of the most reported in the literature. In the drying of olive leaves, Kamran et al. (2015) observed an increase in phenolic content at 105 °C compared to the temperature at 60 °C and attributed this behavior to the inactivation of the PPO enzyme at high temperature. This result may explain the non-observance of a significant difference between the drying conditions in the present work, since intermediate temperatures (50 ºC and 70 ºC) were used, a condition in which the enzyme is possibly still activated. According to Reis et al. (2013), it could be noted that a species of Cumari pepper drying at temperatures of 45 ºC to 65 ºC did not show a statistically significant difference for the *TPC*.

Table 5. Total Phenolic Compounds (TPC) and Antioxidant Activity (AA) for the fresh and dried Brazilian olive leaves.

Test	Condition	*TPC* (mg$_{GAE}$ g$_{ds}^{-1}$)†	*AA* (%)†
	fresh	28.44^a ± 1.26	92.73^a ± 0.11
1	50 ºC, 0.9 m s^{-1}	19.38^b ± 0.17	89.43^b ± 0.60
2	70 ºC, 0.9 m s^{-1}	18.74^b ± 0.52	88.03^{bc} ± 0.57
3	50 ºC, 1.5 m s^{-1}	19.62^b ± 0.23	87.49^{bc} ± 0.55
4	70 ºC, 1.5 m s^{-1}	19.77^b ± 0.32	87.15^c ± 0.70

†Average values (n=3) in the same column with different superscripts (*a*,*b*,*c*) are significantly different at ($p < 0.05$) on the basis of Tukey test; *AA* - antioxidant activity; ds - dried solid; GAE - gallic acid equivalent; *TPC* - total phenolic compounds.

It could be verified that there were statistically significant differences ($p > 0.05$) of the *TPC* and *AA* for dried samples compared to the fresh sample. Furthermore, a significant difference for *AA* was observed between the drying tests 1 and 4. This distinction can be related to the time of the drying process, and according to Figure 3, Test 1 (50 ºC, 0.9 m s^{-1}) showed the highest drying time (220 min), with the highest value of *AA*. According to Nicoli et al. (1999), a longer heating time can promote a recovery of the *AA* by the formation of new antioxidant compounds. Test 4 (70 ºC, 1.5 m s^{-1}) showed the shortest drying time (50 min), and the lowest *AA* can be explained by the losses without the formation of new products. Considering Hassimotto et al. (2005) classification for *AA*, which values above 70% indicated a good activity, values between 40% and 70% demonstrated an intermediate activity and values below 40% a low activity, it was found that both the fresh and the dried leaves in different conditions presented good *AA*.

Considering the *AA* and *TPC* of the leaves after the drying process, it could be inferred that the best drying condition was at 70 ºC and 1.5 m s^{-1}, since it was the condition that presented lower drying time and consequently represents a lower cost of the process.

Similarly, Elhussein & Şahin (2018) dried olive leaves in an oven at 50 °C and 80 °C, they obtained for TPC 35.77 $mg_{GAE}\ g_{ds}^{-1}$ and 30.22 $mg_{GAE}\ g_{ds}^{-1}$, and for AA 89.17% and 88.06%, respectively. In fact, these values are in agreement with those found in this research. They concluded that oven drying of olive leaves can be accepted as better when considering energy consumption when compared to other methods such as microwave drying. In a more recent study Helvaci et al. (2019) studied olive leaf drying and also used convective drying, their results indicated that the stability of the phenolic content was mainly affected by air temperature, whereas the antioxidant capacity was affected by both air temperature and by speed. The optimal drying conditions found were at 50 °C air temperature and 1 m s^{-1} air speed, this speed was close to the optimal condition of the present research which was 1.5 m s^{-1}, thus ensuring minimal losses of phenolic compounds and antioxidant activity. An important result was revealed in Afaneh et al. (2015) research, when they found that dried olive leaves had the highest content of oleuropein when compared to fresh leaves, denoting that drying of the leaves was necessary for a high recovery of oleuropein, which is an important compound phenolic found in this leaves.

Table 6 presents the values of the color parameters (*a**, *L** and *b**) for the fresh and dried samples, as well as the total color variation (*ΔE*). The parameter a* corresponds to chromaticity on a green (−) to red (+) axis, and b* to chromaticity on a blue (−) to yellow (+) axis. As noted, the drying process affected the color of the olive leaves. In the CIE L* a* b* color space, all parameters were significantly ($p < 0.05$) affected by drying under all conditions tested, except for the parameter a* in tests at 50 ºC. One value of b* plus high was registered in the dry samples, translating a more intense yellow color. These changes in color coordinates indicated that the olive leaves acquired a yellowish hue after drying.

Table 6. Color parameters and total color variation.

Test	Condition	(a*)†	(L*)†	(b*)†	ΔE†
-	fresh	$-7.67^{a} \pm 0.10$	$18.95^{a} \pm 0.81$	$32.02^{a} \pm 1.35$	-
1	50 ºC, 0.9 m s^{-1}	$-7.98^{a} \pm 0.14$	$39.54^{b} \pm 2.51$	$40.56^{b} \pm 0.75$	$22.34^{a} \pm 2.12$
2	70 ºC, 0.9 m s^{-1}	$-4.67^{b} \pm 0.44$	$28.86^{c} \pm 2.19$	$45.89^{c} \pm 2.33$	$17.35^{b} \pm 2.97$
3	50 ºC, 1.5 m s^{-1}	$-7.65^{a} \pm 0.06$	$38.43^{b} \pm 2.24$	$40.52^{b} \pm 1.61$	$21.38^{a} \pm 1.41$
4	70 ºC, 1.5 m s^{-1}	$-5.00^{b} \pm 0.28$	$35.39^{b} \pm 2.18$	$42.66^{bc} \pm 3.10$	$20.12^{ab} \pm 0.19$

†Average values (n=5) in the same column with different superscripts (*a*,*b*,*c*) are significantly different at ($p < 0.05$) on the basis of Tukey test; *L*, a** and *b** - color parameters; Δ*E* - total color variation.

Regarding the parameter *a**, no significant differences could be observed between the fresh leaf and the dried at 50 °C (test 1 and 3), with an increase for the tests at 70 °C (test 2 and 4), that was related to the decrease of the green color of the leaf, possibly as a function of the degradation of the chlorophyll by the heat treatment. This was also verified by Balasubramanian et al., (2011) in the drying of betel leaves and by Martinazzo et al. (2010) in the drying of leaves of lemon grass.

Increases in *L** and *b** parameters were observed for all drying conditions compared to fresh leaf, being related to bleaching and increase of yellow tone by the process, and this was also verified in the literature in the drying studies of leaves of "pitangueira" (Assis, 2012) and olive leaves (Boudhrioua et al., 2009). The total color variation (*ΔE*) were higher than 2, which indicated that the color variation could be observed visually (Rzaca & Witrowa-Rajchert, 2008).

The color parameters of plants matrices can reflect the type of molecules present in their composition. Thus, in this study, the color of the olive leaves was measured in order to assess the color tone and intensity. It was also important to understand the effect of the drying process in relation to the color of the leaf. The color variation in vegetable drying may be influenced by the degradation of chlorophyll content, carotenoids, as well as by enzymatic and non-enzymatic reactions during the process (Śledź et al., 2013).

The obtained results of the morphological analysis of the surface of the dried and fresh olive leaves are presented in Figure 5. It was possible to observe a preservation of the green color in the temperature of 50 ºC, being coherent with the variation of the previously verified color parameters. In the analysis by the magnification of 400x, an increase of the surface roughness of the dried leaves can be observed compared to the fresh. It can be observed also a higher roughness of the surface of sample dried at 70 ºC in comparison to 50 ºC, which can be explained by the higher drying rate. According to Silva (2014), it could be possible to carry out the drying of guaco leaves, and it has been also verified a higher roughness of the surface for conditions with a higher drying rate.

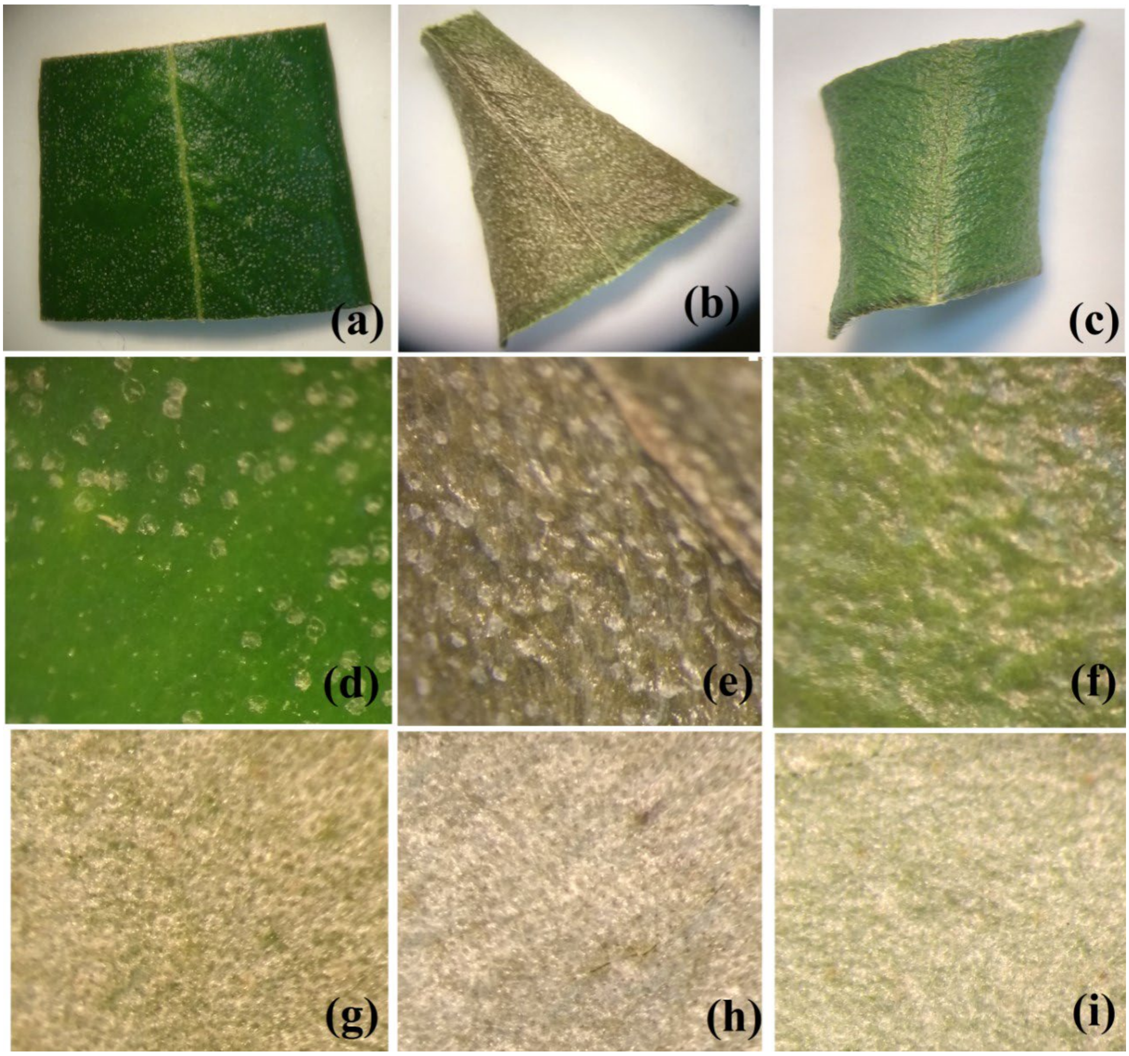

Figure 5. Morphological analysis of olive leaves through the stereoscope. a) fresh, upper surface, 65x; b) dried, 70 °C, upper surface, 65x; c) dried, 50 °C, upper surface, 65x; d) fresh, top surface, 400x; e) dried, 70 °C, upper surface, 400x; f) dried, 50 °C, upper surface, 400x.

It is worth mentioning that the drying process is essential when considering the sale of olive leaves and the production of their extracts. Several researches have explored the bioactive potentials of olive leaves through the production of their extracts and in many cases the drying stage precedes the extraction, being a pre-treatment stage of the leaves, as evidenced in the studies by Altiok et al. (2008); Giacometti et al. (2018); Marangoni et al. (2015); Martiny et al. (2020a, 2020b); and Şahin et al. (2017). Thus, it is evident that the study of drying olive leaves is very important.

4 Conclusions

Research and innovation are carried out in the processing of vegetable raw materials. Currently, consumers demand products with their functional properties and quality characteristics as close as possible to those of fresh plant material, in addition to long shelf life, and dry products meet these criteria. In this sense, the drying of valuable raw materials, such as olive leaves, is a matter of extreme importance to ensure physical, chemical and sensory quality. Thus, this work studied the effects of drying operation in the olive leaves of geographical origin in southern Brazil, desorption isotherms and drying characteristics in hot air have been established. The drying process of Brazilian olive leaves in a fixed bed dryer showed that increasing the air temperature and air velocity had an influence on the increase of the drying rate and a reduction of the processing time. The experimental drying data were fitted to three empirical mathematical models, and the GAB model described the best representation of the experimental drying values.

The values of the effective diffusivity obtained from the theoretical model of the second law of Fick were exposed in the range of $2.61.10^{-9}$ m^2 s^{-1} to $10.11.10^{-9}$ m^2 s^{-1}. The high antioxidant activity is strongly associated to the phenolic content of leaves and samples showed a good antioxidant activity, even after drying process. It was possible to observe that there were losses of phenolic compounds for all conditions of drying around 31%. According to the results for phenolic compounds and antioxidant activity of the leaves, there were significant difference between fresh and dried samples. It could be indicated that the adequate drying condition was at 70 ºC and 1.5 m s^{-1}, since it was the condition that showed lower drying time and consequently represented a lower cost of the process. Leaves whitening and yellowing after drying for all conditions were observed. The decrease in green tonality was only verified for the higher temperature (70 ºC). An increase in surface roughness can also be observed with drying from the morphological analysis.

The data of this research demonstrated that the Brazilian olive leaves had the capacity to supply compounds with bioactive potential even after drying, which can be used as a resource and should not be discarded as a residue, thus reinforcing the questions about sustainability in olive growing. The drying Brazilian olive leaf was representing a good way of preserving leaves exploiting their bioactive potential, so future uses of the leaves include the production of extracts with potential use in the food and pharmaceutical industries.

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