Conventional method and microwave drying kinetics of *Laurus nobilis* leaves: effects on phenolic compounds and antioxidant activity

Cinética da secagem convencional e por micro-ondas de folhas de *Laurus nobilis*: efeitos sobre os compostos fenólicos e a atividade antioxidante

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Abstract

*Laurus nobilis* constitutes one of the usually studied Mediterranean trees due to its therapeutic effects, nutritional properties, and ornamental use. In the current investigation, a comparative study was conducted between two types of drying methods, a microwave-assisted drying (MWD) (from 180 to 900 W) and a conventional method (open-air and oven-drying using temperatures from 40 °C to 120 °C). Drying kinetics were carried out as a function of moisture content according to time. Total Phenolic Content (TPC) as well as reducing power of obtained products were determined. Kinetic results showed that the time required for dried bay leaves became shorter as temperature and microwave power increased; drying with the microwave was much more efficient than conventional methods (open-air and oven-drying). Antioxidants of the *Laurus nobilis* leaves were better conserved using open-air and microwave drying. Our results underlined a significant correlation between TPC and reducing power. Microwave drying using 300 W during 130 s was the most efficient combination that provided dried bay leaves with the highest content of phenolic compounds and antioxidant activity.

Keywords: Bay leaf; Open-air drying; Oven drying; Microwave drying; Phenolic compounds; Antioxidant capacity.
Resumo
O loureiro, Laurus nobilis, constitui uma das árvores mediterrânicas geralmente estudadas devido aos seus efeitos terapêuticos, propriedades nutricionais e uso ornamental. Na atual investigação, realizou-se um estudo comparativo entre dois tipos de métodos de secagem, uma secagem assistida por micro-ondas (de 180 a 900 W) e um método convencional (secagem ao ar e em forno usando temperaturas de 40 °C a 120 °C). Estudou-se a cinética de secagem determinando o teor de umidade em função do tempo. Determinou-se também o teor em fenóis totais (TTF), bem como o poder redutor dos produtos obtidos. Os resultados cinéticos mostraram que o tempo necessário para a secagem das folhas de louro diminui à medida que a temperatura e a potência do micro-ondas aumentam; secar com o micro-ondas é muito mais eficiente do que os métodos convencionais (ao ar e em forno). As propriedades antioxidantes das folhas de Laurus nobilis mantiveram-se melhor quando se usou a secagem ao ar e por micro-ondas. Os nossos resultados sublinharam uma correlação significativa entre o TTF e o poder redutor. As condições mais eficientes de secagem que permitiram obter folhas de louro secas com o maior conteúdo de compostos fenólicos e a melhor atividade antioxidante consistiram na secagem por micro-ondas usando 300 W durante 130 segundos.

Palavras-chave: Folha de louro; Secagem ao ar livre; Secagem em forno; Secagem por Microondas; Compostos fenólicos; Capacidade antioxidante.

1 Introduction
The bay (Laurus nobilis L., Lauraceae family) is a largely studied medicinal plant regarding its phytochemical constituents (Chahal et al., 2017; Caputo et al., 2017) and therapeutic virtues such as treatment of several neurological, dermatological, and urological disorders as well as gastrointestinal issues like epigastric bloating, impaired digestion, flatulence, and eructation (Fidan et al., 2019; Batool et al., 2020).

Drying constitutes one of the oldest methods used by humans for food preservation, and also taking a prominent place in modern food processing (Maroulis & Saravacos, 2003; Li et al., 2010). The procedure of drying consists of moisture reduction to a level that permits safe storage for an extended period. Furthermore, this method allows a substantial reduction in volume and weight too, minimizing packaging material, storage space, and transportation costs. The growing demand for shelf-stable and high-quality dried products requires design, simulation, and optimization for the drying process (Di Scala & Crapiste, 2008). In addition, conventional drying involves exposing processed food to elevated temperatures and prolonged treatment periods, leading to aroma modifications, color changes, and nutrients loss of the treated material (Ozkan et al., 2007; Dwivedy et al., 2012).

For these purposes, the development of new methods and processes of drying is crucial (Correia et al., 2015). During the recent few decades, microwave drying method has taken more and more importance in drying process than conventional ones. This drying method possesses several advantages such as a raised drying rate and minimized processing energy with reducing water level (Sharma & Prasad, 2004; Tulasidas et al., 1995).

The aim of the current investigation was to draw, on one hand, a comparison-between microwave and conventional (open-air and oven-drying) methods. On the other hand, we planned to study their effects on phenolic compounds and antioxidant activity of L. nobilis leaves.
2 Materials and methods

2.1 Plant material

Samples of bay leaves, used in the drying experiments and collected from Oued-Ghir area in the Bejaia province (Algeria), were first washed by tap water and then with distilled water. After that, the leaves were wiped with an absorbent paper and stored at a lower temperature until starting the drying process.

2.2 Air drying

Bay leaves, dried under open-air, were placed on drying beds at a room in a single layer and raised 1.5m from the ground. Psychometric conditions during the drying period for temperature and relative humidity were 12 °C to 27 °C and 65% to 75%, respectively.

2.3 Oven drying

Different aliquots of bay leaves (5g each) were dried in an oven (MEMMERT, Germany) using temperatures of 40, 60, 80, 100, and 120 °C. The oven was characterized by the interior of heating chamber with dimensions of 56 (width) x 40 (length) x 48 cm (height). The bay leaves were spread on a wire mesh tray and placed on top of drying chamber with an air velocity of 1.5 m/s. The variation of moisture content was measured by an analytical balance (RADWAG, Poland) correlated with sensibility fitting parameter for 0.1mg each 5 min (60 °C), 2 min (80 °C) or 1 min (100 °C and 120 °C).

2.4 Microwave drying kinetics

The drying process was achieved using a microwave oven (NN-S674MF model, Samsung, Malaysia) characterized by cavity dimensions of 22.5 cm (width) × 37.5 cm (length) × 38.6 cm (height). The microwave was equipped with a digital control system for irradiation time and microwave power (100 to 1000 W) and a working frequency of 2450 kHz (Dahmoune et al., 2013).

For the drying process, 5 g of bay leaves were dried in the microwave using five variable microwave powers (180, 300, 450, 600, and 900 W). The mass of leaves was periodically recorded (every 5 seconds) till reaching the stability.

The results of the drying kinetics, for the three methods (open-air, oven-drying, and microwave), were expressed as a function of moisture content and reported in percentages (%). Analyses were performed in triplicate and the initial moisture content was determined by drying in oven at 105 °C/24 h.

2.5 Grinding and sieving of leaves

Dried leaves, obtained from each drying process, were ground by an electrical crusher (IKA A11 basic, Germany) and collected powders were then sieved. Powder fractions with particle size <125 μm were stored in glass vials, sealed and kept in dark until needed for antioxidant extractions.

2.6 Antioxidants extraction

An aliquot of powder (1g) was extracted using 28 mL of solvent (50% ethanol). The extraction was performed by the microwave according to the protocol of Dahmoune et al. (2013). The obtained extracts were paper filtered (Whatman n°1) and kept at the refrigerator for further analyses.
2.7 Total phenolic content

The Total Phenolic Content (TPC) was determined according to the method of Singleton & Rossi (1965). An aliquot of extract (500 μL) was mixed with 2.5 mL of Folin-Ciocalteau Reagent (FCR) (10%) and 2 mL of sodium carbonate (7.5%) was added after two minutes. The mixture was incubated in darkness for 15 min under the temperature of 50 °C. The absorbance was read at 760 nm using a UV-Vis spectrophotometer (Spectro Scan 50, Nicosia, Cyprus). The results were expressed as mg Gallic Acid Equivalent (GAE) per g according to the calibration curve obtained by gallic acid \((y = 10.80x + 0.021)\).

2.8 Reducing power

The extract (0.5 mL) was mixed with 2.5 mL of phosphate buffer (0.2 M, pH 6.6) and 2.5 mL of 1% potassium ferricyanide. After incubating at 50 °C for 20 min of the obtained mixture, 2.5 mL of 10% trichloroacetic acid (TCA) was added. Then, 2 mL of the last mixture was homogenized with 2 mL of distilled water and finally, 0.5 mL of 0.1% ferric chloride was added. The absorbance of the resulting solution was measured at 700 nm (Pan et al., 2010). The results were expressed as mg GAE per g according to the calibration curve of gallic acid \((y = 2.71x + 0.002)\).

2.9 Statistical analysis

Experimental results were expressed as means ± standard deviation from triplicate analysis. The STATISTICA 5.5 software was used to compare data by Analysis of Variance (ANOVA), Least Significant Difference test (LSD test) and differences between means were considered statistically significant at \(p < 0.05\).

3 Results and discussion

3.1 Kinetics of drying

Drying of products guarantees water removal and better storage while preserving nutritional quality and bioactive compounds. In fact, water constitutes a key factor determining the degradation of phenolics through chemical oxidation and enzymatic action such as polyphenol oxidases that modified their structures (Tomás-Barberán & Espín, 2001). Conventional (open-air and oven-drying) and microwave drying were adopted methods in the current study aiming to compare the performance of the two procedures.

The most common natural drying method is air drying, due to its simplest, affordable, and easily accessible means for resource-constrained households to preserve seasonal products (Maseko et al., 2019). In order to avoid drying difficulty during the cold periods, oven-drying constitutes an appropriate procedure that could retain more bioactive compounds than sun drying, reduces drying time, and improves sensory attributes such as color and texture (Mdziniso et al., 2006). The range of oven temperature variations used in the present study was from 40 to 120 °C. The use of temperatures below 40 °C leads to the approximation of the air temperature and the use of temperatures higher than 120 °C leads to the rapid burning of the product.

Figure 1 illustrates the drying kinetics of bay leaves using open-air and oven-drying. Fresh bay leaves had low water content (57%) comparatively to the most plant leaves used in culinary preparations such as coriander (90%), mint (85%), and parsley (82%) (Alibas et al., 2019; Patil et al., 2019; Alishah et al., 2018).

The water content of the sample, dried in the open-air, gradually decreased to stabilize from the 4th day at a value of 26.10%. Drying with the oven at 40 °C allowed a faster and more effective moisture decrease of bay leaves than open-air; the water content stabilized after three days of drying at the level of 8.69%. At the
start of drying, free water was easily removed from the sample compared to the end of the process when remained water becomes bound to different plant structures, therefore it evaporates with difficulty.

The drying temperature had a significant effect on the drying kinetics of bay leaves; an increase of drying temperature reduced the time required for drying. Oven-drying using temperatures of 60 °C to 120 °C allowed reaching practically identical levels of moisture, but the time required was even shorter as temperature increased. The use of very high temperatures, the boiling temperature of water (100 °C) or higher (120 °C), caused an accelerated elimination of the water of bay leaves. However, recourse to temperatures below the boiling point of water showed an extension of drying kinetics.

Four days was the required time for drying under open-air. Using the oven, the time necessary to dry leaves decreased with increasing temperature, therefore, this procedure has required three days to reach the temperature of 40 °C, whereas 80, 48, 32, and 25 min were necessary to reach the temperatures of 60, 80, 100, and 120 °C, respectively. These findings could agree with those of Misha et al. (2013) who found that the times necessary for using kenaf core drying were 56, 75, and 127 min at 55, 50, and 45 °C, respectively. Concerning coriander leaves, Hihat et al. (2017) used the oven at the same temperatures as in our study, found that moisture loss had also a proportional relationship with temperature increase and inversely proportional to the time required for drying.

The final water content of the bay leaves depended on the temperature used for drying; in other words, it was higher when the temperature was lower. Indeed, the drying in open-air (about 23 °C) produced bay leaves with a water content of 26.01%, while the use of high temperatures (80 °C to 120 °C) allowed reducing moisture content to a very low level (about 5%).

**Figure 1.** Comparison between open-air and oven at 40°C (A) and oven at 60°C to 12°C (B) drying kinetics of *Laurus nobilis* leaves.
3.2 Microwave-assisted drying kinetics

Drying curves obtained by different microwave powers are presented in Figure 2. Drying kinetics of *L. nobilis* leaves showed that moisture content decreased rapidly at the start of the microwave drying process and further, it gradually decreased until the end of the drying. The statistical analysis revealed that in the exception of 180 W, all other microwave powers (300 to 900 W) displayed the same drying efficiency. Indeed, the moisture content of bay leaves dried using the power of 180W was stabilized at 20.49% after 170s. It was important to note that the drying with the microwave powers from 300 to 900 W showed a stabilization after 130 s at an average moisture level of 9.1%. According to the obtained results, it could be observed that the drying rate of bay leaves remained practically the same if a certain energy threshold was reached. The obtained results were not completely in accordance with those found by Kumar et al. (2014) who showed that the time required to achieve moisture equilibrium (6%) of ginger slices decreased when the microwave power increases. It should be mentioned that 300W was the most appropriate power for drying bay leaves, since it was similar to higher powers (450 to 900 W), in terms of time and drying efficiency and with less energy consumption. The results obtained in the present investigation showed that, as compared to conventional method, the drying time required to achieve equilibrium moisture content can be significantly shortened by microwave.

The use of low temperatures (as in the case of open-air) and microwave power (e.g. 180 W) were not effective for drying due to low energy. So, for suitable drying, a certain energy threshold must be exceeded to evaporate the water of the bay leaves. On the one hand, this energy must be sufficient to oppose the hygroscopic effect exerted by the constituents of the product, especially sugars, on water molecules and; on the other hand, the surrounding humidity. Therefore, the increase of the temperature power of drying caused the reduction of relative humidity thus increased water evaporation (Misha et al., 2013).

The principle used for microwave drying was different from drying by a heat source. During conventional drying, the heat was transferred to the surface of the material by conduction, convection, and/or radiation, and driving into the interior by thermal conduction. For microwave drying, the microwaves penetrate the material and interact with water that is a polar molecule. As water molecules are dipolar in nature, they rotate following the electric field that alternates at very high frequency. Such rotation produces friction of molecules and generates heat inside the material. Seeing that foods are water-rich, microwaves can generate significant volumetric heat. Heated water increase diffusion rate and pressure gradient that facilitates to drive off moisture from inside to outside of the material.

The advantages of microwave drying, when compared with convective drying, were a reduction in the drying time of up to 25% to 90% and an increase in drying rate of 4 to 8 times. The efficiency of microwave drying is due to the fact that the energy directly interacts with water molecules, that eliminates the need to transfer heat.
from the surface into the interior of the product, inducing the increase of driving force for moisture transfer due to the generation of elevated internal vapor pressure directed to the external (Feng et al., 2012).

### 3.3 Total phenolic content

The TPC of *L. nobilis* leaves, obtained by different drying methods (open-air, oven-drying, and microwave), are illustrated in Figure 3. For conventional drying, phenolic concentrations varied from one temperature to another and oscillated from 38 to 51 mg GAE/g; drying in open-air and oven-drying using 120 °C could achieve the highest levels of TPC. The different fluctuations on TPC may be due to the variation of the used temperature in relation to the time required to drying. Indeed, the increase in the drying temperature can reduce the time of exposure to heat due to the quick drying of leaves but, on the other hand, it also promotes the degradation of the bioactive compounds.

From TPC results obtained by microwave drying, it was noticed that the lower phenolic content was obtained by the power of 180 W. The rise of the power to 300 W leads to an increase in TPC to reach the highest level then decreased for the highest powers. The increase of TPC for 300 W can be due to the increase of vapor pressure and temperature generated by microwaves that could break cell walls allowing the release of phenolic compounds that become more accessible during the extraction (Dong et al., 2019). However, the use of powers from 450 to 900 W leads to a significant TPC diminution that can be related to the degradation of these bioactive compounds by high temperatures generated by elevated microwave levels. The concentration of polyphenols declined during drying, and the behavior may be attributed to the chemical degradation, the formation of insoluble oxidation compound, the production of polymers, and the decomposition of thermolabile compounds (Obied et al., 2008; Henríquez et al., 2014). The obtained results were in line with those of Saha et al. (2019) who found that the drying of corncob by microwave using 130 W provided the highest TPC; while drying with 600 and 900 W caused the diminution of phenolic compounds and antioxidant activity.

**Figure 3.** Effect of conventional (A) and microwave (B) drying on total phenolic content of *Laurus nobilis* leaves. Results with different letters for conventional or microwave drying method are significantly different at $p < 0.05$, with $a > b > c > d$. 
3.4 Antioxidant activity

The results of conventional drying showed that the highest antioxidant activity was obtained by the leaves dried at open-air followed by those at 120 °C and the others provided significantly lower activities (Figure 4). The antioxidant activity profile followed the same pattern as TPC. Open-air drying allowed better preservation of bay leaves antioxidants. Oven-drying (40 °C to 100 °C) induced a significant reduction of these bioactive compounds, indicating the presence of thermolabile phenolic compounds.

The increase in phenol content and antioxidant activity, observed for dried leaves at 120 °C, might be due to the formation of the browning compounds rather than to the bioactive ones. This is supported by several studies that have found that Maillard compounds, produced during heating, can interfere during phenolic compounds determination and antioxidant activity evaluation (Pérez-Burillo et al., 2019; Mondaca-Navarro et al., 2017).

Concerning microwave drying, it was noticed that the power of 300 W provided a better antioxidant activity followed by 450 W. Antioxidant activity manifested by the dried leaves with a microwave followed the same pattern as phenolic compounds.

Statistical analysis indicated a very highly significant value ($p < 0.001$) of Pearson correlation coefficient ($r = 0.83$) between phenolic contents and antioxidant activity of dried bay leaves obtained by different drying methods. This indicated that phenolic compounds of $L. nobilis$ leaves were responsible for 83% of its antioxidant activity. These findings are in line with those of several researchers (Izli et al., 2017; Bachir bey et al., 2014).

![Figure 4](image)

**Figure 4.** Effect of conventional (A) and microwave (B) drying on antioxidant activity of Laurus nobilis leaves. Results with different letters for conventional or microwave drying method are significantly different at $p < 0.05$, with a > b > c > d > e.

4 Conclusion

According to the obtained results, it could be noticed that bay leaves dried in open-air (23 °C) and with oven-drying using low temperatures (40 °C) took a prolonged time to moisture stabilization (3 or 4 days). The required time of drying to achieve moisture equilibrium was reduced by using high temperatures and
mostly shortened by the microwave drying. It is stated that \textit{L. nobilis} leaves dried in the open-air (4 days) and microwave (300 W/130 s) displayed the highest TPC and antioxidant activity. Thus, taking account of drying time and antioxidant parameters, we could consider that the microwave-assisted method using a power of 300 W during 130 s was selected to produce better dried bay leaves.

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