Microwave and convective air on drying of *araucaria* angustifolia seeds

Utilização de micro-ondas e ar quente na secagem de sementes de *araucaria angustifolia*

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Highlights ____

Potential use of microwaves to dry *Araucaria angustifolia* seeds. Influence of microwave power on drying temperature and time. Variation of phenolic compounds in drying processes.

Abstract __

Microwave drying has shorter processing times compared with convection drying, which results in highquality products and reduced energy consumption. The objective of this study is to evaluate the drying process of *Araucaria angustifolia* seeds using hot air at atmospheric pressure by applying microwaves (300 W and 500 W), and comparing them with the conventional drying process using hot air (80 °C). The drying time using hot air with microwaves at 500 W and 300 W was reduced by 83.33% and 73.89%, respectively, compared with conventional drying. The drying process occurred in the transient regime, with the predominance of a period of decreasing drying rate. The internal temperature of Araucaria seeds increased with the exposure time to the drying process, whereas the moisture content decreased. This tendency was greater for seeds subjected to electromagnetic fields, especially those exposed to higher power. The total phenolic compounds (TPCs) were lower by microwave drying when compared with conventional drying. The TPC loss was influenced by the internal temperature of the seed. Alternative methods to promote the increased retention of phenolic compounds during the drying process include decreasing the microwave power before the end of drying, combining traditional microwave-associated drying methods, and controlling the applied microwave power.

Key words: Phenolic compounds. Power. Process. Seed.

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Resumo _

Comparados aos secadores por convecção, os secadores dielétricos têm tempos de secagem mais curtos, proporcionando produtos de melhor qualidade e consequentemente menor consumo de energia. O objetivo deste estudo foi avaliar os processos de secagem de sementes de *Araucaria angustifolia* usando ar quente à pressão atmosférica, aplicando micro-ondas (300 W e 500 W) e compará-los ao processo de secagem convencional com ar quente (80 °C). O tempo de processo de secagem usando ar quente com micro-ondas foi reduzido em 83,33% na potência de 500 W e 73,89% a 300 W, quando comparado à secagem convencional. Os processos de secagem ocorreram no regime transiente, com predominância do período de taxa decrescente. Observou-se que os valores da temperatura interna aumentaram com o tempo de exposição do material ao processo, enquanto a umidade diminuiu. Essa tendência foi maior para sementes submetidas a campos eletromagnéticos, principalmente aquelas expostas a maior potência. Os compostos fenólicos totais foram menores na secagem por micro-ondas quando comparados à secagem convencional. A perda de compostos fenólicos foi influenciada pela temperatura no interior do produto. As alternativas para promover maior retenção de compostos fenólicos no processo de secagem seriam a diminuição da potência antes do final da secagem, a combinação dos métodos tradicionais de secagem associados às micro-ondas e o controle da potência a ser aplicada no micro-ondas.

Palavras-chave: Compostos fenólicos. Potência. Processo. Sementes.

Introduction _____

Araucaria angustifolia is a gymnosperm with pine cones formed by approximately 100 seeds, known as pine nuts. Its peel is red and has an edible pulp that is too hard to consume *in natura*. As a result, the seeds must be processed to soften their texture prior to consumption (Zortéa-Guidolin, Carvalho, Godoy, Demiate, & Scheer, 2017; Quinteiro, Alexandre, & Magalhães, 2019).

Phenolic compounds are substances with free and complex structures that exhibit antioxidant properties. These structures are found in fruits and vegetables, and provide health benefits, such as the prevention of cancer and cardiovascular disease (Angelo & Jorge, 2007; Cataneo, Caliari, Gonzaga, Kuskoski, & Fett, 2008; Liu, 2013). Several studies on fruits have reported the effect of drying temperature on phenolic compounds, i.e., an increase in their degradation with the increase in temperature (Garcia-Perez, García-Alvarado, Carcel, & Mulet, 2010; Suvarnakuta, Chaweerungrat, & Devahastin, 2011; Djendoubi, Boudhrioua, Kechaou, Courtois, & Bonazzi, 2012; Karaaslan et al., 2014).

Phenolic compounds found in Araucaria seeds include procyanidins (catechin and epicatechin derivatives), phenolic acids (protocatechuic acid and ferulic acid hexoside), and flavonoids (Koehnlein et al., 2012; Santos et al., 2018). According to Cordenunsi et al. (2004) and Koehnlein et al. (2012), when Araucaria seeds (pine nuts) are cooked, the amount of phenolic compounds increase because of their migration from husk to the seed during cooking.



The pine nut is a seasonal product native to southern Brazil, where it is highly appreciated and frequently sold in informal markets. The Araucaria seed has high moisture levels and water activity; hence, it is easily contaminated by microorganisms during storage, which hinders its commercialization (Cladera-Olivera, Pettermann, Noreña, Wada, & Marczak, 2008). The development of new food products from Araucaria seeds can provide an alternative method of commercialization during the off-season at an increased value. Drying is one of the main methods of food preservation. However, the high temperatures and long drying times in conventional drying can damage the food, changing its texture, color, taste, and nutritional value. Microwave drying can reduce such damage (Menon, Stojceska, & Tassou, 2020).

Microwave drying of foods with high moisture content is an efficient method. In conventional drying, heat transfer occurs because of the temperature gradient between the dry surface and the humid center, making it longer to complete the process. On the other hand, microwaves can penetrate the food and heat it because of the interaction between the electromagnetic field and dipolar molecules, especially water molecules, causing an instantaneous rise in internal temperature and shorter drying times (Béttega, Rosa, Corrêa, & Freire, 2014).

A literature review has shown that several studies have been conducted on microwave drying. The studies are related to the production of husked cherry coffee (Cunha, Canto, & Marsaioli, 2003), coconut husk (Valadez-Carmona, Cortez-García, Plazola-Jacinto, Necoechea-Mondrágon, & OrtizMoreno, 2016), dried carrots (Béttega et al., 2014), strawberries (Böhm, Kühnert, Rohm, & Scholze, 2006), macadamia nuts (Silva, Maximo, Marsaioli, & Silva, 2007), and granulated solids (Berteli, Nogueira, & Marsaioli, 2015). However, there is a lack of studies on the microwave drying of Araucaria seeds. Many studies in the literature also exhibit an information gap and a lack of data accuracy, assuming that several studies have been performed using adapted domestic microwaves.

The objective of this study is to evaluate the use of microwaves to dry *Araucaria angustifolia* seeds, their behavior during drying, and the total phenolic compounds (TPC) after processing.

Materials and Methods _____

Sampling

Araucaria angustifolia seeds (pine nuts) were purchased from a local market in Pinhão, Paraná, Brazil. Samples were preselected to exclude germinated seeds and seeds that were mechanically damaged or attacked by insects or fungi. The selected samples were stored in sealed plastic bags at -21 °C until further use.

Cooking process

The cooking process was carried out in batches of 200 g of pine nuts in 2 L of potable water in a pressure cooker at 2.5 atm for 30 min. The cooked seeds were peeled and cut in half longitudinally. Afterward, the samples were identified and subjected to the drying process.

Centesimal characterization of in natura and cooked Araucaria seeds

According to the methodologies described in the literature (Instituto Adolfo Lutz [IAL], 1986), pine nuts were characterized by their moisture, protein, ash, and lipid content. Characterization was performed for *in natura* and cooked seeds. All analyses were conducted at least three times.

Drying

Drying tests were performed on the cooked and peeled Araucaria seeds. In microwave drying, the air temperature was 59 °C at 300 W and 500 W. In the conventional (CONV80) drying process with circulation and forced air renewal, the velocity was 1 m s⁻¹ at 80 °C. The mass of Araucaria seeds used in each experiment was 200 ± 3.91 g. During the drying process, the internal temperature of the seeds was monitored using thermocouples. For conventional drying, the experiments were performed in triplicate. For microwave drying, experiments were conducted with one sample owing to cell size limitations. Before the tests, the microwave drying system was experimentally validated. The experiments were conducted in a multifunctional pilot plant designed for the application of microwaves in food processing at the Engineering and Post-harvest Group of the Institute of Food Technology.

Based on previous tests, a moisture content of approximately 20% (wet basis) was established as the endpoint parameter of the microwave drying process. This value was determined based on the texture characteristics, considering the possibility of dry seed consumption as chips or its use as a raw material in food product formulations.

After drying, the seeds were labeled and stored under vacuum in low-density nylon/ polyethylene transparent bags.

Extraction and determination of total phenolic compounds

The extracts were obtained using the methodology described by Santos et al. (2018), with some modifications. Cooked and ground Araucaria seeds (2.5 g) were solubilized in 100 mL of 45% (v v⁻¹) ethanol and stirred at 780 rpm for 1 h and 10 min at 63.5 °C. The extracts were filtered and stored at -20 °C until phenolic analysis.

The TPCs were determined using the Folin-Ciocalteau method (Bucić-Kojić, Planinić, Tomas, Bilić, & Velić, 2007). The extract (1 mL), Folin-Ciocalteu reagent at 10% (v v⁻¹) (4 mL), and sodium carbonate solution at 7.50% (m v⁻¹) (2 mL) were placed in a 25 mL flask and filled with distilled water. After homogenization and storage (2 h in the absence of light), the absorbance was measured at 765 nm. The standard curve was constructed using gallic acid (AG) at 0.2, 0.5, 1.0, and 2.5 mg AG L⁻¹. The results are expressed in mg AG 100-1 g⁻¹ of the dry sample. The analyses were performed in triplicate.

Statistical analysis

The results were evaluated using the analysis of variance and Tukey's test with a 95% confidence level.



Results and Discussion _____

The proximate analysis of *in natura* and cooked Araucaria seeds (Table 1) showed that the moisture content of *in natura* seeds was 46.24%, similar to those reported in the literature (Cordenunsi et al., 2004; Gama, Masson, Haracemiv, Zanette, & Córdova,

2010), whose values were close to 50%. The cooked seeds in the present study contained 50.67% moisture, similar to that found (Capella, Penteado, & Balbi, 2009). This increase in moisture during cooking (p < 0.05) is attributed to the absorption of water by the seed, which changes in its physical and sensory structure.

Table 1Results of centesimal analysis of *in natura* and cooked Araucaria seeds

Parameters	<i>In natura</i> seeds	Cooked seeds	F-calc	p-value
Moisture % (wb)	46.24 ± 0.44	50.67 ± 0.67	214.11	0.009
Ash % (db)	2.37 ± 0.02	2.58 ± 0.02	1.41	0.829
Proteins % (db)	6.88 ± 0.18	6.81 ± 0.16	1.26	0.884
Lipids % (db)	2.08 ± 0.14	2.28 ± 0.03	24.99	0.077

wb (wet basis) and db (dry basis). Results are expressed as mean \pm standard deviation. Fisher calculated test at 95% confidence level.

With regard to the ash, protein, and lipid content, the results for *in natura* and cooked seeds were similar, indicating that the cooking process did not significantly affect these physicochemical characteristics (p > 0.05). The values for *in natura* and cooked seeds obtained in this study are close to those reported in the literature (Zortéa-Guidolin et al., 2017; Capella et al., 2009; Cladera-Olivera et al., 2008).

Considering the drying process (Figure 1), it can be observed that the samples did not reach their equilibrium moisture content because this condition was not required in the process. The endpoint for the microwavetreated samples was a final moisture content of approximately 20% (wb), which corresponds to a moisture of 0.25 g H_2O g⁻¹ (db). Figure 1 shows that for the microwave process, the drying time was reduced by 83.33% and 73.89% at 500 W and 300 W, respectively, compared with conventional drying. After 3 h, conventional drying resulted in a final moisture content of 28.30% (wb), which corresponds to 0.3947 g H_2O g⁻¹ (db). The water loss of the Araucaria seeds subjected to microwave drying was faster than that in the conventional system. In this last case, the exposure time of the seed to heat was longer compared with the other methods.



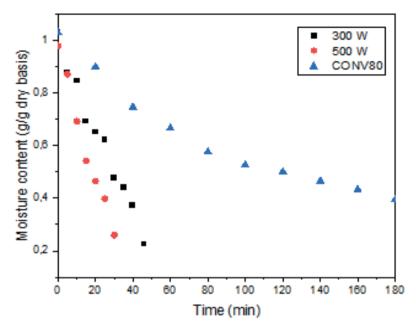


Figure 1. Drying kinetics for peeled cooked seeds while drying under different treatments. 300 W = microwaves applied at 300 W; 500 W = microwaves applied at 500 W; CONV80 = conventional drying process with hot air at 80 °C.

A mathematical analysis was performed on the drying kinetics (Table 2), following the first derivative obtained to obtain the drying rate data (Figure 2).

Table 2 Drying models and their derivatives estimated for mathematical adjustment

Treatment	Mathematical adjustment	Determination Coefficient-R ²	Derivative
300 W	Y= 3E-05x ² - 0.0176x + 0.9972	0.9836	Y= 6E-05x - 0.0176
500 W	Y = 3E-04x ² - 0.0317x + 0.9898	0.9905	y = 6E-04x - 0.0317
CONV80	y = 2E-05x ² - 0.0068x + 1.0148	0.9918	y = 4E-05x - 0.0068

300 W = applying microwaves at 300 W; 500 W= applying microwaves at 500 W; CONV80= conventional drying process with hot air at 80 °C.

0.03



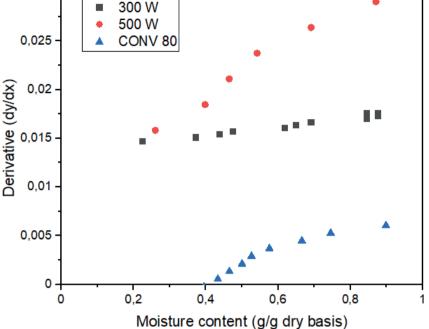
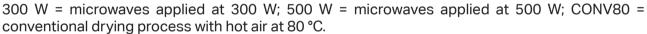


Figure 2. Drying rate curves.



The adjustment followed a secondorder polynomial, whose determination coefficients were greater than 0.9800.

The drying rate curves (Figure 2) show that conventional drying caused the smallest variations in the dying rate; the final moisture content was higher than those at 300 W and 500 W. Graphically, the prevalence of the decreasing rate was observed, which indicates that drying occurs in the transient regime, where the mass transfer is driven by internal diffusion (Nascimento, Biagi, & Oliveira, 2015; Resende, Ullmann, Siqueira, Chaves, & Ferreira, 2011).

Monitoring of internal temperature profile of the seeds during the drying process (Figure 3) showed that the internal temperatures varied for each treatment. In conventional drying, the seeds were subjected to an internal temperature of 60.87 °C. In microwave drying at 500 W, the internal temperature reached 97.58 °C. According to Martins, Melo, Barbosa, Santos and Machado (2002), higher temperatures led to a shorter processing time, but resulted in greater nutrient losses and structural changes in the seed. Drying at 500 W can be advantageous in terms of processing time and cost, but it can cause undesirable product modifications that must be investigated.

Conventional drying provides a mild internal temperature compared with other processes; however, the heat exposure time is much longer, which can also cause significant nutritional losses This fact was confirmed by Soares, Chaves, Silva, Silva and Souza (2007),



who studied the drying of basil at different temperatures. They found that using a lower temperature increased the processing time as well as the loss of chemical compounds. At 300 W, the internal temperature of the seed reached a maximum of 69.05 °C, which is 8.18 °C higher than the temperature reached by the seed in conventional drying; however, the drying time was approximately four times shorter.

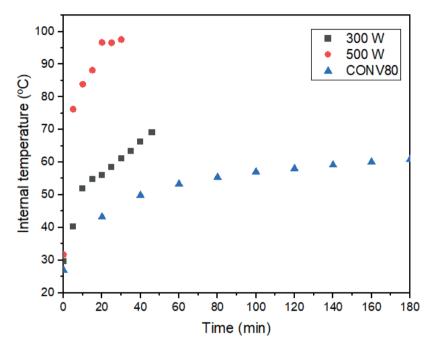


Figure 3. Internal temperature of the peeled cooked seeds while drying under different treatments. 300 W = microwaves applied at 300 W; 500 W = microwaves applied at 500 W; CONV80 = conventional drying process with hot air at 80 °C.

During the drying process, the internal temperature of the seed subjected to 500 W increased rapidly in the first 20 min, after which it exhibited a more asymptotic behavior. This may be because the dielectric properties of the seed lost their capacity for microwave absorption during processing at high temperatures, causing temperature stability.

It was observed that the temperature increased with the exposure time of the seed to the process, while the moisture content decreased. This tendency was greater for seeds subjected to electromagnetic fields, especially those exposed to higher power. The TPCs in Araucaria seeds (Table 3) significantly decreased (p < 0.05) during the drying process. The seeds subjected to microwave drying showed higher TPC losses (8.26 and 10.05 mg AG 100^{-1} g⁻¹ db) compared with conventional drying (16.63 mg AG 100^{-1} g⁻¹ db). In conventional drying, the exposure time was long (3 h), but the seed's internal temperature was low (60.87 °C). In microwave drying at 300 and 500 W, the seed's maximum internal temperatures were 69.05 °C and 97.58 °C, respectively. This result showed that TPC loss was influenced by the internal temperature of the seed.



Table 3 TPCs for different treatments

Treatments	TPC (mg AG 100 ⁻¹ g ⁻¹ db)*
Cooked before drying	19.46 ± 0.35a
CONV80	16.63 ± 0.57b
300 W	8.26 ± 0.14d
500 W	10.05 ± 0.13c

Different letters in the column indicate significant differences (p < 0.05) according to Tukey's test. 300 W = applying microwaves at 300 W; 500 W= applying microwaves at 500 W; CONV80 = conventional drying process with hot air at 80 °C.

Alternative methods to promote the retention of phenolic compounds during the drying process include decreasing the microwave power before the end of drying (Wojdyło, Figiel, Lech, Nowicka, & Oszmiański, 2014), combining traditional microwaveassociated drying methods (Samoticha, Wojdylo, & Lech, 2016), and varying the microwave power used (Coklar, Akbulut, Kilinc, Yildirim, & Alhassan, 2018). In addition, it is necessary to evaluate the physicochemical and functional characteristics of food to establish adequate processing conditions, costs, and product quality.

Conclusion _____

Microwave drying can be used as an alternative method for drying Araucaria seeds. The drying time was shorter for seeds subjected to microwave dying compared with the conventional process. The TPCs were lower in microwave drying than in conventional drying, indicating that temperature influenced this process.

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