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Effect of drying methods on the physicochemical composition and microstructure of pumpkin powders

Efecto de los métodos de secado en la composición fisicoquímica y la microestructura de polvos de calabaza

Efeito dos métodos de secagem na composição físico-química e na microestrutura dos pós de abóbora

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Abstract

To increase the shelf life of fruit and vegetables, to be able to enjoy them in all four seasons, and to preserve their genetic make-up, drying has been found to be the best technique of conservation. The aim of this work is based on the study of the drying kinetics of pumpkin (*Curcubita maxima*) by two drying methods namely oven drying at a temperature of 60 °C and microwave drying at 180 W, with the purpose to model the drying kinetics of thin layers of pumpkin by four mathematical models (Two-Term, Modified Henderson and Pabis, Henderson Pabis and Bousselma *et al.*) and to study the effect of the two drying methods on the nutritional and microstructural properties of pumpkin powders. The results showed that the Microwave drying was faster than oven drying. The Modified Henderson and Pabis and Bousselma *et al*., models were chosen to adequately describe the drying behavior of oven- and microwave-dried thin pumpkin slices, respectively, due to a high R^2 value and low χ^2 and RMSE values. The physicochemical composition of the two powders (POD and PMD) was significantly different ($p < 0.05$) in terms of water content, pH, brix, lipids, and potassium. The analysis of the qualitative composition by FTIR did not show a change between the two powders. Similarly, the structure studied by SEM showed an identical and homogeneous structure. These powders have high nutritional properties, and their incorporation into foods should therefore be recommended.

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Resumen

Para aumentar la vida útil de las frutas y verduras, poder disfrutarlas en las cuatro estaciones y preservar su composición genética, se ha descubierto que, el secado es la mejor técnica de conservación. El objetivo de este trabajo se basa en el estudio la cinética de secado de calabaza (*Curcubita maxima*) mediante dos métodos de secado, a saber, secado en horno a una temperatura de 60 °C y secado en microondas a 180 W, para modelar la cinética de secado de capas delgadas de calabaza mediante cuatro modelos matemáticos (Two-Term, Modified Henderson y Pabis, Henderson Pabis y Bousselma *et al.*) y estudiar el efecto de los dos métodos de secado sobre las propiedades nutricionales y microestructurales de los polvos de calabaza. Los resultados mostraron que, el secado por microondas fue más rápido que, el secado en horno. Se eligieron los modelos modificados de Henderson y Pabis y de Bousselma *et al*., para describir adecuadamente el comportamiento del secado de las rodajas finas de calabaza secadas en horno y microondas, respectivamente, debido a un alto valor de R^2 y bajos valores de χ^2 y RMSE. La composición fisicoquímica de los dos polvos (POD y PMD) presentó diferencias significativas ($p < 0,05$) en cuanto a contenido de agua, pH, grados Brix, lípidos y potasio. El análisis de la composición cualitativa mediante FTIR no mostró cambios entre los dos polvos. De igual manera, la estructura estudiada mediante SEM mostró una estructura idéntica y homogénea. Estos polvos poseen elevadas propiedades nutricionales, por lo que conviene recomendar su incorporación a los alimentos.

Palabras clave: *Cucurbita maxima*, secado al horno, secado por microondas, polvo, características fisicoquímicas.

Resumo

Para aumentar a vida útil das frutas e legumes, para poder apreciá-los em todas as quatro estações e para preservar a sua composição genética, descobriu-se que a secagem é a melhor técnica de conservação. O objetivo deste trabalho baseia-se no estudo da cinética de secagem da abóbora (*Curcubita maxima*) por dois métodos de secagem, ou seja, secagem em estufa a uma temperatura de 60 °C e secagem em micro-ondas a 180 W, para modelar a cinética de secagem de camadas finas de abóbora por quatro modelos matemáticos (Two-Term, Modified Henderson e Pabis, Henderson Pabis e Bousselma *et al.*) e para estudar o efeito dos dois métodos de secagem nas propriedades nutricionais e microestruturais dos pós de abóbora. Os resultados mostraram que a secagem em micro-ondas foi mais rápida do que a secagem em forno. Henderson modificado e Pabis e Bousselma *et al.,* modelos foram escolhidos para descrever adequadamente o comportamento de secagem de fatias finas de abóbora secas em forno e micro-ondas, respetivamente, devido a um elevado valor de R^2 e baixos valores de χ^2 e RMSE. A composição físico-química dos dois pós (POD e PMD) foi significativamente diferente ($p < 0,05$) em termos de teor de água, pH, brix, lípidos e potássio. A análise da composição qualitativa por FTIR não mostrou alteração entre os dois pós. Da mesma forma, a estrutura estudada por MEV apresentou uma estrutura idêntica e homogénea. Estes pós têm elevadas propriedades nutricionais e, por isso, a sua incorporação nos alimentos deve ser recomendada.

Palavras-chave: *Cucurbita maxima*, secagem em estufa, secagem em micro-ondas, pó, caraterísticas físico-químicas.

Introduction

The cucurbits are among the oldest plants cultivated in tropical areas of South America. They are symbolic vegetables of autumn and winter. Their size is 32.6 cm in length and 69.1 cm in diameter (Dhiman *et al.*, 2009). The average weight of the fruits is between 8 and 10 kg, sometimes even up to 20 (Yetesha *et al*., 2023). The percentage of the edible part is 70 to 86 % (Dhiman *et al*., 2009). The main varieties of cucurbits cultivated in the world are: *Cucurbita pepo*, *Cucurbita maxima* and *Cucurbita moschata*. The pumpkin is known for its significant nutritional value. The proximate analysis of the fresh pumpkin fruits contains around 90 % water, the proximate analysis leading to 0.6-1.8 $g.100\ g^{-1}$ proteins, 0.1 $g.100\ g^{-1}$ lipids, 4.6-6.5 $g.100\ g^{-1}$ carbohydrates, 0.5-1.3 $g.100\ g^{-1}$ fibers (Dhiman *et al*., 2009; Muntean *et al*., 2013).

Known pharmacological properties of pumpkins include: anti-cancer, anti-diabetic, hepatoprotective, antioxidant, vermifuge, antibacterial, antiinflammatory and anthypertenstive (Dubey, 2012; Das and Banerjee, 2015; Lucan and Mitroi, 2024).

Pumpkin is used in the culinary preparation of soups, juices, purees, jams and pies. It can also be used as a functional food in baked goods, cookies, chocolate, beverages, meat and dairy products (Lucan and Mitroi, 2024). Pumpkin can also be processed into flour by drying and grinding (Benseddik *et al*., 2020; Grassino *et al*., 2024). This flour is a particular raw material and serves like a natural colorant for fruit preparations. Recent research has shown that pumpkin powder can substitute white flour in bakery and cookie products with lot of advantages (Sathiya Mala *et al*., 2018; Ghendov-Mosanu *et al*., 2023). Pumpkin has also been used to make probiotic foods (Lucan and Mitroi, 2024). Drying vegetable products is a very old preservation method, which consists of reducing the amount of water in food to extend its shelf life. It concentrates the food's flavors and nutrients. Drying by oven and by microwave are two methods of food dehydration, including vegetables, but they operate on very different principles and therefore have distinct effects on the final product. The drying oven uses hot air convection to extract moisture from the food. On the other hand, microwave drying uses electromagnetic waves that agitate the water molecules present in the food. Drying kinetics models are therefore significant for deciding on ideal drying conditions, which are important parameters in terms of equipment design and optimization, and product quality improvement. Thus, to analyze the drying behavior of fruits and vegetables, it is important to study the kinetic model of each particular product and to generate generalized drying curves (Kalsi *et al*., 2023).

In this study, we try to transform pumpkin (*Cucurbita maxima*) pulp into flour by two drying techniques to evaluate the influence of the drying process on the kinetics of water loss, the physicochemical and structural properties of the obtained powders.

Materials and methods

Vegetal material

The pumpkin used was an orange Hadil variety. It was purchased at the local market in Batna, Algeria. It was kept refrigerated at 4 °C until use.

Drying Procedure

The pumpkin was washed, dried and cut into cubic pieces. The pulp was then cut into round slices with an average diameter of 3 cm and an average thickness of 1 mm using a food processor (Moulinex type). Two drying methods were used: microwave drying (MWD) and oven drying (OD). For convective drying, it was carried out in a Memmert-type oven; a temperature of 60 °C was applied. In microwave drying mode, a domestic Iris-type oven was used with a drying power of 180 W. The drying kinetics were repeated five times.

The moisture ratio (MR) was calculated to evaluate moisture loss, like showed in the Eq.1 (Bousselma *et al*., 2021):

$$MR = \frac{M - Me}{M0 - Me} \quad (1)$$

(Where, M: represents moisture content at any time t; M0: is initial moisture content; Me: equilibrium moisture content).

Four thin-layer drying models, including Henderson and Pabis, Modified Henderson and Pabis, Two-term, and Bousselma *et al*. (2021), were fitted to the drying data to select the best model suitable for describing the drying process of pumpkin slices (table 1).

The Sigma plot version10 (Systat Software Inc, Chicago, IL, USA) was used to determine the model constants. The best fit of the model was based on the root mean square error (RMSE), coefficient of determination (R^2), and chi square (X^2) (Table 2).

Obtaining powders

Dried pumpkin slices were ground using a domestic grinder, then passed through a 250-micrometer sieve to obtain powders.

Methods of analysis

The water content was determined by drying in an oven at 105 °C until a constant weight was obtained. The pH was analyzed using a Hanna type pH meter at 20 °C (AFNOR, 1986). The Brix was determined at 20 °C using a digital refractometer of the Reichert AR 200 type. The ashes were determined according to the official AFNOR method (1986). The minerals (K, Na, and Ca) were determined

Table 1. Drying models used in this study

Model name	Equation	References
Two-Term	$MR = a\exp(-Kt) + b\exp(-K' t)$	(Soysal *et al*., 2006)
Bousselma *et al*.	$MR = (a+bx)/(1+ cx+dx^2)$	(Bousselma *et al*., 2021)
Henderson and Pabis	$MR = a\exp(-kt)$	(Bousselma *et al*., 2021)
Modified and Henderson and Pabis	$MR = a\exp(-kt) + b\exp(-k't) + c\exp(-k''t)$	(Olabinjo *et al*., 2020)

MR: moisture ratio, t: drying time, a, b, c, dand K d are the coefficients of models.

Table 2. Statisticals metrices

Name of statistical parameter	Formula
Coefficient of determination (R^2)	$R^2 = 1- \frac{\sum_{i=1}^{N}(MR_{exp} - MR_{pred})^2}{\sum_{i=1}^{N}(MR_{exp} - \overline{MR}_{exp})^2}$
Root Mean Square Error (RMSE)	$RMSE = \sqrt{\left(\frac{1}{N}\right)\left(\sum_{i=1}^{N}\left[\left(y_{exp} - y_{pred}\right)\right]^2\right)}$
Chi-square (X^2)	$\chi^2 = \left[\frac{\sum_{i=1}^{N}(MR_{exp} - MR_{pre})^2}{N-n}\right]$

by flame spectrometry (Jenway PFP7, UK). Lipids contents were determined with hexane using soxlhet. Fiber content was determined according to the Weende method using a FIWE Fiber Analyzer (Velp Scientifica).

FTIR spectroscopy

The structural properties of the pumpkin powder constituents were analysed by FTIR spectroscopy. Spectra were recorded using an attenuated total reflection (ATR) spectrophotometer (Agilent Cary 630 ATR) in the range of 400 - 4000 cm^{-1} with a resolution of 4 cm^{-1} (Santiago-García *et al*., 2023).

Scanning electron microscopy

Surface morphological characterization of pumpkin powder was examined using Scanning Electron Microscopy (FEG, Thermo Scientific Apreo 2C), operated at 5 K.

Statical analysis

Statistical analysis was performed using XLSTAT software. Values are presented as mean ± standard deviation. Data were evaluated statistically by Student's T test ($p < 0.05$ was considered statistically significant).

Results and discussion

Physicochemical characteristics of fresh pumpkin

The physicochemical composition of fresh pumpkin was 92.63 % moisture, brix 4.35 % and pH of 6.20. The humidity rate is in agreement with that cited by Adoui *et al.* (2021), which is 92.27 for the same species. The values of pH and Brix are close to those given by Márquez Cardozo *et al*. (2021), which are 3.76 % for Brix and 5.92 for pH. Similarly, Papanov *et al*. (2021), provided pH values of 6.48 and 7.20, respectively, for the two varieties of *Cucurbita maxima* (Argentina and Danka Polka).

Drying kinetics pumpkin for slices

Drying kinetics of pumpkin by microwave and vacuum oven were expressed as moisture ratio as a function time (figure1).

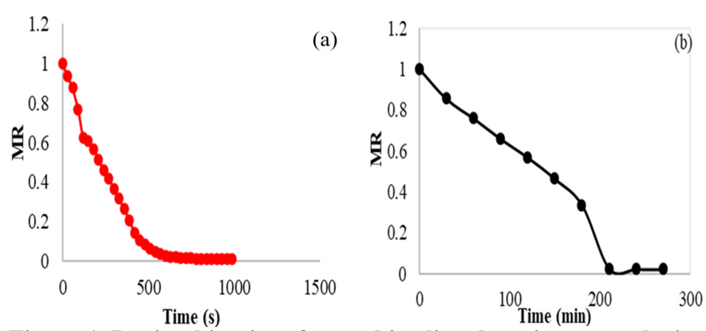


Figure 1. Drying kinetics of pumpkin slices by microwave drying (a) and oven drying (b).

The water content of fresh pumpkin was approximately 92.63 %. The pumpkin slices were dried to a constant weight. At the start of drying, the curves show a steady decrease. This decrease corresponds to the elimination of free water. Initially, the water content in the pumpkin was high, and less microwave energy was absorbed; the pumpkin was heated by radiation, and so water evaporation was accelerated. Drying removed 89.20 % of the water from fresh pumpkin by MW and 87.63 % by convective drying (vacuum oven). Microwave drying is faster than convective drying and reduced the time needed to dry pumpkin slices by 84 % compared with convective (conventional) drying.

In this work, the drying kinetics of pumpkin dried by two drying methods (MWD and OD) were modeled by four mathematical models (Modified Henderson and Pabis, Henderson and Pabis, Two-Term and Bousselma *et al*. (2021). Figures 2 and 3 illustrate the results obtained. The calculated values of the statistical parameters used are presented in table 2, with the most appropriate model indicated in bold type.

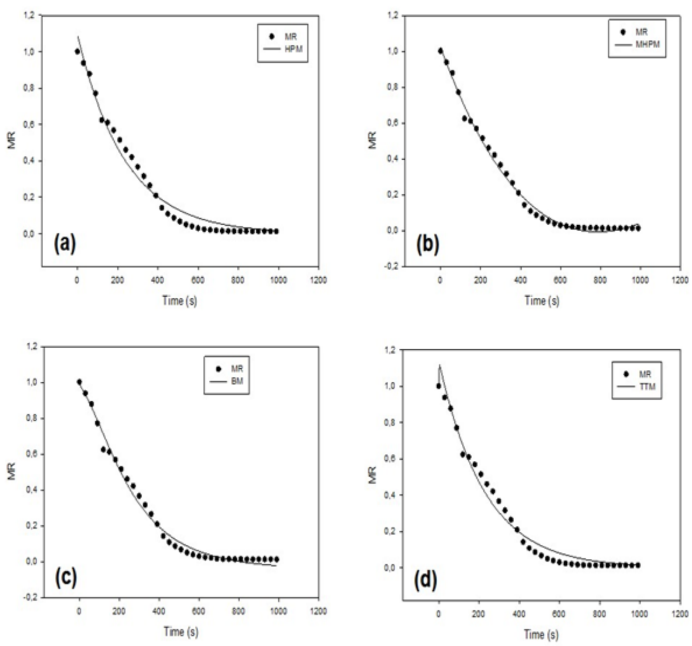


Figure 2. Modeling the drying kinetics of the microwave-dried pumpkin layer. (a) Henderson and Pabis Model, (b) Modified Henderson and Pabis Model, (c) Bousselma et al. Model and (d) Two-Term Model.

The four models were compared in terms of the values of the coefficient of determination (R^2), the reduced chi-squared (χ^2), and the root mean square error (RMSE). In the studied experimental conditions, the values of R^2, χ^2, and RMSE are respectively between 0.8860 and 0.9932, 8.43776E-12 and 0.005306, and 1.26783E-09 and 0.070667. The high R^2 values and low χ^2 and RMSE values for the four models simulated in this study indicate good consistency between these models and the experimental results (Tunde-Akintunde and Ogunlakin, 2013). From these results, we concluded that the MHP (Modified Handerson Pabis) and Bousselma *et al.* (2021) models were chosen to adequately describe the drying behavior of microwave and oven-dried pumpkin thin slices, respectively, due to a high R^2 value and low χ^2 and RMSE values (table 3). The modified Handerson Pabis model was adapted to describe the pumpkin drying process under the drying conditions of the species *Curcubita moschata* at 105 °C (Hong *et al.,* 2017).

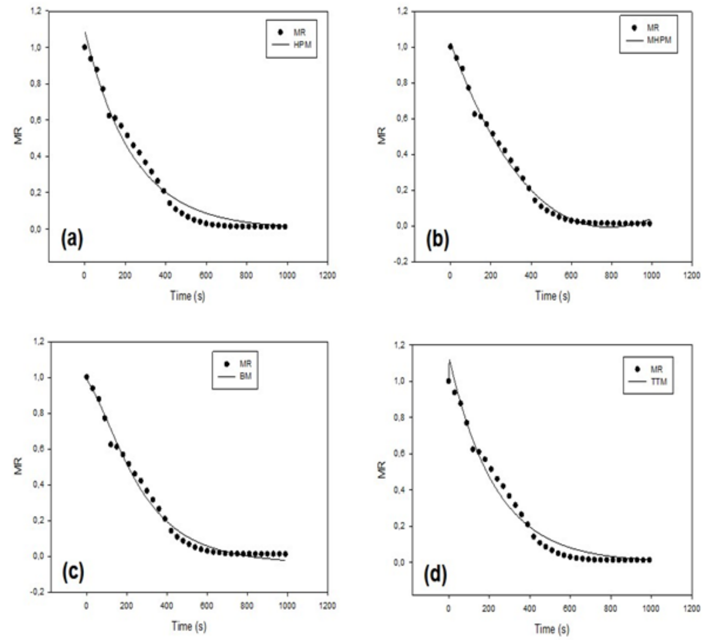


Figure 3. Modelling the drying kinetics of the oven-dried pumpkin layer. (a) Henderson and Pabis Model, (b) Modified Henderson and Pabis Model, (c) Bousselma *et al*. Model and (d) Two-Term Model.

Physicochemical characteristics of pumkin powder

The physicochemical characteristics of pumpkin pulp powders are given in table 4. The water content of the powders is between 10 and 11.83 %. This water limit was favorable to the preservation of the powders obtained. The moisture values found were lower than those quoted by Malkanthi and Hiremath (2020), which was 14.80 % for the same species.

Pumpkin powders obtained by oven drying (POD) and microwave drying (PWD) are shown in figure 4.

The pH of the powders was slightly acidic, at 6.46-6.82, with a significant difference. These values were lower than the result of Malkanthi and Hiremath (2020), which was 5.75 for pumpkin powder dried at 60 °C. The ash content of the two obtained powders was between 7.50 and 7.77 %. This limit was similar to that cited in the literature, which is 6.1-7.24 % (Das and Banerjee, 2015).

The mineral profile is characterized by the abundance of potassium, with values ranging from 536.66 to 673.33 mg.100 g^{-1}, with a significant difference. Both types of pumpkin flour have a low sodium content, these values are similar to that (27.28 mg.100 g^{-1} for the species *Cucurbita moschata*) cited by Bemfeito *et al*. (2020).

The physicochemical characteristics of pumpkin powders were normal regarding to values obtained in several studies. Jabeen *et al*. (2018) showing 9.9 g of moisture, 2.3 g of fat, and a high amount of fiber 11.46 g in pumpkin flour. The global composition of pumpkin flesh powder gave values for ash, fat, fiber, moisture, and carbohydrates respectively of 6.64, 0.18, 11.25, 18.03 and 48.40 g.100 g^{-1} DW. Dietary fibre plays a preventive role against chronic diseases (Badr *et al*., 2011).

FTIR Analysis

FTIR analysis of the two powders is shown in figure 5. The spectra exhibit key vibrational signatures including: a prominent hydroxyl band at 3377 cm^{-1} (O-H/N-H stretching vibrations) characteristic of polysaccharides and associated hydroxyl-containing compounds (Abid *et al*., 2017; Rico *et al*., 2020).

Table 3. Fitting parameters for models drying pumpkin slices.

Pumpkin slices	Model	Model constants	R^2	X^2	RMSE
MWD	Two-Term	a: -0.142 b: 2.5784 c: 1.1424 d: 0.0044	0.9820	0.004426509	0.00364536
OD		a : 25.7098 b : 0.0002 c : -24.7076 d : 4.9835E-015	0.9680	6.28499E-09	6.14085E-05
MWD	Bousselma et al.	a : 0.9858 b : -0.0012 c : 0.0005 d : 8.6805E-006	0.9903	2.78141E-05	0.004785997
OD		a: 0.9643 b : -0.0038 c : -0.0017 d : 1.0278E-005	0.9721	3.72101E-07	0.000472504
MWD	Henderson and Pabis	a : 1.0880 b : 0.0042	0.9718	0.005306	0.070667
OD		a : 1.0881 b : 0.0074	0.8860	0.00314051	0.050124
MWD	Modified Henderson and Pabis	a : 8.722 b : 0.0010 c : 6.7165 d : 1.3386E-016 g : 14.4184 h : 0.0004	0.9942	1.5395E-09	1.26783E-09
OD		a: 21.9473 b: 0.0002 c: 10.4093 d: 8.0393E-011 g: 10.5353 h: 4.7004E-019	0.9680	8.43776E-12	1.83714E-06

MWD: Microwave drying. **OD:** Oven drying.

Table 4. Physicochemical characteristics of pumpkin powders

Parameters	POD	PMD
Water (%)	11.18 ± 0.38^a	10.00 ± 0.13^b
Dry matter (%)	88.82 ± 0.38^a	90.00 ± 0.13^b
pH	6.82 ± 0.01^a	6.46 ± 0.00^b
Brix (%)	72.66 ± 1.15^a	67.33 ± 1.15^b
Fibers (%)	17.50 ± 0.50^a	15.66 ± 1.15^a
Lipids (%)	1.81 ± 0.02^a	2.12 ± 0.02^b
Ash (%)	7.50 ± 0.18^a	$7.77 \pm 0{,}17^a$
Potassium (mg.100 g^{-1})	$536{,}66 \pm 5.77^a$	673.33 ± 15.27^b
Sodium (mg.100 g^{-1})	24.67 ± 2.31^a	24.00 ± 2.65^a
Calcium (mg.100 g^{-1})	93.33 ± 5.77^a	86.67 ± 5.77^a

POD: Powder Oven Drying; **PMD:** Powder Microwave Drying. Values are mean ± standard deviation of triplicate measurements. Means in the same line with different superscript are significantly different ($p < 0.05$).

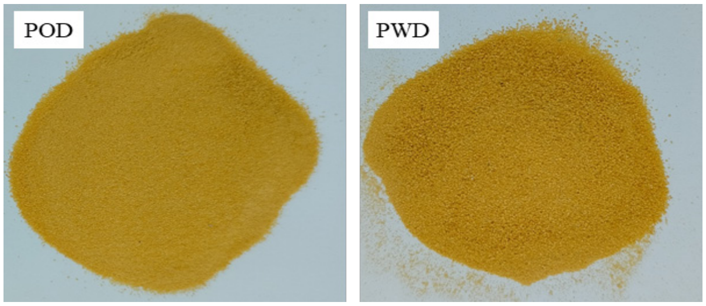


Figure 4. Powder of pumpkin POD and PMD.

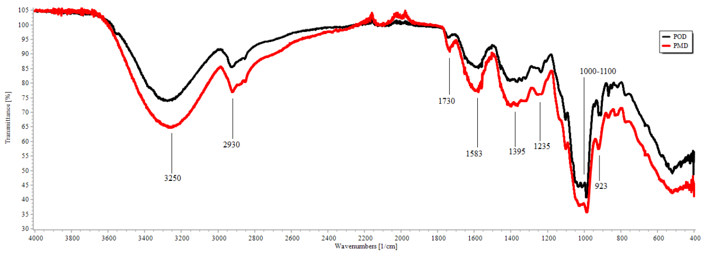
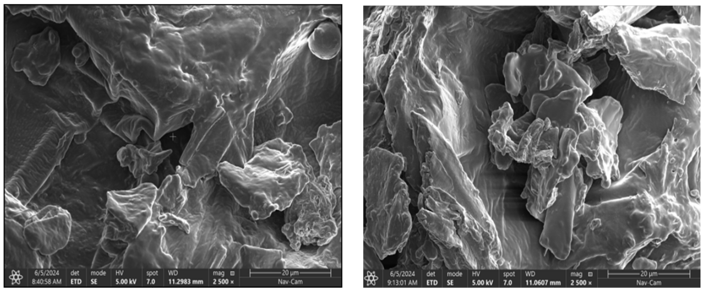


Figure 5. FTIR Spectra of pumpkin powders (POD and PMD).

Distinct carboxylate absorptions at 1395 cm^{-1} (symmetric) and 1583 cm^{-1} (asymmetric stretching) (Adilah *et al*., 2018); an ester carbonyl band at 1730 cm^{-1} (C=O stretching) (Leopold *et al*., 2011); and complex polysaccharide patterns in the 800-1200 cm^{-1} region (arabinogalactans and glycosidic bond vibrations) (Kačuráková *et al*., 2020). Notably, the remarkable spectral congruence between both drying methods evidences the maintenance of structural integrity in the primary cell wall constituents (cellulose, pectin, hemicelluloses), in agreement with existing literature on *Cucurbita* materials (Quintana *et al*., 2018; Indrianingsih *et al*., 2019).

SEM Micrography

According to figure 6, both flours exhibited a compact, fibrous, amorphus structure. Both flours have the same structure, so the drying technique (convective and microwave drying) had no effect on their structure. In comparison with the literature, Indrianingsih *et al.* (2019) and Nurdjanah *et al.* (2023), also showed a compact structure for pumpkin flour.



POD PMD

Figure 6. SEM micrograph of pumpkin powder (POD and PMD).

Conclusion

The Adapted Due to their low χ2 and RMSE values and high R^2 value, the Modified Henderson and Pabis and Bousselma *et al*. (2021) models are selected to accurately depict the drying behavior of thin pumpkin slices that are microwave-dried and oven-dried, respectively. The pumpkin powders obtained by both drying techniques are very nutritious, rich in minerals and fibers, and could be considered as a functional food. The two drying techniques employed had a minimal impact on nutritional quality. The microstructure of the powders is identical. Pumpkin processing is a promising niche in the agri-food industry.

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