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Hygroscopic behavior and morphological characteristics of okra powder obtained by convective drying

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Abstract

Okra is a vegetable that has a fast vegetative cycle of high yield, in addition to considerable nutritional and commercial value, thereat stands out agronomically. Its composition allows it to be consumed while it is still green, but its shelf life in natural form is short. Therefore, the objective of this work was to evaluate the hygroscopic behavior and morphological characteristics of okra powders obtained by convective drying. The okra was cut into slices of approximately 2 mm and dried in an oven with forced air circulation at temperatures of 50, 60, 70 and 80 °C until the hygroscopic balance was reached and ground in a knife mill to obtain the powders. Then, the hygroscopic behavior of the powders was evaluated through water adsorption isotherms at a temperature of 25 °C and mathematical models of GAB, Peleg, Oswin, Halsey and Henderson were adjusted to the experimental data of water contents and activity of water. The powders were also submitted to morphological analysis by scanning electron microscopy (SEM) and X-ray diffraction analysis. The results showed that all the models were well adjusted to the isotherms of the okra powders, with emphasis on the GAB model, with determination coefficients (R^2) greater than 0.99 and average percentage deviations (P) less than 5%; isotherms were classified as type II, typical of products with a large amount of sugar and solutes and with low adsorption. The powders presented morphologically irregular, asymmetrical structures with amorphous characteristics.

Keywords: *Abelmoschus Esculentus*, adsorption isotherms, powder product, X-ray diffraction

Introduction

The okra (*Abelmoschus esculentus* (L.) Moench) is a plant in the Malvaceae family, native to Africa, which has spread to all continents, being appreciated for consumption after cooking. It is known by different denominations, such as okra, abelmosco, guingombô, kingombó, quiabeiro, quiabeiro-chifre-de-veado, quiabeiro comum, *lady finger*, gombo (Sousa et al., 2020; Temenouga et al., 2016). It presents a fast vegetative cycle, producing the whole year and is widely consumed in different parts of the world (Roy et al., 2014). It is of great importance in food, providing minerals, proteins, carbohydrates, as well as being a source of phenolic compounds with antioxidant potential (Gemede et al., 2015; Adekiya et al., 2017; Dubey & Mishra, 2017). It is also a rich source of mucilage, with considerable levels of pectin and lignin (Kpodo et al., 2017).

It has a short shelf life, although it is harvested and consumed when it is partially developed and physiologically immature, characterized by high perishability since it has a high content of free water in its constitution, thus allowing its metabolism to remain active after harvest, occurring continuous chemical transformations, quickly causing it to harden (Freitas & Figueiredo, 2000). Consequently it suffers losses of commercial value and lack of interest on the part of the consumer, justifying the use of measures and techniques that can increase the useful life in shelf conditions, such as the use of coatings, bleaching and refrigeration for the whole product, and drying that allows the added value of the raw material with the ability to preserve most of its properties, in addition to the ease of transport and storage of the product.

One of the main techniques to make the conservation of agricultural products viable is drying, which comprises a unitary operation to remove water from a

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product by evaporation or sublimation, by applying heat under controlled conditions, which decreases its water activity, allowing to minimize the deterioration caused by microbiological, chemical and enzymatic reactions (Santos et al. 2014). It occurs by transferring heat from the surrounding medium to the material and transferring the mass of the liquid or vapor present inside the material into the atmosphere, in the form of vapor (Tadini et al., 2016). Convective drying has as great advantages the low complexity and simplicity of the equipment, whose functionalities are mainly aimed at temperature control. When properly applied, it can produce good quality products, at a relatively low cost.

Some works are reported in the literature on drying okra: Pendre et al. (2012) studied the effect of different temperatures (50 to 80 °C) and different thicknesses (1 to 4 cm) of okra slices and verified the influence of these variations on the protein, fiber and ascorbic acid content after drying; already Brito and Araújo (2015) determined the total phenolic content, and antioxidant activity in the fruit and okra flour, to obtain the flour the researchers carried out the drying operation at a temperature of 50 °C for 6 hours in an oven; and Jiang et al. (2017) studied different food processing methods such as freeze-drying followed by vacuum microwave drying and drying followed by vacuum microwave and freeze drying to obtain okra snacks and evaluated the effect of these techniques on the content of antioxidants and phenolic activity. Among the works cited above, no research in the literature evaluates the hygroscopicity of powdered okra. Being an analysis considered essential for the evaluation of this type of product because it is linked to the physical, chemical and microbiological stability of the material. One way is through its water adsorption isotherms. The knowledge of adsorption isotherms helps in determining the drying time, characterization of the product, in addition to determining the useful life and the type of packaging suitable for its storage (Oliveira et al., 2014; Cavalcante et al., 2018).

Given the above, the objective of this work was to evaluate the hygroscopic behavior of dried okra at temperatures of 50, 60, 70 and 80 °C, by determining its water adsorption isotherms, and to perform the morphological characterization of the particles and the X-ray diffraction analysis of the powders.

Material and Methods

The experiments used okra (*Abelmoschus esculentus* (L.) Moench) from the green subgroup, of the Santa Cruz variety, in a green maturation stage, from the municipality of Caturité, State of Paraíba, Brazil (latitude 7 ° 25 ' 12 '' S, longitude 36 ° 01 '37' 'O, altitude 405 m).

The okra was transported to the laboratory, selected manually to eliminate those with physical damage or unwanted maturation stage; then, they were subjected to a wash in running water, sanitized in chlorinated water (50 ppm) for 15 minutes and, subsequently, rinsed in running water.

For drying, the okra with an average diameter of 11 mm was cut into slices with a standardized thickness of 2 mm (Figure 1A), placed in stainless steel trays and subjected to drying in an oven with forced air circulation, at temperatures 50, 60, 70 and 80 °C, until the samples reach hygroscopic equilibrium. Then, the dehydrated okra was ground in a knife mill and sieved in stainless steel sieves with 32 mesh for standardizing the powders (Figure 1B).

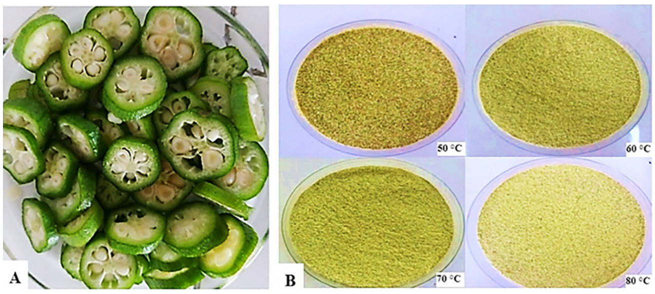

Figure 1. Okra: Okra sliced (A); Powdered okra at different drying temperatures (B).

The hygroscopic behavior of the powders was evaluated using water adsorption isotherms, determined using the indirect static method, described by Capriste & Rotstein (1982), at a temperature of 25 °C. The measurements of the water activity of the samples were carried out in an AquaLab 3TE hygrometer (Decagon) with intervals of 5 minutes until reaching very close values, being added another 10 minutes, followed by 20, 30 and 60 minutes successively, and the water content of equilibrium on a dry basis at each measurement point (Xe) which was determined by the relationship between the equilibrium water mass (me) and the dry mass (ms) of the samples (Equation 1).

$$Xe = \frac{m_e - m_s}{m_s} 100 \quad (1)$$

The mathematical models of GAB (Park & Nogueira, 1992), Peleg (Peleg, 1993), Oswin (Chinnan, 1985), Halsey (Halsey, 1985), and Henderson (Henderson, 1952) (Table 1) were adjusted to the experimental data of the water adsorption isotherms of okra powders, using non-linear regression and using the Statistica 7.0 program.

Table 1. Mathematical models used to adjust the water adsorption isotherms of powdered okra.

Description	Model	Equation
GAB	$Xe=\frac{X_m CKa_w}{(1-ka_w)(1-Ka_w+CKa_w)}$	(2)
Peleg	$Xe= K_1(a_w)^{n1}+K_2(a_w)^{n2}$	(3)
Oswin	$Xe= a\left(\frac{a_w}{1-a_w}\right)^b$	(4)
Halsey	$Xe=\left(\frac{-a}{\ln(a_w)}\right)^{\frac{1}{b}}$	(5)
Henderson	$Xe=\left(\frac{\ln(1-a_w)}{a}\right)^{\frac{1}{b}}$	(6)

Where: Xe - equilibrium water content, % dry basis; aw - water activity, dimensionless; Xm - water content in the molecular monolayer, % dry basis; C, K and T - parameters that depend on the temperature and nature of the product, dimensionless; K1, K2, n1, n2, a, b - adjustment parameters of the models, dimensionless.

The criteria used to determine the best fit of the models were the determination coefficient (R^2) and the average percentage deviation (P), calculated according to Equation 7.

$$P=\frac{100}{n}\sum_{i=1}^{n}\frac{|X_{exp}-X_{teor}|}{X_{exp}} \quad (7)$$

Where:

P - average percentage deviation, %;

Xexp - values obtained experimentally, % dry basis;

Xteor - values predicted by the model, % dry basis;

n - number of experimental data.

After obtaining the powders, the morphological analysis of the particles of the powders was also performed, using scanning electron microscopy (SEM) on Shimadzu model SSX-550 Superscan equipment, operating at 15 kV. To obtain the SEM images, the samples were metalized with a gold alloy for 360 seconds, with a current of 10 mA in a Shimadzu IC-50 metallizer, under high vacuum conditions, in order to provide a reflective surface for the electron beams. Then, the samples were visualized under the microscope and the morphological structures were photographed in magnifications of 100X and 1000X.

lso after obtaining the powders, X-ray diffraction analysis was performed on the okra powders with the scanning method, which consists of the incidence of X-rays on the samples in powder form, compacted on a support, using the equipment from brand Shimadzu XRD-6000 with CuKa radiation, a voltage of 40 KV, current of 30 mA, step size of 0.020 2θ and time per step of 1.00 s, with scanning speed of 2° (2θ) / min, with angle 2θ, covered from 4 to 50°. The values of the crystalline planes with a distance of λ wavelength of radiation - X, were determined according to the Bragg equation (Equation 8), which consists of recording the intensity of diffracted rays versus twice the diffraction angle (2θ).

$$n\lambda=2d(hkl)\sin\theta \quad (8)$$

Where:

n - order of reflection;

λ - wavelength of the electromagnetic radiation used;

d (hkl) - distance between the planes;

θ - Crystalline plane angle of Bragg.

Results and Discussion

Table 2 shows the parameters of the GAB, Peleg, Oswin, Halsey and Henderson models, the determination coefficients (R^2) and the average percentage deviations (P) of the adjustments of these models to the data of the water adsorption isotherms, in the temperature of 25 °C, from the okra powders obtained at drying temperatures of 50, 60, 70 and 80 °C.

It is observed that the determination coefficients resulted in values from 0.9896 and the data of P <10% in all models, indicating that the tested models can be used in the prediction of water adsorption isotherms at 25 °C of okra powders.

Among the models evaluated, in general, the GAB model was the one that best adjusted to the experimental data of the isotherms, presenting the highest coefficients of determination (R^2 > 0.9950) and P < 5%. The GAB model has been widely used to represent the isotherms of agricultural products dehydrated by convective drying, with good adjustments to the experimental values of the water adsorption isotherms of a wide variety of materials, such as in dehydrated manioc flour; quinoa starch (Chenopodium quinoa Willd.); green coconut pulp powder, among others (Chisté et al., 2015; Pumacahua-Ramos et al., 2017; Lavoyer et al., 2013).

In the different powder samples obtained at the four drying temperatures, it was observed that the values of the water content of the molecular monolayer (Xm) of the GAB model, which corresponds to the amount of water strongly adsorbed in specific sites of the material, considered relevant to guarantee the stability of the product, which provides the longest storage time with a minimum loss of quality at a given temperature (Botrel et al., 2017); were reduced with the increase of the drying temperature, verifying that the dehydrated powder at a temperature of 80 °C will possibly present less loss in the quality of the product during storage, and consequently greater efficiency in its storage time.

Table 2. Parameters, coefficients of determination (R²) and average percentage deviations (P) of the models adjusted to the water adsorption isotherms, at 25 °C, of the okra powder under different drying temperatures.

Model	Temperature (°C)	Parameters				R^2	P (%)
		X_m		C	K		
GAB	50	9.4339		2.1730	0.9597	0.9988	4.19
	60	9.1218		4.7094	0.9530	0.9955	3.41
	70	8.6914		8.6423	0.9561	0.9991	2.25
	80	8.5930		8.7990	0.9528	0.9995	1.85
Peleg	T (°C)	K_1	n_1	K_2	n_2	R^2	P (%)
	50	47.5439	2.0507	114.5384	13.9828	0.9984	3.78
	60	34.8730	1.2717	96.0329	10.4365	0.9955	4.33
	70	93.0827	9.6069	30.1354	10.0107	0.9982	4.14
	80	32.2135	1.0842	90.5189	10.0159	0.9988	3.42
Oswin	T (°C)	a		b		R^2	P (%)
	50	12.3736		0.7492		0.9985	4.71
	60	14.1990		0.6678		0.9951	3.04
	70	14.4780		0.6465		0.9986	3.11
	80	14.6515		0.6375		0.9993	1.91
Halsey	T (°C)	a		b		R^2	P (%)
	50	15.0597		1.1917		0.9969	6.24
	60	24.0318		1.3161		0.9938	5.11
	70	27.0167		1.3528		0.9982	3.11
	80	28.8567		1.3709		0.9989	2.85
Henderson	T (°C)	a		b		R^2	P (%)
	50	17.8523		1.5226		0.9964	5.61
	60	20.3460		1.3057		0.9896	9.53
	70	20.5335		1.2538		0.9904	9.63
	80	20.7121		1.2314		0.9915	9.77

The differences between the values of Xm obtained for fruits and vegetables in different works can be attributed to the variation of the composition of the product, mainly related to the content of sugars present (Telis & Sobral, 2001). The water content in the monolayer corresponds to the amount of water strongly adsorbed in specific places on the surface of the products, considered a critical value above which the rates of some degradation reactions increase and the matrix stability decreases (Mosquera et al., 2012). Therefore, in low and intermediate water activities, the water content increases almost linearly with the increase in water activity; however, at high values, the water content increases rapidly resulting in the deterioration of the product.

The K parameter remained practically stable with the drying temperature increments, adjusting within the range considered acceptable, between 0.7 and 1.0 (Syamaladevi et al., 2009). The K value provides a measure of the interactions between molecules in the multilayer with the adsorbent, which generally varies between the energy value of the molecules in the monolayer and that of liquid water, that is, if K = 1 the multilayer has the property of water liquid (Farahnaky et al. 2016). The sorption constant C, which corresponds to the function of the interactions between the active sites of the product and the water molecules (Pinto et al., 2019); there was an increase in the parameter with the increase of the drying temperature, observing high interactions between the active sites of the product and the water molecules.

According to the parameters C and K of the GAB model, the isotherms of the okra powders are classified as Type II since they presented $0 < K \leq 1$ and $C > 2$ (Andrade et al., 2017). Type II curves are typical of products with a large amount of sugar and solutes and with low capillarity adsorption, being characteristic of most foods (Stępień et al., 2020).

The Oswin model that has the parameters a and b, and according to Blahovec (2004) must present values of a greater than 0 and of b less than 1 to indicate a mathematical and physical consistency in the interactions of the molecules. These values also indicate that there is no inflection point in the curve and changes in the concavity of the functions, showing that the execution of the experiment was efficient (Alcântara et al., 2009). The parameter b of the Halsey and Henderson models characterizes the type of interaction between the vapor and the solid. In case of high values, the attraction between the solid and the vapor is very specific and does not extend very far from the surface; on the other hand, if the values are close to 0, the predominant forces of attraction are Van der Waals and are capable of acting at great distances from the surface. In the case of adjusted samples, it is observed that the values of b were greater than 1.0; therefore, the attraction between the adsorbed and the adsorbent can be considered low, indicating that okra powders have low interaction strength with water, easily adsorbing it (Halsey, 1985; Waughon & Pena, 2007).

Figure 2 shows the water adsorption isotherms at 25 °C of the okra powders obtained at temperatures of 50, 60, 70 and 80 °C, adjusted by the GAB model. It can be seen that in the aw range between 0 and 0.7, the dehydrated powder at the lowest temperature (50 °C) presents a well-defined distinction concerning the curves for the powders obtained at higher temperatures, indicating a greater effect of the difference dehydration temperature in this sample to the others, which can be justified by the greater water activity present in the sample when starting the experiment.

Figure 3 shows the images referring to the morphological analysis (SEM) of the particles of the okra powders obtained at drying temperatures of 50, 60, 70 and 80 °C. It can be seen in the micrographs that, despite the differences in the dehydration temperature, the microstructures were similar in all powders. The structures are morphologically asymmetrical, with varying sizes and shapes. The irregularity in the shape of the particles of the powders can be attributed to the process of obtaining, since the dehydrated samples were crushed in a knife mill, with the disintegration of the structures, causing random fragmentation and asymmetries, which affect the behavior of the powder, especially the characteristics that they are functions of compactability, such as solubility and flow capacity. According to Ferrari et al. (2012) materials with compacted structures and surfaces contribute to increasing the values of their physical properties such as density and wettability rate.

The variation in the drying temperature did not produce any detectable differences concerning the size of the particles, but it caused the formation of more laminated structures, with figure 3A showing more fixed structures than the others, Figures 3B, C and D showed more fragments. loose, interspersed with voids. According to Harnkarnsujarit et al. (2012) porosity can be influenced by the dehydration temperature, which favors the formation of larger pores and thicker walls.

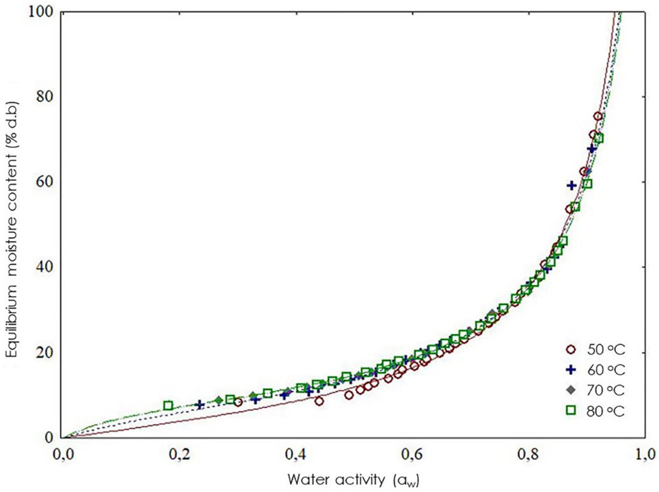

Figure 2. adsorption isotherms of water at 25 °C of okra powders obtained in dryings of 50 to 80 °C, with adjustments by the GAB model.

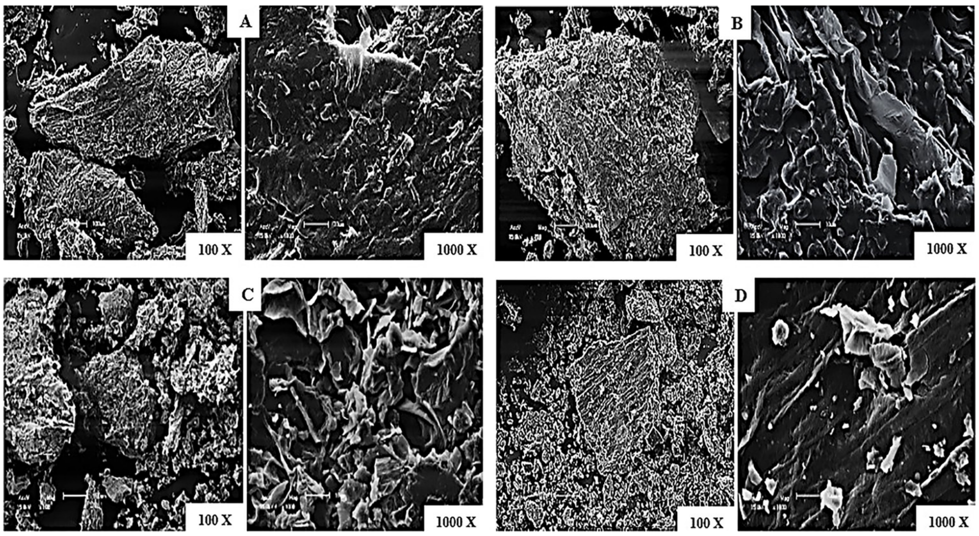

Figure 3. Morphology of the powdered okra particles obtained at the drying temperatures of 50 °C (A), 60 °C (B), 70 °C (C) and 80 °C (D), photographed in magnifications of 100 and 1000 X.

Figure 4 shows the X-ray diffraction analysis of powdered okra obtained at dehydration temperatures of 50, 60, 70 and 80 °C. X-ray diffraction is a common technique used to confirm the crystalline-amorphous state of dry powder products. In general, the crystalline material shows a series of sharp peaks, while amorphous products produce a broad background pattern (Caparino et al., 2012).

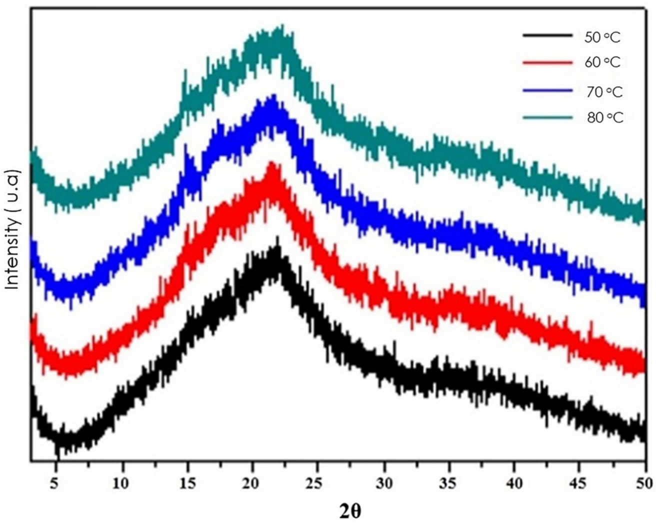

Figure 4. X-ray diffraction spectra of okra powders dehydrated at temperatures of 50 to 80 °C.

The X-ray diffraction patterns of the powdered okra exhibit amorphous characteristics, without the formation of crystalline peaks, with the diffractograms revealing that the amorphous domain of the powders is concentrated in 23 (2θ). It is noted that for the same type of sample, the diffraction intensity is reduced, which is denoted by the tendency of the curves to become flattered or with less difference between minimums and maximums. The rapid drying of the material, in the presence of low molecular weight compounds, tends to produce dry products in the metastable state because of the insufficient time to crystallize, though in drying at a lower temperature the material remains longer on exposure, consequently contributing to the formation of crystalline characteristics (Jayasundera et al., 2011).

Irreversible changes in the structure of polymeric compounds, cause the degradation of their molecular structure and loss of crystallinity (Anastasiades et al., 2002). Mahdavi et al. (2016) stated that more amorphous characteristics are needed for a product to function as a carrier, both for water-soluble and fat-soluble compounds. Thus, the absence of crystalline peaks in the okra powders provides evidence of a possible application as a carrier or drying aid.

This characteristic is constantly verified in the literature in products such as okra, which have mucilages in their constitution, which are secretions of a mixed nature consisting mainly of acid or neutral heteropolysaccharides, proteins and phenolic substances, which are presented as colloidal solutions that in contact with the water becomes slimy (Singh et al., 2014). These gels resulting from different polymers have numerous applications mainly in the food industry in the form of emulsifiers and stabilizers.

Conclusions

The water adsorption isotherms of okra powders obtained at drying temperatures of 50, 60, 70 and 80 °C are classified as Type II, and all tested models can be used for the prediction of isotherms, highlighting the GAB model for the best representation. All powders were presented in morphologically irregular, asymmetrical structures with amorphous characteristics, with the potential to use okra powder as drying aids.

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