

Effect of Dehydration Methods on Okra Chemical and Physical Composition

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Abstract

The agricultural processing industry is continually working to meet consumer demand for new products, diversifying the supply of non-perishable items ready for consumption, conveniently prepared to maintain the main characteristics of the raw material. The objective of this work was to dehydrate whole okra by lyophilization and convective drying at 50, 60, 70 and 80 °C and to evaluate the influence of drying processes on the chemical and physical quality of powdered products. The powders had acceptable contents of lipids, sugars, proteins, pectin, chlorophyll and carotenoids, high hygroscopicity and low solubility. Lyophilization produced powders with characteristics closer to those of the fresh raw material. Regarding the contents of ashes, pectin, lipids and chlorophyll b, the samples obtained by convective drying showed characteristics close and even superior to those of the lyophilized powder.

Keywords: *Abelmoschus esculentus*, lyophilization, convective drying, vegetables

1. Introduction

Okra (*Abelmoschus esculentus* (L.) Moench), belonging to the Malvaceae family, is a commercially important plant native to Africa, and its cultivation is expanding in almost all tropical and subtropical areas of the world (Lengsfeld et al., 2004; Jesus et al., 2014). In Brazil, okra was introduced with the slave trade and spread to all regions of the country, especially in the northeast and southeast, which have a very favorable climate for its development (Jain et al., 2012; Zheng et al., 2014).

Okra is consumed when it is not yet ripe, alone or combined with other vegetables in different preparations (Agbo et al., 2010). It is antihelmintic, antiparasitic, emollient and indicated in the treatment of various diseases such as inflammation in the intestine, stomach and kidneys (Sousa et al., 2015). In addition to being beneficial to the digestive system, it contributes to the proper functioning of the intestine, due to its high content of polysaccharides and microemulsions (Adelakun et al., 2011). Its seeds are good sources of oil, proteins, fats, fibers and sugars (Fan et al., 2014; Mota et al., 2006). It is a rich source of mucilage, with considerable pectin and lignin contents (Kpodo et al., 2017).

Expansion of okra supply and consumption should consider ready and semi-ready products, with extended shelf lives, obtained by applying conservation methods that, in addition to extending the shelf life, add value to the raw material (Gamboa-Santos et al., 2013). Drying is a widely used food preservation process in which water removal minimizes many of the deterioration reactions caused by moisture, which affect the quality of the product. Dried fruits and vegetables and their application as powder have aroused the interest of the food industry (Karam et al., 2016).

According to Chopda and Barrett (2001), there is a wide variety of drying techniques available and the choice of the method should take into account the relationship between the costs involved and the final quality desired. At the extremes of lowest and highest costs are convective drying and lyophilization, respectively. Convective drying has as great advantages the low complexity and the simplicity of equipment, whose functions are mainly aimed at temperature control. When well applied, it can originate products with good quality and at a relatively

low cost. For heat-sensitive nutrients, it may result in losses, especially of volatiles, which represents its main disadvantage.

Lyophilization comprises a drying process in which a sample is previously frozen and then the amount of water is reduced by sublimation and subsequent desorption, undergoing the processes of initial freezing, primary drying and secondary drying (Marques et al., 2009). Its advantages and disadvantages are the opposite of those of the convective drying. It has high costs, time-consuming processing stages and high energy expenditure. On the other hand, it results in products with maximum retention of principles and nutrients of the fresh raw material. Lyophilized foods are products with high added value because they retain much of their original nutrients, since low temperatures are employed in their processing, which is efficient compared with other methods of dehydration (Vieira et al., 2012).

Given the above, this study was conducted to evaluate the dehydration of whole okra by lyophilization and convective drying at temperatures of 50, 60, 70 and 80 °C and evaluate the influence of these processes on the chemical and physical characteristics of the final product.

2. Material and Methods

2.1 Raw Material

The raw material used was ridged okra (*Abelmoschus esculentus* (L.) Moench) of the subgroup green, Santa Cruz variety, at green maturity stage, from the region of Caturité-PB, Brazil.

Okra were taken to the laboratory, manually selected to eliminate those with physical damage or at undesirable maturity stage, then subjected to washing in running water and sanitization in chlorinated water (50 ppm) for 15 min, and rinsed in running water (Silva et al., 2016).

2.2 Drying Methods

For the drying procedures, okras were cut into disc-shaped slices with standardized thickness of 2 mm (Figure 1).

2.2.1 Lyophilization

Okra slices were frozen at -18 °C for 48 hours and taken to the benchtop lyophilizer (Liobras L101) for 72 hours. Then, the slices were ground in knife mill and sieved through 32-mesh stainless-steel sieves to obtain the powder (Figure 2).

2.2.2 Convective Drying

For convective drying, okra slices were placed on stainless-steel trays and subjected to drying in a forced-air oven at temperatures of 50, 60, 70 and 80 °C until reaching hygroscopic equilibrium, determined by weighing the samples until constant weight. After that, the slices were ground in knife mill and sieved through 32-mesh stainless-steel sieves (Figure 3).



Figure 1. Okras cut into slices

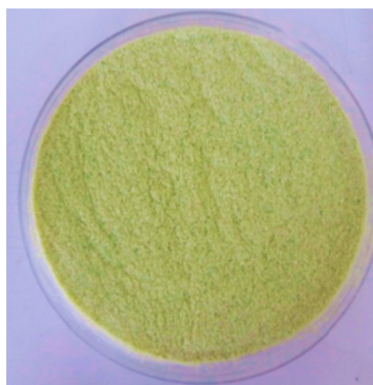


Figure 2. Lyophilized sample powder

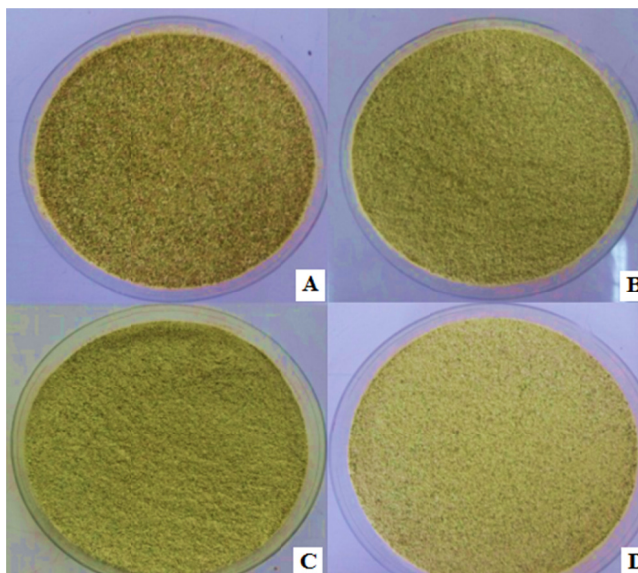


Figure 3. Powders of samples subjected to convective drying at temperatures of 50 °C (A), 60 °C (B), 70 °C (C) and 80 °C (D)

2.3 Chemical and Physical Characterization

2.3.1 Chemical Characterization

The obtained powders and fresh sample were characterized in triplicates with respect to water content, by the standard method of the oven at 105 °C, until constant weight; ashes, by calcination of sample in muffle furnace at 550 °C; total titratable acidity, by the acidimetric method, using 0.1 M sodium hydroxide solution; and proteins, quantified by nitrogen determination using the Kjeldahl method, according to the methodologies described by AOAC (2000).

Lipid content was determined by the method of Bligh and Dyer (1959), and expressed in % on dry basis (d.b.); pectin content was determined by the neutralization of charges of free uronic acid residues by calcium ions, expressed in pectin percentage (d.b.) (Pearson, 1970).

Total sugars were determined according to the methodology of Yemm and Willis (1954), in which the samples were subjected to analysis in spectrophotometer at 620 nm and the values were quantified based on the glucose standard curve. Reducing sugars were determined according to Miller (1959), using 3,5-dinitrosalicylic acid (DNS) as oxidizing agent, with readings in spectrophotometer at 540 nm, and expressed in mg/100 g d.b.

Water activity was determined at 25 °C in an Aqualab water activity meter (3TE-Decagon Devices), whereas pH was determined by the potentiometric method, with a Tecnal TEC-2 pH meter.

2.3.2 Characterization of Pigments and Colorimetric Parameters

Contents of chlorophyll (a, b and total) and carotenoids were quantified after extraction using 80% acetone and calcium carbonate, with absorbance readings in spectrophotometer at 470, 646 and 663 nm, according to Lichtenthaler (1987), and expressed in µg/g on dry basis.

Colorimetric parameters were determined by direct reading in a portable spectrophotometer Hunter Lab Mini Scan XE Plus, model 4500 L, using the Cielab color system, quantifying the parameters: L*-luminosity; a*-transition from green (-a*) to red color (+a*); and b*-transition from blue (-b*) to yellow color (+b*). The values of a* and b* were used to determine the values of chroma (C*) (Equation 1) and hue angle (h) (Equation 2):

$$C^* = \sqrt{(a^*)^2 + (b^*)^2} \quad (1)$$

$$h = \tan^{-1} b^*/a^* \quad (2)$$

2.3.3 Physical Characterization

Hygroscopicity of the powder samples was determined according to the methodology proposed by Cai and Corke (2000), and expressed as grams of adsorbed water per 100 g of sample mass. Solubility was determined

using the method described by Schoch (1964) using a centrifuge at 3200 rpm for 15 minutes, and the supernatant was collected and dried in an oven at 105 °C for 24 h, to determine the soluble mass. Wettability was determined by the method of Schubert (1993), expressed by the following ratio: mass (g)/time required for sample disappearance (min).

Apparent density (ρ_{ap}) was determined using a 10-mL cylinder previously weighed and then filled with the powder, based on the mass/volume ratio. Tapped density was determined with the same setup used to determine apparent density, and the cylinder with the sample was tapped against the bench 50 times from a preestablished height of 2.5 cm, calculating the mass/tapped volume ratio (Tonon et al., 2009).

Carr index (CI) and Hausner Ratio (HR) were determined by the methodology of Wells (1988), calculated from the data of apparent density (ρ_{ap}) and tapped density (ρ_t), according to Equations 3 and 4:

$$CI = \frac{\rho_t - \rho_{ap}}{\rho_{ap}} \times 100 \quad (3)$$

$$HR = \frac{\rho_t}{\rho_{ap}} \quad (4)$$

2.3.4 Scanning Electron Microscopy

Morphological analysis of the particles was carried out in a Shimadzu SSX-550 Superscan scanning electron microscope (SEM), operating at 15 kV. To obtain the images with the SEM, the samples were metalized with a gold alloy for 360 seconds with 10 mA current in a Shimadzu IC-50 metallizer, under high vacuum conditions, to provide a reflecting surface for the electron beams. Then, the samples were visualized in microscope and the morphological structures were photographed with magnifications of 500x.

2.4 Statistical Analysis

The results of chemical and physical analyses were subjected to analysis of variance and the means were compared by Tukey test at 0.05 probability level, using the program Assistat, Beta version 7.7 (Silva and Azevedo, 2009).

3. Results and Discussion

3.1 Chemical Characterization

Table 1 shows the chemical parameters of fresh okra and its powders dehydrated by lyophilization and convective drying at temperatures of 50, 60, 70 and 80 °C. Fresh okra showed high water content, which was already expected because vegetables are usually commercialized with water content equal to or higher than 90% w.b. This condition, according to Franco and Landgraf (2005), favors the development of molds, yeasts and pathogenic bacteria, which requires immediate consumption or processing aiming at minimum shelf life or prolonged periods of storage.

Table 1. Mean values and standard deviations of the parameters evaluated in okra (*Abelmoschus esculentus* (L.) Moench) fresh and dehydrated by lyophilization and convective drying

Parameters	Fresh	Lyophilization	Convective drying			
			Temperature (°C)			
			50	60	70	80
Water content (% d.b.)	840.20±0.21a	4.82±0.06 d	9.12±0.36 b	8.77±0.07 b	7.01±0.34 c	5.59±0.15 d
Water activity	0.994±0.00 a	0.128±0.002 e	0.269±0.002 b	0.227±0.002 c	0.219±0.004 c	0.175±0.011 d
Total titratable acidity (% citric acid, d.b.)	1.05±0.01 a	1.06±0.04 a	0.83±0.02 b	0.61±0.07 c	0.57±0.07 c	0.42±0.03 d
pH	6.29±0.01	6.32±0.00	6.39±0.01	6.43±0.02	6.49±0.00	6.55±0.03
Ashes (% d.b.)	6.49±0.06 b	7.44±0.44 a	7.01±0.06 ab	7.27±0.03 a	7.38±0.12 a	7.39±0.02 a
Lipids (% d.b.)	9.38±0.61 a	3.16±0.22 b	2.97±0.10 b	3.02±0.10 b	3.10±0.29 b	3.69±0.29 b
Proteins (% d.b.)	29.74±0.53 a	25.65±1.19 b	23.54±0.18 c	21.80±0.34 c	19.64±0.46 d	18.98±0.76 d
Total sugars (mg/100 g d.b.)	63.50±0.71 a	39.70±0.07 b	28.14±0.03 c	24.93±0.08 d	21.24±1.99 e	18.55±0.77 f
Reducing sugars (mg/100 g d.b.)	10.06±0.54 a	6.38±0.05 c	7.53±0.25 b	7.45±0.05 b	7.25±0.33 bc	7.18±0.55 bc
Pectin (% d.b.)	13.60±0.34 a	13.69±0.68 a	8.57±1.08 c	12.63±0.14 b	13.41±0.20 a	13.43±0.11 a

Note. Means followed by the same letter in the rows do not differ statistically by Tukey test at 0.05 probability level.

In the powders produced after drying, water content tended to decrease with the increment of temperature in the convective drying processes. There was no statistical difference between the powders dehydrated at temperatures of 50 and 60 °C, with significant reduction in the powder dehydrated at 70 and 80 °C, and the powder obtained at 80 °C was statistically similar to the lyophilized powder. Castro et al. (2017), studying the influence of spouted bed drying at temperatures of 70, 80 and 90 °C on taro (*Colocasia esculenta*), observed that water contents decreased with the increase in drying temperature from 82.05% to 6.81, 6.16 and 5.61% w.b., respectively. The water contents found in the unpeeled okra powders after both drying methods are within the standards of the Brazilian legislation for products dried and processed in the form of powder and flour, which establishes water content of up to 15% (Brasil, 2005).

As observed for water content, the fresh okra had high water activity. Such high values can cause several physicochemical and microbiological alterations during the storage of the product (Bejar et al., 2012) and need to be reduced in order to increase its shelf life and add value. The lyophilized powder had the lowest water activity, with statistical difference ($p < 0.05$) between the powders produced by convective drying, and this variable tended to decrease as drying temperature increased. The low water activity found in okra powders is sufficient to provide stability during storage, which is guaranteed at $a_w < 0.6$ according to Fellows (2000). Monteiro et al. (2018) dehydrated slices of pumpkin (*Cucurbita moschata* var. 'Menina Brasileira') by different methods and obtained water activities of 0.145 and 0.438 for the methods of lyophilization and air circulation oven, respectively. Silva et al. (2017) dried onion and organic beet in air circulation oven at temperature of 70 °C and found water activities of 0.280 and 0.285, respectively.

Okra showed low acidity (TTA), a common characteristic of vegetables, as reported by Pereira et al. (2016), who characterized chard, lettuce and cabbage physicochemically and found contents of 0.08, 0.07 and 0.30% of citric acid (d.b.). Lyophilized okra powder was statistically similar to the fresh material, preserving its acidity after dehydration. In the samples subjected to convective drying, increasing temperature led to a trend of reduction in acidity. According to Melo et al. (2015), this is probably due to the thermal degradation of the organic acids. Since TTA encompasses all acids present in the product, any loss observed in some of the constituent acids can interfere in the results.

The pH in the fresh sample was close to neutrality, with values higher than 6.0. Nascimento et al. (2013) evaluated okras irrigated by different depths of saline water and found pH between 6.04 and 6.19 in okra at the lowest water depth. In powders obtained by convective drying, pH values tended to increase as the drying temperature increased. The lyophilized powder had the lowest value and was the closest one to the fresh sample, indicating lower alteration, followed by the samples obtained at gradually higher temperatures. The inverse correlation between pH and total titratable acidity was observed in all samples, corroborating the effect of heating on both parameters.

For ash contents, there were statistical differences between the fresh material and the powders, with similarity between fresh okra and the powder dehydrated by convective drying at 50 °C and no statistical difference between the other samples dried by convection. Crocetti et al. (2016), characterized beet (*Beta vulgaris* L.), fresh and dehydrated by convective drying (50 °C) and lyophilization, and found ash contents of 8.87 % d.b. in the fresh material and 9.98 and 9.05% in the powders, respectively. Karaman et al. (2014), studying the effects of three drying methods (lyophilization, oven drying and vacuum drying) on the physicochemical properties of persimmon (*Diospyros kaki*), observed that the ashes were statistically stable in the powders obtained through all drying methods.

Regarding the lipids, a degradation was observed after dehydration in comparison to the fresh okra, which had content of 9.38% d.b., whereas the contents in the powders ranged between 2.97 and 3.69% d.b. There was no statistical difference between the powder obtained by lyophilization and those by convective drying, with a slight trend of concentration of lipids as the drying temperature increased. Higher values than those in the fresh material were found by Soares et al. (2012), characterizing okra seed flour, 14.01% w.b. Silva et al. (2016) characterized flours of eggplant (*Solanum melongena* L.) genotypes obtained by convective drying (60 °C) and found values between 4.0 and 6.0%, respectively.

Okra had considerable contents of protein and the dehydration process caused a statistical reduction in these values in comparison to the fresh material. The values in the lyophilized powder were approximately 4% lower, followed by the samples obtained from convective drying, in which the contents decreased gradually with the increment in temperature. Higher value, 22.14% w.b., was reported by Soares et al. (2012) for okra seed flour. Gonçalves et al. (2016), drying peels of green banana (*Musa acuminata*) at temperatures of 55, 65 and 75 °C,

reported reductions in protein contents with the increment of temperature, equal to 7.42, 7.37 and 6.99%, respectively, a behavior similar to that found in the present study.

The contents of total sugars drastically changed after drying, decreasing with the lyophilization, the best treatment, by approximately 37% compared with the fresh sample. The values were progressively lower as temperature increased during the convective drying until a minimum point at 70 °C, which represents a 71% reduction in comparison to the initial value. Guiné et al. (2011), evaluating the drying of pumpkin slices in air circulation oven at temperatures of 30 and 70 °C, observed reduction of 65% in total sugars as the drying temperature increased.

The contents of reducing sugars also decreased after dehydration. However, the convective drying stood out for maintaining the contents of reducing sugars, which were superior or at least statistically similar to those found after lyophilization. A slight decline in the contents of reducing sugars was observed as drying temperature increased, and such degradation is probably due to caramelization processes. According to Lan et al. (2010), the effect of temperature associated with the reduction in water activity leads to rapid increase in browning rate in the Maillard reaction, affecting the composition of the pigment formed, a reaction which involves reducing sugars and amino acids. Similar behaviors in relation to reducing sugars and total sugars were observed by Sojak et al. (2014) in the analysis of quality parameters of giant pumpkin (*Cucurbita maxima*) dehydrated using several technologies of convective drying (chamber dryer, tunnel dryer and fluid bed dryer) at temperatures from 40 to 80 °C. In this study, considerable reductions in the sugars were found at the highest temperatures (70 and 80 °C).

For pectin contents, there was no degradation in the samples dehydrated by lyophilization and at temperatures of 70 and 80 °C by convective drying, which were statistically equal to the fresh material. Wang et al. (2016) found value of 16.70% in the extraction of pectin from the lyophilized mango peel, whereas Kpodo et al. (2017) evaluated pectin contents in six genotypes of okra (*Abelmoschus esculentus* L.), isolated by aqueous extraction at pH 6.0, and obtained values of 11 and 14%, within the same range found in the present study.

3.2 Characterization of Pigments and Colorimetric Parameters

Table 2 shows the values of pigments and colorimetric parameters of fresh okra and its powders obtained by lyophilization and convective drying. Color and pigments are the most important quality attributes influencing the overall acceptance of products by consumers and can be used to visually assess the effects of drying on the product, because these are the bioactive chemical compounds which impart the color observed in the product (Xiao et al., 2014; Huang et al., 2016).

Table 2. Mean values and standard deviations of chlorophylls and colorimetric parameters evaluated in okra (*Abelmoschus esculentus* (L.) Moench), fresh and dehydrated by lyophilization and convective drying

Parameters	Fresh	Lyophilization	Convective drying			
			Temperature (°C)			
			50	60	70	80
Chlorophyll a (µg/g d.b.)	14.21±0.42 a	8.90±0.01 b	7.79±0.09 c	7.50±0.02 d	6.79±0.03 e	5.23±0.03 f
Chlorophyll b (µg/g d.b.)	25.44±0.77 a	2.20±0.01 c	2.63±0.04 b	2.76±0.09 b	2.29±0.05 c	1.80±0.01 d
Total chlorophyll (µg/g d.b.)	38.56±1.12 a	16.06±0.02 b	14.47±0.13 c	14.06±0.06 d	12.62±0.04 e	9.74±0.05 f
Carotenoids (µg/g d.b.)	18.62±0.30 a	3.43±0.01 b	1.90 ±0.05 d	2.08±0.04 d	2.10±0.02 d	2.66±0.05 c
Luminosity (L*)	50.53±0.04 f	70.04±0.02 a	58.41±0.23 e	59.85±0.22 d	60.29±0.11 c	65.80±0.06 b
Green intensity (-a*)	0.72±0.05 e	5.09±0.10 a	3.42±0.03 b	2.90±0.05 c	2.86±0.08 c	1.25±0.05 d
Yellow intensity (+b*)	26.85±0.07 a	23.46±0.03 c	20.73±0.29 f	22.17±0.08 e	22.71±0.15 d	24.51±0.34 b
Chroma (C*)	26.84±0.07 a	24.02±0.04 b	21.23±0.29 d	22.38±0.07 c	22.88±0.15 c	24.59±0.35 b
Hue angle (°)	88.51±0.10 a	77.75±0.23 f	80.68±0.09 e	82.56±0.13 d	82.98±0.17 c	87.08±0.09 b

Note. Means followed by the same letter in the rows do not differ statistically by Tukey test at 0.05 probability level.

Fresh okra has higher contents of pigments and the dehydration process led to their degradation. The lyophilized powder stood out for retaining chlorophyll *a*, preserving approximately 62.63% of the molecule and 41.66% of total chlorophyll, in comparison to the fresh material, statistically differing from the powders obtained by

convective drying. This was already expected because the lyophilization process minimizes the changes in the product's characteristics (Azeredo, 2004).

In the convective drying, increasing dehydration temperature led to gradual reduction of chlorophylls *a*, *b* and total. Such degradation at higher drying temperatures is due to protein denaturation, which leaves the chlorophyll unprotected. Consequently, chlorophyll degradation occurs and this process varies also according to pH and variations of temperature, light and oxygen in the sample (Bohn & Walczyk, 2004). Similar behavior was reported by Reis et al. (2012) in the convective drying of basil (*Ocimum basilicum* L.) leaves, which caused degradation of chlorophylls with the increase in drying temperature.

As occurred with the chlorophylls, carotenoids also degraded in comparison to the fresh material. The lyophilized powder had the highest retention, about 18% of the initial value. In the convective drying, carotenoid contents tended to increase with the increment in dehydration temperature, i.e., greater retention of carotenoids, which may be related to the shorter drying time and lower exposure to hot air. Nóbrega et al. (2014) found similar behavior in acerola residue, a content of 8.99 µg/g in the fresh material and a trend of greater retention of carotenoids as temperature increased, with values between 4.52 and 5.53 µg/g in the powders obtained by fixed bed drying, at temperatures from 60 to 80 °C.

Regarding the colorimetric parameters, the powders showed a significant difference in all measurements in comparison to the fresh sample. Luminosity (L^*) in the powders, in all cases, was higher than that found in fresh okra, with highest value in the lyophilized powder and increasing values in powders obtained by convective drying as the temperature increased. This is probably related to the shorter residence time of the samples in the drying at higher temperatures. Ren et al. (2017), studying the effects of drying methods (freeze drying, hot air drying, oven drying and vacuum oven drying) on organic and non-organic onions (varieties *Red Baron* and *Hyfort*), observed that the freeze-dried material showed greater luminosity compared to the other drying methods. Krumreich et al. (2016), studying 'uvaia' (*Eugenia pyriformis*) dehydrated by lyophilization and convective drying, found luminosity values of 89.19 and 64.76, respectively, corroborating the results in the present study.

The green component ($-a^*$) increased with the drying and maximum difference from the fresh sample was found in the lyophilized powder. The increase in convective drying temperature led to reduction in green color, indicating a less pronounced effect of exposure time in comparison to temperature. According to Buchaillot et al. (2009), during the drying process, magnesium molecules are converted to pheophytin and pyropheophytin, causing a decrease in the green color of the samples. Unlike the other colorimetric parameters, yellow intensity decreased in all powders in comparison to the fresh sample. The values in the powder obtained at 80 °C were the closest ones to those in the fresh sample, followed by the lyophilized powder and the samples from the highest to lowest temperatures.

In relation to chroma (C^*), highest values were found in the fresh material, followed by the powders obtained by convective drying at 80 °C and the lyophilized powder. Chromaticity increased with the increment in the convective drying temperature, varying between 21.23 and 24.59. Chroma values, when they are close to zero, correspond to neutral colors (gray) with greater opacity of the samples; however, values close to 60 are equivalent to vivid colors, i.e., more intense shades (Mendonça et al., 2003). Similar values were found by Oliveira et al. (2016) in 'baru' (*Dipteryx alata* Vogel) dried at temperatures of 80 and 100 °C in air circulation oven, 22.24 and 24.26.

Hue angle (h) exhibited the same behavior of chroma, with higher values in the fresh sample and in the powder dried at 80 °C, followed by the other powders dehydrated at gradually lower temperatures, differing from the lyophilized powder, which had the lowest value. All values were distant from the red region, which corresponds to the angle $h = 0^\circ$, and were close to the yellow region, equivalent to the angle $h = 90^\circ$ (Alves et al., 2008). Alessi et al. (2013) observed average hue angles of 46.32 in fresh mini tomatoes (*Sweet Grape*) and 45.24 in tomatoes dehydrated in the oven at temperature 60 °C, corroborating the data found here.

3.3 Physical Parameters of the Powder

The mean values of physical parameters for okra powder dehydrated by lyophilization and convective drying are presented in Table 3. High hygroscopicity was observed in all powders studied and, according to Gea (2006), powders with hygroscopicity higher than 25.0% are extremely hygroscopic. Highest value was found in the powder obtained at temperature of 50 °C, followed by reductions as temperature increased, with the lyophilized powder in intermediate position. Fernandes et al. (2014), in powders from the pulp of tomatoes cv. 'Saladete', dehydrated at temperatures of 60 and 80 °C in air circulation oven, found hygroscopicity values ranging from 41.40% to 57.88%. These authors observed that hygroscopicity is directly linked to water content, so that powders with lowest water contents were the least hygroscopic. Hygroscopicity assessment in foods is closely

associated with the physical, chemical and microbiological stability of these products (Tonon et al., 2009), allowing one to select the most suitable package to store the samples taking into account the relations between product value, package cost and permeability level of package.

Table 3. Mean values and standard deviations of the physical parameters evaluated in okra powders dehydrated by lyophilization and convective drying

Parameters	Lyophilization	Convective drying			
		Temperature (°C)			
		50	60	70	80
Hygroscopicity (%)	82.97±0.38 bc	86.64±0.98 a	83.98±0.02 b	82.94±0.03 bc	81.81±0.54 c
Wettability (g/min)	2.05±0.04 a	0.91±0.03 c	1.62±0.05 b	1.62±0.01 b	2.12±0.09 a
Solubility (%)	62.08±1.01 a	29.39±0.23 e	31.02±0.58 d	34.40±0.56 c	40.42±1.24 b
Apparent density (g/cm ³)	0.122±0.01 d	0.511±0.06 a	0.510±0.01 a	0.484±0.01 b	0.355±0.01 c
Tapped density (g/cm ³)	0.193±0.01 e	0.631±0.02 a	0.628±0.01 b	0.572±0.01 c	0.418±0.01 d
Carr Index (%)	36.67±2.89 a	19.00±1.73 b	17.33±0.59 c	15.33±0.58 d	15.00±0.00 d
Hausner Ratio	1.58±0.07 a	1.23±0.03 b	1.21±0.01 b	1.18±0.01 c	1.18±0.00 c

Note. Means followed by the same letter in the rows do not differ statistically by Tukey test at 0.05 probability level.

For wettability, the highest values were found in the powders obtained by lyophilization and convective drying at 80 °C, which did not differ statistically. In powders obtained by convective drying, wettability tended to increase as drying temperature increased, which can be related to the lower water content obtained under this condition. Wettability rate is characterized by the susceptibility of the particles to be penetrated by water and is related to both food chemical composition and physical factors, especially size and shape of particles and temperature of the reconstitution water (Tonon et al., 2009). Duarte et al. (2017), subjected fruits of the Cerrado region, ‘marolo’ (*Annona crassiflora*) and ‘cagaita’ (*Eugenia dysenterica*), to lyophilization and found wettability rates of 4.54 and 1.15 g/min, respectively, the latter of which was close to the values found in okra powders.

Powder solubility is an important property of food raw materials since slightly soluble powders may lead to difficulties in processing and result in economic losses for the industry (Sharma et al., 2012). The powder obtained by lyophilization stood out from the others with the best solubility and, as observed for wettability, followed by the samples dried at the highest temperatures, significantly decreasing until temperature of 50 °C was reached. Higher values have been found by Caparino et al. (2012) in lyophilized whole mango pulp powder, 89.70%, and by Franco et al. (2016) in yacon (*Smallanthus sonchifolius*) powder obtained by convective drying at temperatures of 50, 60 and 70 °C, in the range from 80.89 to 84.16%. Kuck and Noreña (2016) claim that the lower the water content, the more soluble the product, and this behavior is corroborated by the data found in the present study.

Highest values of apparent density were found in powders obtained at the lowest temperatures of convective drying; these values gradually decreased with increasing temperature but were all higher than those found in the lyophilized powder. A correlation between apparent density and solubility was observed, suggesting that the lower the density the higher the solubility and, according to Sogi et al. (2015), this leads to lower formation of lumps. Similar values to those observed in powders obtained by convective drying were found by Ahmed et al. (2016), who evaluated the effects of particle size of commercial lentil flours and found mean apparent density of 0.480 to 0.600 g/cm³. Similar value to that found in the lyophilized powder was reported by Sogi et al. (2015) for Tommy Atkins mango powder obtained by lyophilization, which showed apparent density of 0.170 g/cm³.

Tapped density ranged from 0.193 to 0.631 g/cm³; its lowest value was found in lyophilized powder and its highest value was found in the powder obtained by convective drying at 50 °C, as observed for apparent density. Similar values of tapped density were found by Fernandes et al. (2014) in tomato powder produced by foam mat drying at temperatures of 60 and 80 °C, in the range from 0.180 to 0.454 g/cm³.

The mean values of Carr index (CI), or compressibility index, decreased from the lyophilized powder to the powders produced at the highest temperatures, not differing statistically between the temperatures of 70 and 80 °C, but with the same trend of reduction. The Carr index measures the flowability of powders: good, 15-20%; fair, 20-35%; bad, 35-45%; and very bad, CI > 45% (Santhalakshmy et al., 2015). Based on these criteria,

powders obtained by convective drying at all temperatures evaluated are considered as of good (CI from 15 to 20%), whereas the lyophilized powder is classified as of bad (CI from 35 to 45%). Caliskan and Dirim (2016) dried sumac (*Rhus coriaria*) with 20% of maltodextrin by atomization and lyophilization, and observed flowability of 33.94 and 25.02%, respectively.

The Hausner Ratio (HR), which corresponds to the relationship between tapped and apparent density and is used to evaluate the cohesiveness of powder products, exhibited the same behavior as the Carr Index, with highest value obtained in the lyophilized powder and consistently decreasing values in the powders obtained by convective drying, as temperature increased. According to Santhalakshmy et al. (2015), powders with $HR < 1.2$ are classified as of low cohesiveness, HR from 1.2 to 1.4 as of intermediate cohesiveness and $HR > 1.4$ indicate powders with high cohesiveness. Therefore, the lyophilized powder showed high cohesiveness, powders obtained by drying at 50 °C (1.23) and 60 °C (1.21) showed intermediate cohesiveness, and powders obtained at 70 and 80 °C (1.18) showed low cohesiveness.

3.4 Analysis of Scanning Electron Microscopy

Figure 4 shows the photomicrographs taken by the scanning electron microscope (SEM) of the okra powders obtained by lyophilization and convective drying at temperatures of 50, 60, 70 and 80 °C. Despite the difference between the drying methods employed and between the four temperatures used in the convective drying, the microstructures were similar in all powders. Morphologically asymmetric structures with varied sizes and shapes were observed, and the microstructures of powders obtained by lyophilization (Figure A) and convective drying at 80 °C (Figure E) were smoother and with less roughness than those of powders produced at temperatures of 50 to 70 °C. Irregularity in the shape of particles of the powders can be attributed to the obtaining process, since dehydrated samples were ground in knife mill, which caused disintegration of structures, random fragmentation and asymmetries.

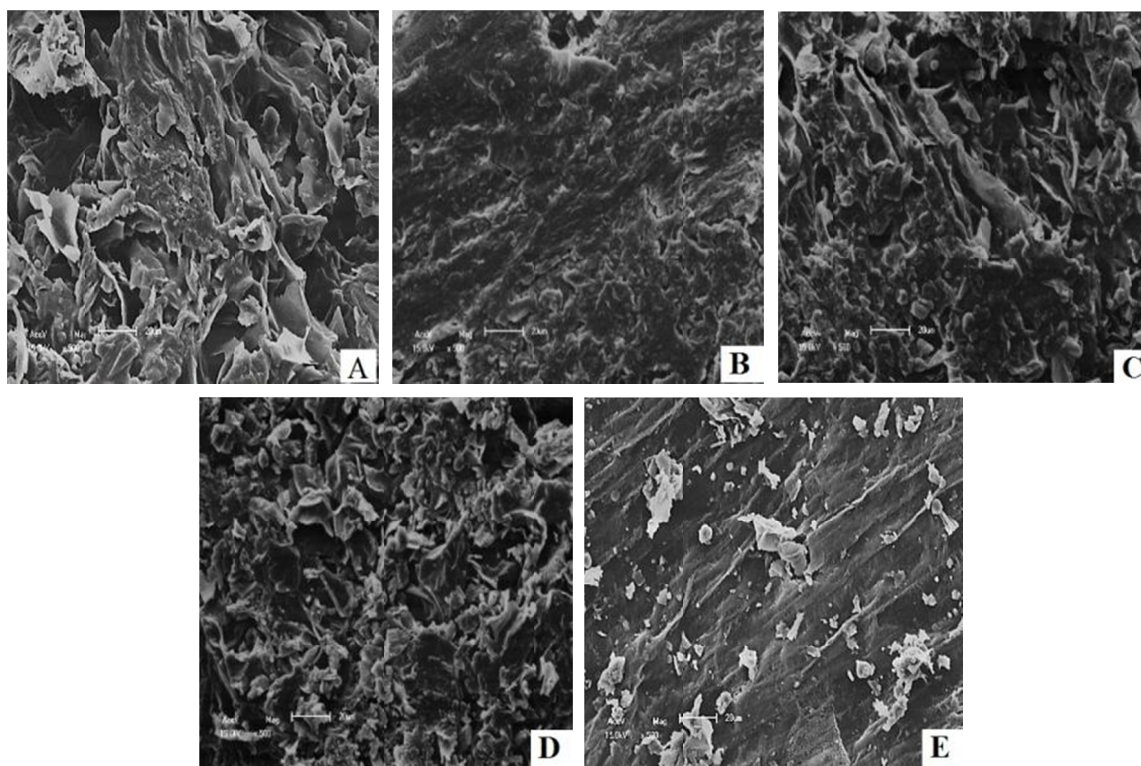


Figure 4. Morphology of okra powders obtained by lyophilization (A) and convective drying at 50 °C (B); 60 °C (C); 70 °C (D); and 80 °C (E)

The high porosity of the lyophilized powder, quantified by the apparent density, can be associated with the somewhat loose layers of material interspersed with voids, identified in Figure A. According to Harnkarnsujarit et al. (2012), porosity can be influenced by the type of freezing used, and freezing temperatures close to -18 °C favor the formation of larger pores and thicker walls, whereas lower freezing temperatures lead to the formation

of smaller pores and thinner walls. Caparino et al. (2012) dehydrated whole mango pulp using different drying methods (Refractance Window[®] drying, drum drying, spray drying and lyophilization) and, by analyzing the microstructure of the samples, found that lyophilization led to the formation of more porous powders compared to the other types of drying.

In powders obtained by convective drying, the increase in dehydration temperature caused lower porosity and lower presence of irregular surfaces up to temperature of 80 °C, and their appearance was similar to that of the lyophilized powder but differs from it for having fewer voids. Agglomeration of the structures, possibly due to higher water content, was observed particularly in the powders obtained at 60 °C (Figure C) and 70 °C (Figure D).

4. Conclusions

The powders had acceptable contents of lipids, sugars, proteins, pectin, chlorophyll and carotenoids, high hygroscopicity and low solubility. Lyophilization produced powders with characteristics closer to those of the fresh raw material. Regarding the contents of ashes, pectin, lipids and chlorophyll b, the samples obtained by convective drying showed characteristics close and even superior to those of the lyophilized powder.

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