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# Production and Physical and Physicochemical Characterization Powder *in Natura* and Freeze-Dried of Moringa Seeds

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**Abstract**

To apply low temperatures, different of other processes, maintain the structure of products and better preserve the thermosensitive components, the freeze-drying has called attention of various researchers. Aimed with the research to produce and characterize the powder *in natura* obtained by seeds of moringa, elaborate different pastes with addiction of 20, 30, 40 and 50 mL of distilled water, freeze-dry it, characterize it physical and physicochemical and select the best powder. The freeze-drying was produced through a benchtop freeze dryer. After drying it, from the powder were determined the real density, bulk density, compacted density, porosity, compressibility index, Hausner factor, hygroscopicity, solubility, color, moisture content and water activity, ashes, total acidity, pH, proteins, lipids and carbohydrates. The *in natura* powder was classified as non-hygroscopic, high solubility in water, low moisture content and water activity, high protein, lipids, carbohydrates and low acidity. Freeze-drying was presented as an appropriate method for the preservation of moringa constituents, with a formula selected with the addition of 50 mL of water.

**Keywords:** constituents, extract, *Moringa oleifera*, drying

## 1. Introduction

The *Moringa oleifera* is a tree of extreme importance known as the tree of life, because of its different properties and applications (Bichi, 2013). The seeds are rich in proteins, calcium, iron, vitamin C and carbohydrates. For this reason the moringa is being used in medicine, cosmetics, food supplements and mainly as coagulant for the treatment of water (Ghebremichael et al., 2009).

The seeds of moringa present higher potential of coagulation/flocculation than the other parts of the plant. One of the active components presented in the seeds of moringa is the protein identified as a polyelectrolyte, capable to promote the coagulation of water (Nwaiwu et al., 2012). The protein of seeds of moringa is the compound of greater importance in the process of clarification of water. Thus, the coagulation action of moringa is based in the presence of cationic protein found in the seeds, being the absorption and the neutralization of charges, possibly, the moringa main mechanism of coagulation (Ndabigengesere et al., 1995).

Considering that the seeds of moringa present better potential on the treatment of water, make it necessary to apply technologies that preserve its constituents. Among the technologies, the freeze-drying stands out because it preserves some of the material properties, due to the use of low temperatures. The freeze-drying has being used and recommended to dry products of high added value that have aroma or delicate textures or present sensibility to heat. Hamid et al. (2016) report that the process of drying of moringa can be assured not to exceed 60 ºC avoiding the moisture content of protein be damaged.

Baptista et al. (2015), Madrona et al. (2017), and Conceição et al. (2015) have studied the use of moringa seeds in water treatment. Although exists many works about treatment of water using seeds of moringa, there were not found records of works on the efficiency of these natural coagulant obtained from the freeze-dried powder. Furthermore, it was not found records in literature about the physical and physicochemical quality on the extract of seeds of moringa after freeze-drying. Thus, aimed as objective to produce and characterize the powder *in* *natura* obtained by seeds of moringa, elaborate different pastes with addiction of 20, 30, 40 and 50 mL of distilled water, freeze-dry it, characterize physical and physicochemical and select the best powder.

## 2. Material and Methods

### *2.1 Place of Conduction of the Research*

The work was conducted at the Laboratory of Processing and Storage of Agricultural Products, of the Federal University of Campina Grande, Campina Grande, Brazil.

### *2.2 Raw Material*

The seeds used on the research were collected at Federal University of Campina Grande Campus in the city of Cajazeiras, Brazil. At the Laboratory of Processing and Storage of Agricultural Products, the seeds were selected, peeled and milled in domestic blender.

### *2.3 Elaboration of Pastes for Freeze-Drying*

Freeze-dried in a benchtop freeze dryer model L101. Added 20, 30, 40 and 50 mL of distilled water to form the pastes (Figure 1). Following, placed the pastes in plastic forms and submit to freezing in a freezer up to -18 ºC for 24 hours (Santos, 2016). After, freeze-dried the freeze samples were to the temperature of -54 ºC for 72 hours.

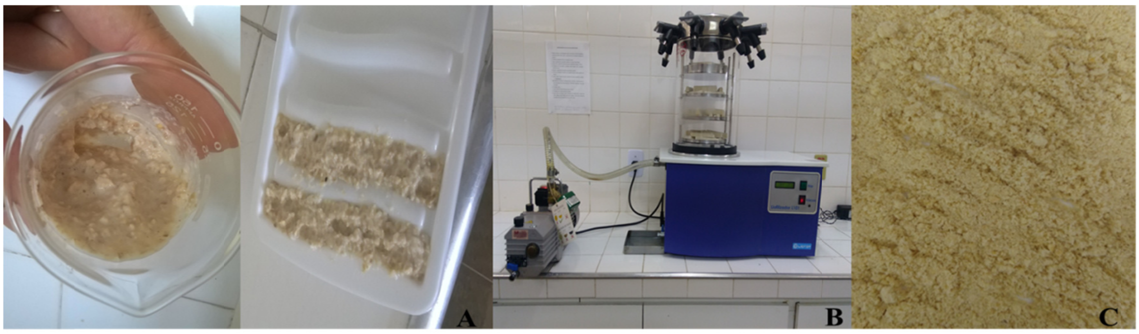


Figure 1. Obtaining freeze-dried powder

*Note.* A: production of pastes; B: freeze drier; C: freeze-dried powder.

### *2.4 Physical and Physicochemical Analyzes of Powder in Natura and Freeze-dried*

On the powder *in natura* and freeze-dried used the physical and physicochemical analyses of real density according to the methodology of Hawkes (2004) adapted; bulk density, according to the adapted method of Caparino et al. (2012); determined the compacted density according to Tonon et al. (2013); the porosity by the method of Krokida and Maroulis (1997); the compressibility index (CI), through the comparison between bulk density and the compacted density of powder, according to Yusof et al. (2012); the Hausner factor from the bulk and compacted density, Hausner (1967); calculated the hygroscopicity according to proposed method of Goula and Adamopoulos (2010), with modifications; determined the solubility according the methodology of Durigon (2016) modified; obtained the color in colorimeter with the data of a* and b* calculated the Chroma (C*), and the parameters L*, a* and b* obtained the browning index, according to methodology adapted from Palou et al. (1999).

The following determinations, with the exception of lipids, were performed according to methodology proposed by Instituto Adolfo Lutz (2008): the moisture content of water was determined by the drying method of the samples in a greenhouse up to 105 ºC; the activity water through the direct reading in "Aqua-Lab", model 4TE; obtained the ashes by incineration of samples in furnace; the total acidity by titulometry and the pH by direct reading of samples homogenized in digital pHmeter; quantified the moisture content of protein by the method of Micro-Kjeldahl; obtained the quantity of lipids by modified method of Bligh and Dyer (1959) and quantified the carbohydrates removing of 100 the sum of moisture contents of water, lipids, proteins and ashes.

*2.5 Statistic Analyzes*

The completely randomized design was adopted, with four treatments (20, 30, 40 and 50 mL) and three repetitions. Submitted the results to analyses of variance and compared the averages by the teste of Tukey with 5% of probability using the statistic software Assistat 7.7 (Silva & Azevedo, 2016).

## 3. Results and Discussion

On Table 1, there are the results obtained by the physical analyses of the powder *in natura* obtained from seeds of moringa. Similar results from this work on density were reported by Medeiros and Lannes (2010) when verified the physical properties of substitutes of cocoa in powder obtaining values of density varying between 0.49 and 0.69 g $mL^{-1}$. Phomkaivon et al. (2018) when evaluating the flour of purple sweet potato obtained values of bulk density between 0.276 and 0.389 g $cm^{-3}$. Similar results for compacted density were reported by Lavoyer (2012) when studying the powder obtained from coconut, with values between 0.58 and 0.62 g $mL^{-1}$. Other very important characteristic of a powder is its porosity, in other words, the fraction of the volume of powder that is not occupied by particles. This research noticed lower value of porosity of powder *in natura*.

Table 1. Physical characterization of powder *in natura* obtained from seeds of moringa

| Physical parameters | Average and standard deviation |
|---|---|
| Real density (g $mL^{-1}$) | 0.59±0.006 |
| Bulk density (g $mL^{-1}$) | 0.48±0.007 |
| Compacted density (g $mL^{-1}$) | 0.62±0.004 |
| Porosity (%) | 18.77±0.85 |
| Compressibility index (%) | 21.44±0.76 |
| Hausner factor | 1.27±0.01 |
| Hygroscopicity (%) | 1.43±1.98 |
| Solubility (%) | 74.03±1.00 |
| Luminosity L* | 67.63±0.16 |
| a* | 2.84±0.05 |
| b* | 20.37±0.10 |
| Chroma C* | 20.57±0.10 |
| Browning index (%) | 13.47±0.59 |

The indirect measures of fluidity or drainage of powder *in natura* were measured through the calculations of compressibility index and Hausner factor. In classification of Santhalakshmy et al. (2015), the powder *in natura* is poor in fluidity. The fluidity depends of various factors as morphology, size and distribution of particles, density, moisture content, production process and chemical composition of material (Lavoie et al., 2002). For the Hausner factor, the powder presented intermediate cohesiveness, according to classification of Wells (2005), in which the values lower than 1.2 are classified as of low cohesiveness; between 1.2 to 1.4 are intermediate cohesiveness; and >1.4 are considerate of high cohesiveness.

Depending of its use, the high hygroscopicity constitutes difficulty to use the product by high affinity to water and due to its complex composition (Carlos et al., 2005). Possibly, it is not going to be a problem for the powder of moringa, because according to the classification of Gea Niro Research Laboratory (2003), the powder *in natura* was considerate not hygroscopic. The powder of seeds presented high solubility in water, this parameter is elementary for the dissolution of powder in liquid. Oliveira et al. (2013) studying the solubility of particles of moringa obtained values of solubility of 87.15 to 91.44%, therefore, higher than those obtained in this research.

One of the attributes of quality and consumer preference is the color, thus, for the parameters of color the sample *in natura* presented lighter color, there was predominance of yellow color (+b*) over the red color (+a*), lower intensity or color saturation, this attribute is totally independent of tonality and of luminosity; noticed lower browning index.

For physicochemical analyzes noticed on Table 2, that the powder *in natura* of seeds of moringa presented lower concentration of water, according to what recommends the legislation that describes the maximum limit of acceptance of 15% of moisture content (Brasil, 2005). Bolarinwa et al. (2019) obtained moisture content ranging from 20.0 to 22.90% when making bread fortified with powder obtained from moringa seeds. In terms water activity, Ribeiro and Seravalli (2007) indicated the food can be classified in function of three groups: food of lower moisture content ($a_w$ until 0.6); intermediate moisture content ($a_w$ between 0.6 and 0.9) and with high moisture content ($a_w$ with values higher than 0.9). Considering the exposed data, powder water activity was classified as intermediate, what can be difficult for the growing of fungus and bacteria.

Table 2. Physicochemical characterization of powder *in natura* obtained from seeds of moringa

| Physicochemical parameters | Average and standard deviation |
|---|---|
| Moisture content (%, w.b*) | 5.24±0.01 |
| Water activity ($a_w$) | 0.62±0.006 |
| Ashes (%) | 2.77±0.004 |
| Total acidity (%) | 0.24±0.03 |
| pH | 6.67±0.06 |
| Proteins (%) | 31.92±0.32 |
| Lipids (%) | 31.46±1.19 |
| Carbohydrates (%) | 28.59±1.43 |

*Note.* * Wet basis.

In study carried out by Passos et al. (2012) with the powder of moringa, the authors found 0.95% of ashes, value lower than what was verified in this research. The powder presented lower acidity, attending the requirements foreseen by Brazilian legislation that determine a minimum of 0.8% of acidity in citric acid (Brasil, 2005). Passos et al. (2012), found pH value of 7.47%, being higher to those obtained in this work.

Teixeira et al. (2013) when evaluating the hamburger elaborated with different concentrations of wheat of leaves of moringa obtained values of proteins of 21.12, 20.68, 20.34 and 20.20%, and for lipids the authors verified quantities of 7.30, 6.73, 7.06 and 6.68%. Moyo et al. (2011) evaluating the chemical composition of dry leaves of moringa the authors found protein value of 30.29%. Passos et al. (2012) found for the powder of moringa values lower than those found in this work for proteins (23.29%) and lipids (17.37%). Gandji et al. (2018) studying the chemical composition of powder of the dry leave of moringa verified 21.1% of proteins and 38.2% of carbohydrates. According to Zaku et al. (2015), all parts of the plant of moringa have important nutrients ratio to absorption our organism. The leaves, as the seeds, are rich in proteins, minerals, beta-carotene and antioxidants composts that are frequently deficient among the diets of the population of countries in development (Leone et al., 2015).

Observed a pattern among the formulations, real density presented a variation of 0.05 g $mL^{-1}$, which values obtained for bulk density presented no statistical difference (Table 3). The density of a powder is directly connected to the structure of particles and, consequently, the capacity of flow and compression, representing an important parameter on the stage of development of a formulation, although, there is no scale to determine the limits (Murakami et al., 2009). Observed that the compacted density reduced with the addition of water, adding 20 mL to the one that presented higher value and 50 mL to the one of lower compaction. The powders presented values of density very different to those obtained by Zea et al. (2013) for the mix of guava and dragon fruit freeze-dried (1.474 and 1.503 g $cm^3$), respectively. According to Ceballos et al. (2012), density is one of the factors that interfere in the wettability of powders, important characteristic to tackle on the first stage of reconstitution of a product in powder. The porosity also increased with the addition of water, observed the freeze-dried powder is much porous and with tendency to present lower densities, because the pores have small size.

In respect to values to index of compressibility and Hausner factor, the addition of 50 mL resulted in lower value, differing statistically from the others. The freeze-dried powder, fits the classification of Santhalakshmy et al. (2015) with the flow poor and bad, respectively, once the values between 15 and 20% indicate good flow, 20-35% poor flow, 35-45% indicate bad flow and higher that 45% very bad flow. Distinct results (15.89 to 25.02%) were obtained by Caliskan and Dirim (2016) when studying the freeze-dried powder of Rhus coriaria. The determination of properties of flow of powder assists in the projection of equipment for storage, transport, or general manipulation of solids in bulk (Staniforth, 2005). For the Hausner factor, the powders presented intermediate cohesiveness for the addition of 50 mL and high cohesiveness for the other additions, according to the classification of Wells (2005).

Table 3. Physical characterization of freeze-dried powder *in natura* obtained from seeds of moringa

| Physical parameters | Formation of freeze-dried paste | | | | |
|---|---|---|---|---|---|
| | 20 mL | 30 mL | 40 mL | 50 mL | CV% |
| Real density (g $mL^{-1}$) | 0.39b | 0.44a | 0.43ab | 0.44a | 3.91 |
| Bulk density (g $mL^{-1}$) | 0.34a | 0.32a | 0.31a | 0.30a | 0.4 |
| Compacted density (g $mL^{-1}$) | 0.54a | 0.51b | 0.49c | 0.40d | 1.18 |
| Porosity (%) | 15.93c | 24.49b | 29.11ab | 32.01a | 3.80 |
| Compressibility index (%) | 37.19a | 37.44a | 37.15a | 25.40b | 2.57 |
| Hausner factor | 1.59a | 1.61a | 1.59a | 1.34b | 3.01 |
| Hygroscopicity (%) | 2.51a | 2.74a | 2.61a | 2.60a | 0.55 |
| Solubility (%) | 75.42a | 74.64a | 76.21a | 73.88a | 2.54 |
| Luminosity L* | 73.14b | 75.52a | 75.36a | 75.00a | 0.12 |
| a* | 1.65a | 1.64a | 1.66a | 1.61a | 0.30 |
| b* | 19.49a | 18.77b | 17.86c | 16.94d | 1.02 |
| Chroma C* | 19.57a | 18.77b | 17.98c | 17.11d | 0.56 |
| Browning index (%) | 10.84a | 9.82a | 9.80a | 8.64a | 2.81 |

*Note*. The measures followed by the same letter do not differ statistically with each other by the test of Tukey to 5% of probability. CV: Coefficient of Variation.

Different statistics were observed among the powders on the hygroscopicity and the solubility. The hygroscopicity of powders are related to capacity to absorb the moisture content of the environment. According to classification of Gea Niro Research Laboratory (2003), the powders can be classified as not hygroscopic (< 10); slightly hygroscopic (10.1-15.0); very hygroscopic (20.1-25.0); extremely hygroscopic (> 25.0); based on these values of reference, the powders were classified as not hygroscopic. Cavalcante et al. (2017) evaluating the parameters of drying of pulp of soursop in spray dryer verified that the hygroscopicity of the powders variated from 7.56 to 13.12%, being such slightly hygroscopic. In terms of solubility, the powders present high solubility, promising results for the objective of the work, because the solubility developed a key role on the dissolution of powders in water. Sogi et al. (2015) obtain similar values (66.80 to 78.69%) of solubility in study with different process of drying of Tommy Atkin mango.

The powders present high luminosity with values above 70. About the colors a* and b*, verified that the powder *in natura* presented statistic difference only for the parameter b*, having the powders presented predominance of yellow. For Chroma C* observed decrease with the addition of distilled water on the formation of freeze-dried paste, being the addition of 50 mL the one which presented lower value. The index of browning of lower powders, indicating the prospective of not occurrence of enzymatic browning or not enzymatic during possible storage. The color of powder can be affected by countless variables such as: genotype, process of milling, drying, storage, among other factors (Pathare et al., 2013).

On Table 4, there are described the average values obtained for the physiochemical characterization of freeze-dried powder. The powders presented lower moisture content of water, though are according to what is stablished by Health Surveyence National Agency that permits the maximum of 5% of freeze-dried products, (Brasil, 1978).

Table 4. Physicochemical characterization of freeze-dried powder *in natura* obtained from seeds of moringa

| Physicochemical parameters | Formation of freeze-dried paste | | | | |
|---|---|---|---|---|---|
| | 20 mL | 30 mL | 40 mL | 50 mL | CV% |
| Moisture content (%, d.b*) | 1.73a | 1.82a | 1.68a | 1.76a | 3.34 |
| Water activity ($a_w$) | 0.051a | 0.053a | 0.051a | 0.051a | 1.09 |
| Ashes (%) | 2.90bc | 3.02ab | 2.82c | 3.19a | 2.38 |
| Total acidity (%) | 0.48a | 0.45a | 0.54a | 0.54a | 3.80 |
| pH | 5.93 | 6.12 | 5.40 | 5.34 | 2.51 |
| Proteins (%) | 33.96a | 33.80a | 34.22a | 34.31a | 1.68 |
| Lipids (%) | 27.33b | 27.14b | 38.12a | 30.96ab | 2.05 |
| Carbohydrates (%) | 34.06a | 34.20a | 23.14b | 29.77ab | 2.11 |

*Note*. The measures followed by the same letter do not differ statistically with each other by the test of Tukey to 5% of probability. *: Dry basis. CV: Coefficient of Variation.

In terms water activity, the powders were classified as having low water activity, according the classification of Ribeiro and Seravalli (2007). When analyzing the profile and the physicochemical characterization of masses of tamarind enriched with leaves of moringa, Andriambelo et al. (2015), verified values of activities varying between 0.49 and 0.59. Also in accordance with the authors, to avoid the risk of chemical deterioration of food such as lipid oxidation, enzymatic and not enzymatic browning, developing of microorganisms during the conservation of food in environment temperature, the value activity water must be between 0.2-0.3.

Observed statistic difference for the ashes. In study about pulps commercialized in Alagoas, Temóteo et al. (2012) observed the acidity of 0.94% in citric acid for the powder of freeze-dried pulp of acerola. This value is higher than the one found on the study for freeze-dried powder of *M. oleifera*. Different results (3.18%), also, were found by Oliveira (2012) when drying through freeze-drying the pulp of cajá.

Moreira et al. (2013) obtained lower value for the pH (3.75) and higher for acidity (3.28%) in the pulp of dehydrated Tommy Atkin mango by freeze-drying. The pH is one of the intrinsic factors to products that are related to the development of microorganism, enzymatic activities, retention of flavor, odor and of general conservation of product (Sousa et al., 2018). In function of this parameter, according to Souza et al. (2008), the food can be classified in: slightly acid (pH > 4.5), acids (4.0 to 4.5) and very acid (< 4.0). In face of this classification, the powders presented lower acids.

The quantity of proteins in formulations not presented difference. Considering the resolution - RDC nº 54, that Dispose about the Technical Regulation on Complementary Nutritional Information, the physicochemical analyzes showed that the powder can be considerate with higher moisture content of protein, since that has quantity higher that those stablished for this RDC (minimum of 12 g by 100 grams), (Brasil, 2012). Macambira et al. (2018) found in the chemical composition of the bran of leaves of moringa 18.31% of proteins. All parts of moringa are rich in nutrients and favorable compounds to the well functioning of organism

The powder presented great quantities of carbohydrates. The high moisture content in carbohydrates is indicative of vegetal potentially energetic. Basso (2017) verified in this study the chemical composition of jackfruit that the process of freeze-drying not reduced to quantity of ashes, proteins and lipids. Celestino (2010) quotes as advantages of freeze-drying the concentration of nutritional compounds, increasing the value o these products. Affirmative that in part is in accordance with Ghribi et al. (2015), Oberoi and Sogi (2015), and Samoticha et al. (2016), that proved on their researches the efficiency of process of freeze-drying on the preservation of its constituents.

The powder can be considerate to have quality, because present lower moisture content and water activity, not hygroscopic, and are of higher solubility. Such characteristics are commonly described as important for products in powder (Bakar et al., 2013). This work is one of the pioneers in the production, study and physical and physicochemical characterization of freeze-dried powder of seeds of moringa. For this reason there are not many sources of comparison with these aspects.

## 4. Conclusion

The powder *in natura* was classified as not hygroscopic of high solubility, low moisture content and water activity, high of protein, lipids and carbohydrates and of low acidity. For the physical analyzes of freeze-dried powder, the addition of 50 mL of distilled water promoted lower values of compressibility index and Hausner factor and higher porosity. Classified the freeze-dried powder as not hygroscopics and of high solubility in water. The freeze-drying caused reduction of moisture content and activity water, turning the freeze-dried powders more stable and contributing to the maintenance of its physicochemical and nutritional quality. The freeze-drying presented for the moringa as appropriate method in the preservation of their constituents, being the formulation with addition of 50 mL of water selected.

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