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PRODUCTION OF FREEZE-DRIED MORINGA EXTRACT

^{1,*}Semirames do Nascimento Silva, ²Francisco de Assis Cardoso Almeida, ²Josivanda Palmeira Gomes, ¹Antônio Jackson Ribeiro Barroso and ³Luzia Márcia de Melo Silva

¹Doctor Student, Federal University of Campina Grande - Campus of Campina Grande, Brazil ²Teachers D.Sc. Federal University of Campina Grande - Campus of Campina Grande, Brazil ³Teacher, Federal Institute of Alagoas, Alagoas, Brazil

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ABSTRACT

Lyophilization is a preservation technique that preserves the characteristics of the product in a particular way, a fact that is not always the case in other techniques. The objective was to produce the lyophilized powdered extract of moringa seeds and to determine their physical and physicochemical characteristics. The work was conducted at the Laboratory of Storage and Processing of Agricultural Products, Federal University of Campina Grande, Paraíba. The experimental design was completely randomized, composed of four treatments and three replicates; using a Liotop® L101 benchtop freeze drier, in which the apparent and actual density, porosity, compacted density, compressibility index, Hausner factor, solubility, color, water content and activity, ashes, titratable total acidity, pH, proteins, lipids and carbohydrates. The results showed, for the dilution of 50 mL as the best, because it presented higher yield, real density, porosity, lower compressibility index, Hausner factor, water, higher amounts of proteins, lipids and lower darkening index.

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INTRODUCTION

Moringa oleifera Lam is the most cultivated tropical species of the Moringaceae family, native to northwest India (Bichi 2013); developing better in tropical and subtropical countries (Kumar et al., 2014, Leone et al., 2015). Unlike other vegetables, it is a perennial species of fast growth. Tolerant to severe climatic conditions such as high temperatures and light frosts, grown in several areas of the world (Gupta et al., 2012). In Brazil the moringa is found in greater numbers in the Northeast region, mainly in the states of Maranhão, Piauí and Ceará (Cysne 2006). Moringa pods, leaves, flowers and seeds can be used in the pharmaceutical, cosmetic and food industries, as well as in the production of lubricants and biofuels (Lilliehöök 2005). In Brazil, programs for the cultivation and use of moringa have been carried out due to their nutritional potential (Ferreira 2008) and several authors, such as: Madrona et al. (2011), Pereira et al. (2011),

*Corresponding author: Semirames do Nascimento Silva,

Doctor Student, Federal University of Campina Grande - Campus of Campina Grande, Brazil.

Bergamasco et al. (2011) have studied its use in the purification and clarification of surface waters. For the better use of moringa seeds, it is necessary to develop technologies for the preservation of their constituents, especially when used in water treatment, since the active principle of the proteins (lectins) responsible for the coagulation / flocculation of the water loses its effect in a short time. In this aspect, drying has been used because it presents greater chemical stability, due to the interruption of metabolic processes that occur even after the material is collected (Pimentel et al., 2008). Among the drying processes, lyophilization stands out for maintaining the constituents of the material, preserving also the sensorial and nutritional characteristics, have a longer shelf life when properly packed, depending on the material, it is possible to stay at room temperature (Vieira et al., 2012). Lyophilized materials are products with high added value by retaining a large part of their original constituents, since it uses low temperatures in their processing. Drying by freeze drying is efficient compared to other drving media, due to characteristics such as product contraction, loss of volatiles, thermal decomposition, enzymatic actions and denaturation of



proteins (Garcia 2009). Based on the above, the objective was to produce the lyophilized powder extract of moringa seeds and to determine their physical and physicochemical characteristics.

MATERIALS AND METHODS

The work was conducted at the Laboratory of Storage and Processing of Agricultural Products (LAPPA), Federal University of Campina Grande (UFCG), Campina Grande, Paraíba, from July to August 2017. The moringa seeds used in the research belong to the cultivation of the UFCG, Campus of Cajazeiras. In the LAPPA, these were selected, peeled and macerated manually with the aid of mortar and pistil. Lyophilization was done in a Liotop® L101 benchtop freeze dryer. The experimental design was completely randomized, with four treatments and three replicates, composed of the addition of 20, 30, 40 and 50 mL of distilled water to form the pulp to be lyophilized. The pulps were plastic shaped and subjected to freezing in a freezer at -18 °C for 24 h. Then, the frozen samples were lyophilized at -50 °C for 25 h (Santos 2016). After dehydration the samples were disintegrated and the extract yield was obtained following Rodrigues et al. (2011), where: Yield% = Mass of extract / Initial mass of sample x 100. The physicochemical analyzes determined were apparent density, according to the method adapted from Caparino et al. (2012), in which 3 g of the powdered extract were weighed in a 10 mL graduated cylinder, without compaction, to determine the total volume occupied by the solid; for the actual density, 1 g of the powder was weighed into a 10 mL graduated beaker, completing the volume of the beaker with oil, determining the amount of oil needed to complete the 10 mL beaker; the porosity was determined by the method of Krokida and Maroulis (1997); the determination of the compacted density was made from the mass contained in a 10 mL beaker after being manually tapped 50 times on the surface of a bench according to Tonon et al. (2013); the compressibility index was determined by comparing the apparent density with the compacted density of the powder; the Hausner factor was determined from the apparent density and compaction (Hausner 1967); the solubility was determined according to the methodology adapted from Durigon (2016); the following determinations were performed according to the methodology proposed by Brasil (2008): the water content was determined by drying the samples in an oven at 105 °C; the ashes were obtained by incineration of the samples in muffle; the titratable total acidity was obtained by titra- metry and the pH by direct reading of the homogenized samples in digital pH meter; the total protein content was quantified by the Micro-Kjeldahl method, which consists in the determination of the total nitrogen; water activity was determined by direct reading on the "Aqua-Lab" equipment; the quantity of lipids was obtained by the modified method of Bligh and Dyer (1959); carbohydrates were quantified by taking the sum of water, lipids, proteins and ashes up to 100; already the color was obtained in colorimeter, according to methodology adapted from Palou et al. (1999) where the parameters L*, a* and b* were obtained. With the data of a* and b* the Chroma (C*) were calculated, and with the parameters L*, a* and b* the darkening index was obtained.

The results were submitted to analysis of variance and the means were compared by the Tukey test at 5% probability, using the statistical software Assistat 7.7 (Silva and Azevedo 2016).

RESULTS AND DISCUSSION

Table 1 shows the data obtained for yield, physical characteristics and color parameters of the freeze-dried powder of moringa seeds, in which a higher yield for the 50 mL dilution was observed, followed by the 40 mL dilution, which were statistically higher than the yield of the 20 and 30 mL dilutions that matched. These results are partially supported by Ribeiro (2014) in a lyophilization drying process with acerola powder, in which Ribeiro obtained yields in the range of 92.1 to 97.4%.

Table 1. Mean values of yield, physical characteristics and color parameters of the freeze-dried powdered extract of moringa seeds

| | | Concentrations | | |
|---|--------|----------------|--------|--------|
| Characteristics | 20 mL | 30 mL | 40 mL | 50 mL |
| Yield (%) | 90.04c | 90.29c | 91.16b | 99.02a |
| Apparently density (g/cm ³) | 0.34a | 0.32a | 0.31a | 0.30a |
| Actual density (g/cm ³) | 0.42a | 0.40a | 0.46a | 0.49a |
| Porosity (%) | 17.81a | 18.29a | 32.80b | 37.40c |
| Compressed density (g/cm ³) | 0.55a | 0.52b | 0.49c | 0.41d |
| Index of compressibility (%) | 37.19a | 37.44a | 37.15a | 25,40b |
| Factor of Hausner | 1.59a | 1.61a | 1.50a | 1.34a |
| Solubility (%) | 75.43a | 74.65a | 76.21a | 73.89a |
| L* | 73,14c | 75,56a | 75,36a | 75,00b |
| Chroma, C* | 19,56 | 18,85 | 17,94 | 17,02 |
| Dimming index | 11,66 | 10,64 | 10,06 | 9,47 |

The averages in the row followed by the same letter do not differ statistically from each other by the Tukey test at 5% probability.

For the physical characteristics of apparent density and real density, statistical equality is observed for the studied dilutions with higher (absolute) values for the actual density, due to the fact that the apparent density takes into account the intergranular voids existing in the mass of the powder. There was also a tendency to reduce bulk density with increasing dilution. According to Ceballos et al. (2012), the density is one of the factors that interferes in the wettability of the powder, an important characteristic as it affects the first phase of the reconstitution of a powder product. For the porosity data, the highest values were obtained with the dilution of the powder in 50 mL of distilled water, followed by the dilution of 40 mL that supplanted the dilutions of 20 and 30 mL, not statistically deferred. This behavior is due to the amount of voids in the powder, which can also be associated with the resistance that the product layer offers to the air movement, being used in the choice of packaging and storage. There was a reduction in the compacted density as the amount of distilled water in the dilutions increased. The compressibility indexes obtained were below the values established in the classification of (Wells 2005), where values <10% indicate excellent flow; 11 to 15% show good flow; 16 to 20% weak flow; 21 to 31% poor flow; and >32% very poor flow, where in the present study the lyophilized powder of the moringa presented poor flow for the dilution with 50 mL of distilled water and very poor flow> 32% for the other dilutions; a fact that may be related to the high percentage of lipids in the powder, hindering its fluidity. Different results (15.89 to 25.02%) were obtained by Caliskan and Dirim (2016) when studying lyophilized powder of Rhus coriaria. For the Hausner factor, the powder at the different dilutions showed intermediate cohesiveness for the dilution with 50 mL of distilled water and high cohesiveness for the other dilutions, according to Wells's classification (2005), where values less than 1.2 are classified as low cohesiveness; between 1.2 and 1.4 are of intermediate cohesiveness; and >1.4

are considered highly cohesive. The post-lyophilates of the moringa showed high solubility in water. A promising result for the proposition of the research to play a fundamental role in the dissolution of substances, especially organic compounds and are in harmony as those obtained by similar Liaotrakoon *et al.* (2012), who worked with lyophilization dried dehydrated red pita. This attribute is desirable for powdered products since, in general, they are easily rehydratable because of their porous structure.

As shown in the table, there was a difference between the dilutions for the luminosity (L*), however the lyophilized powder of the moringa revealed a light color, close to white, with a luminosity greater than 73. This result is important for the industry, since which is a parameter of quality capable of influencing the acceptance of products by consumers. By Chroma, the color intensity is low, indicating that the higher its value, the greater the perceived color intensity (Pathare *et al.*, 2013). The darkening index of the powder was low, indicating no occurrence of enzymatic or non-enzymatic browning. The color of the powder can be affected by numerous variables such as: genotype, grinding process, storage, among others. Table 2 shows the data obtained from the physicochemical analyzes of the freeze-dried powdered extract of moringa seeds.

 Table 2. Mean values of the physico-chemical characteristics of the freeze-dried powdered extract of moringa seeds

| Characteristics | | Concentrations | | |
|----------------------------------|---------|----------------|--------|--------|
| Characteristics | 20 mL | 30 mL | 40 mL | 50 mL |
| Water content (%) | 1.74a | 1.82a | 1.68a | 1.76a |
| Water activity (A _w) | 0.52a | 0.53a | 0.51a | 0.51a |
| Ashes (%) | 2.90bc | 2.82c | 3.03ab | 3.19a |
| Acidity (%) | 0.46a | 0.51a | 0.61a | 0.61a |
| pH | 5.93a | 5.89a | 5.40a | 5.34a |
| Proteins (%) | 33.97 a | 33.80a | 34.23a | 34.31a |
| Lipids (%) | 27.33 a | 27.14a | 29.64a | 36.94b |
| Carbohydrates (%) | 34.06a | 34.20a | 31.83a | 23.80b |

The averages in the row followed by the same letter do not differ statistically from each other by the Tukey test at 5% probability.

The powder had values lower than 2% for water content, these values are in accordance with current legislation that stipulates a maximum of 5% (Brasil 2005). In lyophilized products, the final moisture content varies between 1 and 5%, which makes it possible to preserve the products for a longer period of time, but since it is properly stored, stored and handled. The shelf life of freeze-dried foods can reach up to one year at room temperature (Theodorovski et al., 2014). According to Oliveira (2012), the values of water activity vary from 0 to 1, and are classified as a function of water activity in low humidity (Aw up to 0.6); (A_w between 0.6 and 0.9) and high A_w with values above 0.9. The values of water activity did not statistically defer with the increase of the dilution, but showed a tendency of reduction with the increase of these, being that the powder presented low water activity, which can make impossible the growth of microorganisms and the possible occurrence of chemical reactions and enzymatic. Lower values $(A_w 0.414)$ were obtained by Souza (2011) for the lyophilized powder of the cupuaçu pulp. The ash presented a distinct behavior, with the lowest percentage being the 30 mL dilution. This result, as well as those of the other dilutions studied in the present study, are lower (4.02) than that found by Passos et al. (2012) for non-lyophilized moringa. However, these same authors

obtained a much higher value (25.44%) than the one found in the present study for acidity. This result is probably due to the product in natura versus the lyophilized product. Despite the low value presented, the powder under study is within the Identity and Quality Standard that establishes a minimum acidity of 0.90% (Brasil 2000). Low pH values were observed, thus being acidic. Different result (4.07) was found by Passos et al. (2012) in work done with drying in a greenhouse of moringa seeds. In powders, low pH values are desirable, as they favor the preservation of products due to inhibition of the development of microorganisms such as yeast. According to Machado and Carneiro (2000), the moringa seeds are rich in proteins (33.9%) and lipids (37.2%), a fact proven in this work when lyophilizing the powder obtained from the seeds. The values found in this work and those of Machado and Carneiro (2000) differ from those obtained by Passos et al. (2012), 23.29 and 17.37%, respectively, due to the drying method, since these latter authors worked with drying in an oven. There was a reduction in the amount of carbohydrate for the dilution of 50 mL in comparison to the other dilutions that were statistically equal, yet the powder showed adequate carbohydrate content as defined in the food legislation (Brasil 2005). Higher values (42.53%) were found in lyophilized açaí pulp by Menezes et al. (2008). This work is one of the pioneers in the production, study and physical and physicalchemical characterization of the lyophilized powder of moringa seeds. For this reason there are still not many sources of comparison in these aspects.

Conclusion

The dilution with 50 mL of distilled water showed the best results for lyophilized powder yield (99.02%); actual density (0.49 g / cm 3); porosity (37.40%); compacted density (0.41 g / cm 3); compressibility index (25.40%); Hausner's factor (1.34); pH (5.34); proteins (34.34%); lipids (36.94%); and darkening index (9.47). The results obtained from physico-chemical analyzes of water content, acidity, pH, proteins, lipids and carbohydrates are within the range of what is established by the Brazilian legislation for food.

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