Preservation of the *Moringa oleifera* Constituents by Freeze-Drying

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ABSTRACT

Aims: *Moringa oleifera* is an edible plant. A wide variety of nutritional and medicinal virtues have been attributed to its roots, barks, leaves, flowers, fruits and seeds. The objective of this research was to evaluate the preservation of the constituents of the powder obtained from the moringa seeds by freeze-drying comparing it with the *in natura* (natural extract).
Place and Duration of Study: The work was conducted at the Laboratory of Processing and Storage of Agricultural Products, Department of Agricultural Engineering, Federal University of Campina Grande, Brazil, in the period from August to November 2018.
Methodology: The seeds were peeled and macerated manually. Freeze-drying was done in a Liotop® L101 benchtop freeze drier. After dehydration the samples were disintegrated and the physical and physico-chemical constituents were evaluated before and after freeze-drying in terms of apparent density, real density, porosity, compacted density, compressibility index, Hausner factor, solubility, moisture content and activity, ash, titratable total acidity, pH, protein, lipids and carbohydrates.
Results: The *in natura* powder presented better results for the physical analyzes of the densities: apparent, real and compacted, however, it was observed that for the other physical parameters and physicochemical constituents the freeze-drying promoted the preservation of these in front of the *in natura*. Freeze-drying caused a significant reduction in moisture content, pH and lipid activity, making the powders more stable and contributing to the maintenance of their physico-chemical qualities. The inverse was observed for the ash, protein and carbohydrate contents, where freeze-drying promoted increases in their contents.
Conclusion: Freeze-drying presents as an appropriate method in the preservation of moringa constituents, with emphasis on physicochemical.

Keywords: Drying, Moringa, Plant, Seeds

1. INTRODUCTION

Moringa was introduced in Brazil as an ornamental plant around 1950 and since then has been widely cultivated because of its high food value, mainly leaf and seed, and because of its high medicinal value [1]. In Brazil, in some regions, it is used as an alternative solution to clarify water supply in rural communities [2]. The efficiency of the use of moringa as a natural coagulant in water treatment is reported by several researchers, among them Baptista *et al.* [3].

In this context, the development of new technologies has been pointed to the use and preservation of the constituents of the moringa. Drying technologies are used to meet industry needs by significantly reducing the costs of operations such as packaging, transportation, storage, and providing the consumer with a quality product for a longer period of time [4]. Among these technologies, freeze-drying, due to its low temperature and absence of atmospheric air, preserves the constituents of the natural product, also allowing the chemical, nutritional and sensory properties of the powder to be practically unchanged, besides having characteristics that hinder the development of microorganisms that could promote its deterioration [5].

According to Park *et al.* [6] the type of drying to be used depends, among other factors, on the product to be dehydrated, its chemical constitution and the physical characteristics of the desired product. Freeze-drying has been used and recommended to dry products with high added value, which have delicate aromas or textures or that are sensitive to the use of heat. There is no research on the preservation of moringa powder constituents after freeze-drying and its use in water treatment. For this reason, the objective of this research was to evaluate the preservation of the constituents of the powder obtained from the moringa seeds by freeze-drying comparing it with the *in natura*, since the moringa drying process should be ensured not to exceed 60 °C avoiding that the protein content is damaged [7].

2. MATERIAL AND METHODS

2.1 Place of research

The work was developed at the Laboratory of Processing and Storage of Agricultural Products, Federal University of Campina Grande, Campina Grande, Brazil. To obtain the samples, the seeds were peeled and macerated manually with the aid of mortar and pistil. Freeze-drying was done in a Liotop® L101 benchtop freeze drier. The pulps, to be lyophilized, were obtained by the addition of 50 ml of distilled water to 100 g of the *in natura* powder. Then they were inserted in plastic forms and subjected to freezing in a freezer at -18 °C for 24 hours. Then, the frozen samples were lyophilized at -50 °C for 25 hours [8]. After dehydration, they were disintegrated and the physical and physicochemical constituents of the powders were evaluated *in natura* and lyophilized.

2.2 Physical analyzes

Density apparent - was calculated according to the adapted method of Caparino *et al.* [9]; actual density - this was obtained by weighing 1 g of the powder in a 10 mL graduated cylinder, completing the volume of the beaker with oil, determining the amount of oil needed to complete the 10 mL beaker; porosity - was determined by the method of Krokida and Maroulis [10]; compacted density - was determined 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.* [11]; compressibility index (CI) - was obtained by comparing the apparent density and the compacted density of the powder; Hausner factor - was determined from apparent and compacted density [12]; Solubility - determined according to the methodology adapted from [13]; hygroscopicity - was obtained following the methodology proposed by Goula and Adamopoulos [14]; moisture content - determined by the oven drying method at 105 °C for

24 hours [15]; water activity - obtained through direct reading on the "Aqua-Lab" equipment, Decagon brand, model 3TE.

2.3 Physicochemical analysis

The physico-chemical determinations, except for lipids, were performed according to the methodology of Brazil [15]: Ash - by incineration of samples in muffle at 550 °C for 24 hours; total acidity - given by titration; pH - was obtained by direct reading of the samples in digital pH meter; total protein content - determined by the Micro-Kjeldahl method; lipids - by the modified method of Bligh and Dyer [16]; carbohydrates - removing from 100 the sum of water, lipids, proteins and ashes.

2.4 Statistical analysis

The results were submitted to analysis of variance (ANOVA) and the means comparison was performed by the Tukey test at 5%, using the statistical program Assistat 7.7 [17].

3. RESULTS AND DISCUSSION

Table 1 shows the results obtained for the physical and physicochemical analyzes of the constituents of the *in natura* and lyophilized moringa. It was observed that the physical constituents of the moringa powder, with the exception of solubility and hygroscopicity, presented a statistical difference, and the apparent, real and compacted densities showed higher values for the *in natura* samples. Behavior that is due to the fact that the powder *in natura* present greater and apparent density, due to the greater compaction of its particles. On the other hand, it was verified that the lyophilized powder is more porous and tends to present lower densities, because of the smaller pores. The powders had different values of density than those obtained by Zea *et al.* [18] for guava powder and lyophilized guava and pitahie mix (1.474 and 1.503 g/cm^{-3}, respectively). According to Ceballos *et al.* [19] the density is one of the factors that interferes in the wettability of powders, an important characteristic as it affects the first phase of the reconstitution of a powder product. Thus, because the lyophilized powder is presented with greater porosity than the *in natura*, it may behave differently regarding the resistance and the movement of the air during the drying and storage process. The naturally extracted powder presented better preservation of the physical constituents.

As for the compressibility values, the *in natura* and lyophilized moringa powders fall within the classification of Santhalakshmy *et al.* [20] (20%) to poor (26%), since values between 15 and 20% indicate good fluidity, between 20-35% poor fluidity, between 35-45% poor fluidity and greater than 45% very poor fluidity. As for the Hausner factor, the *in natura* and lyophilized powders had an intermediate-to-easy flow, since materials with a Hausner number greater than 1.4 are classified as cohesive and when less than 1.25 are easily flow able, the *in natura* powder presented the best result because it was easily drained. This property is directly linked to the moisture content of the studied material, that is, the wetter the powder, the greater the cohesiveness, making it more difficult to flow the powder due to the formation of liquid bridges between the particles.

Table 1. Physical and physico-chemical constituents of the moringa powder *in natura* and freeze-dried.

Constitutions of the moringa	Powder		
	In natura	Freeze-dried	CV* (%)
Apparently density (g/cm^{-3})	0.49a	0.30b	1.97
Actual density (g/cm^{-3})	0.65a	0.49b	5.43
Porosity (%)	18.51b	31.34a	3.68
Compressed density (g/cm^{-3})	0.62a	0.41b	1.12
Compressibility Index (%)	20b	26a	4.74
Hausner factor	1.27b	1.34a	1.40
Solubility (%)	74.03a	73.89a	3.13
Hygroscopicity (%)	97.26a	95.08a	1.80
Moisture content (%)	5.24a	1.76b	0.96
Water activity (a_w)	0.62a	0.51b	0.94
Ashes (%)	2.77b	3.19a	0.12
Total acidity (%)	0.22b	0.61a	2.29
pH	6.68a	5.34b	0.88
Proteins (%)	31.92b	34.31a	1.35
Lipids (%)	31.46a	27.33b	3.77
Carbohydrates (%)	28.59b	34.06a	4.33

*Note: The averages in the row followed by the same letter do not differ statistically from each other by the Tukey test at 5% probability.*CV: Coefficient of Variation.*

Both powders (*in natura* and freeze-dried) showed similar solubility values. The rehydration capacity of dry products is of fundamental importance to characterize the quality of products that will be reconstituted, so that the absorption must be fast and in the largest possible volume in order to increase the yield of the products [21]. The powders also presented low hygroscopicity, being defined as the ability of the powder to absorb water from an environment of relative humidity higher than equilibrium. Accordingly, depending on the use of the powders, the high hygroscopicity of the powders is difficult to use the product due to the high affinity for the water and due to its complex composition. High hygroscopicity is undesirable for a powdered product when used in food production in order to promote sticky appearance and hinders solubility of the product, which impairs the quality of the product as a whole; the moringa has antimicrobial, antibacterial and antifungal action. According to Tonon [11], a higher hygroscopicity can be observed in powders with lower humidity, due to the difference of the water concentration gradient between the product and the environment, however this behavior was not verified in the obtained results, which show a higher hygroscopicity in the powder *in natura* whose humidity value was higher.

The lyophilized powder had low content and water activity, and it may be possible to prolong the shelf life of the lyophilized powder by inhibiting the growth of microorganisms and enzymatic activity, without exposing them to high temperatures and, as a result, greater preservation of nutritional quality and sensory characteristics [22]. The powder *in natura* also presented low moisture content, thus, both powders presented values within those required by current legislation, RDC nº 270 - ANVISA, which describes the maximum acceptance limit of 15% [23]. As for the water activity, the powder *in natura* showed intermediate water activity, which may hinder the growth of fungi and bacteria.]. As for the water activity, the powder *in natura* showed intermediatewater activity.

Regarding the analyzes of the physicochemical constituents of the powders, it was observed that there was difference for all parameters evaluated. The ash content in the lyophilized samples was higher than in the *in natura* samples. In a study carried out by Passos *et al.* [24] with the *in natura* moringa powder, the authors found 0.95% of ash, much lower than that found in this research. The powders presented low acidity, meeting the requirements of Brazilian legislation, which determines a minimum of 0.8% acidity in citric acid [23]. In a study on pulps marketed in Alagoas, Temóteo *et al.* [25] observed acidity of 0.94% in citric acid for lyophilized acerola pulp powder, values that are in accordance with current legislation and are superior to that found in this study for Moringa oleifera powder. Different results (3.18%) were also found by Oliveira [26] when the cassava pulp was dried by freeze-drying.

For the pH it was found that the lyophilized powder was acidic. Passos *et al.* [24], working with the *in natura* powder of moringa seeds, a much higher value (7.47%) for pH. Considering the possible toxic effects of microorganisms, when they are at an unfavorable pH, it can be verified that the acid pH value verified in this research is beneficial to lyophilized powder, since it promotes a longer shelf life for the same, without prejudice to its stability.

There was an increase in the ash, protein and carbohydrate content of the lyophilized powder and a reduction in the amount of lipids; this is probably due to the addition of distilled water to form the pulp to be lyophilized. Passos *et al.* [24] found lower values for proteins (23.29%) and lipids (17.37%) for the *in natura* powder of moringa seeds. Basso [27] verified in his study about the chemical composition of the jackfruit that the freeze-drying process did not reduce the amount of ashes, proteins and lipids. Celestino [28] cites as advantages of freeze drying the concentration of nutritional components, increasing their value in the product. Affirmative in part is in agreement with Ghribi *et al.* [29], Oberoi and Sogi [30] and Samoticha *et al.* [31], who proved in their research the efficiency of the process of freeze-drying against the preservation of its constituents. Solubility and hygroscopicity may affect the coagulation of the powder in the water.

4. CONCLUSION

For the physical analysis of the moringa seed powder (apparent density, real and compacted), it was verified that the *in natura* powder presented better results, however, it was observed that for the other physicochemical constituents the freeze-drying promoted the preservation these in front of the *in natura*.

Freeze-drying of the moringa powder caused a significant reduction in moisture content, water activity, pH and lipids. The inverse was observed for the contents of ashes, proteins and carbohydrates, where freeze-drying promoted increases in their contents when compared to *in natura*. Freeze-drying presented to the moringa an adequate method in the preservation of its constituents, with emphasis on the physical-chemical production.

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COMPETING INTERESTS

Authors have declared that no competing interests exist.

AUTHORS' CONTRIBUTIONS

Semirames Authors of N. Silva, Francisco AC Almeida and Josivanda P. Gomes elaborated and conducted the research, the last two are advisors of the doctorate of Semirames of N. Silva, the author Newton C. Santos and Sâmela L. Barros performed the statistical analysis, the authors Raphael LJ Almeida, Victor HA Ribeiro and Virgínia MA Ribeiro wrote the bibliographic review of the research. All authors read and approved the final manuscript.

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