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ORIGINAL ARTICLES

Physicochemical characterisation and bioactive potential of microcapsules of *Moringa oleifera* leaf extract

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Abstract

In this work, moringa leaf extract was encapsulated in gum arabic by lyophilisation, using different extract:gum arabic ratios (1:10, 1:6 and 1:4). The physicochemical characteristics, total phenolic content and antioxidant potential of the microcapsules were evaluated at time 0 and after 10 months of storage at 25 °C. The moisture content, hygroscopicity and solubility of the microcapsules decreased, and the water activity increased after storage. The total phenolic content increased from 28 to 36%, with the highest increase in the microcapsule formulation with less gum arabic. The antioxidant activity also increased significantly, to values between 140 and 307%, with activity being higher in the 1:4 (extract: gum arabic) ratio microcapsules. The microcapsules of moringa leaf extract encapsulated with gum arabic showed good antioxidant potential *in vitro*, especially after storage, and could be a promising alternative for the future applications in the food, pharmaceutical and cosmetic sectors as additives or preservatives.

Keywords: *moringa leaf, bioactive compounds, microcapsules, plant, gum arabic.*

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1. Introduction

Moringa oleifera Lam. (commonly known as 'moringa') constitutes one of thirteen species of *Moringa* belonging to the monogenic family *Moringaceae*, a plant native to northern India and, more specifically, the Himalayan mountains. Several studies have reported *M. oleifera* as a medicinal plant due to the presence of bioactive compounds such as gallic acid, caffeic acid, vanillic acid, ferulic acid, apigenin, and others[1,2]. However, these bioactive compounds are generally unstable and susceptible to oxidative degradation, especially when in contact with moisture, light, heat, and oxygen, thus limiting their bioavailability[3]. This limitation can be reduced by using encapsulation techniques such as lyophilisation, also known as freeze-drying, which consists of three steps: freezing, primary drying (sublimation) and secondary drying (desorption). It is widely used to preserve heat-sensitive substances and improve microcapsule stability and encapsulation efficiency (EE)[3].

An important aspect of the application of microcapsules is their stability over long periods of storage. Dadi et al.[4] evaluated the stability of maltodextrin microcapsules with *Moringa stenopetala* leaf extract obtained by freeze-drying in terms of phenolic compounds content after storage for 60 days at 4 °C, 25 °C and 45 °C; George et al.[5] also evaluated the stability of microcapsules of *M. oleifera* extracts, stored at 4 °C, 25 °C and 45 °C for 4 weeks through the loss in the total phenolic content (TPC) weekly; and Vonghirundecha et al.[6] evaluated the storage stability of maltodextrin microcapsules with *M. oleifera* leaf extract (obtained by a spray-drying technique) at 4 °C and 30 °C for 3 months, by measuring the TPC and ABTS free radical-scavenging effects every 15 days. To the best of our knowledge, stability studies of moringa extract microcapsules have been carried out for a maximum of 3 months, and only the bioactive compound content has been evaluated. Considering that parameters such as moisture, water activity, solubility, and hygroscopicity also need to be evaluated after prolonged storage, the present study aimed to determine the total phenolic content, antioxidant activity and physicochemical characteristics of different *M. oleifera* leaf extract microcapsule formulations after 10 months of storage at 25 °C.

2. Materials and Methods

2.1 Materials

M. oleifera leaves were collected from trees located at the Federal University of Sergipe, São Cristóvão, Sergipe (10°55'24.1"S 37°05'58.5"W) in January 2021. Gum arabic and ethanol were purchased from Perfyl Tech (São Bernardo dos Campos, São Paulo, Brazil); 6-hydroxy-2,5,7,8-tetramethyl chrome-2-carboxylic acid (Trolox), 2,2-diphenyl-1-picrylhydrazyl radical (DPPH)

and 2,2'-azino-bis acid (3-ethylbenzthiazoline) 6-sulfonic acid (ABTS) were purchased from Sigma-Aldrich (Saint Louis, MO, USA). Sodium acetate was obtained from Neon (São Paulo, São Paulo, Brazil). The Folin-Ciocalteu reagent, sodium carbonate, potassium persulphate and acetic acid were obtained from Dinâmica (Indaiatuba, São Paulo, Brazil).

2.2 Preparation of extract from moringa leaves

The preparation of the extract was in accordance with the methodology of Feitosa et al.[7]. The moringa leaves were dried at 50 °C ± 2 °C in an oven with air circulation for 24 h and then crushed in a blender until a uniform powder was obtained. The extract was obtained using 80% ethanol at a leaf flour-to-solvent ratio of 1:5 (m/v). The solution was kept under orbital stirring at 200 rpm for 1 h at 30 °C. The extract was then filtered through filter paper and the supernatant was evaporated in a rotary evaporator at 40 °C and 50 rpm. This extract was previously analysed by Feitosa et al.[7]; its composition of polyphenolic compounds is as follows: 0.849 mg of gallic acid, 0.237 mg of epicatechin gallate, 0.384 mg of catechin, 0.412 mg of benzoic acid, 0.054 mg of 2,5–dihydroxybenzoic acid and 0.019 mg of coumarin per gram of dry powder.

2.3 Encapsulation of moringa leaf extract

A 10% (w/v) solution of gum arabic in distilled water was prepared and agitated in an orbital shaker (SOLAB/SL222) at 250 rpm for 30 min at 35 °C. The moringa flour extract (211 mg/mL) was then added in the proportions 1:10, 1:6, and 1:4 (g extract per mL of gum arabic solution). The solutions were then homogenised in a shaker at 25 rpm for 30 min at 35 °C. A solution containing only 10% gum arabic was used as a control[8]. For the encapsulation, the solutions were first frozen at -18 °C for 48 h and then placed in a freeze-dryer (Christ Alpha 1–2 LD Plus, Brand Christ, Germany) and dried for 48 h at -55 °C, 0.021 mbar pressure and a vacuum of 0.42 mbar[9]. The samples were stored in a desiccator at 25 °C for a period of 10 months. The total phenolic content, moisture, water activity, hygroscopicity, and solubility of the microcapsules were determined at time 0 and after 10 months of storage. The microcapsules were named 1E:10GA, 1E:6GA and 1E:4GA.

2.4 Morphological analysis

The morphological analysis of the microcapsules was performed in a scanning electron microscope (Model TM3000, Hitachi, Japan). The samples were covered with gold and analysed at an accelerating voltage of 5 kV and magnifications of 100x and 1000x.

2.5 Moisture and water activity

The moisture content of the microcapsules (1 g) was determined by drying in an oven at 105 °C and directly reading the water activity using an electronic meter (Aqualab Dew Point 4TEV)[9]. All analyses were performed in triplicate.

2.6 Solubility and hygroscopicity

For the solubility analysis, 1 g of microcapsules was mixed with 100 mL of distilled water and stirred in a magnetic stirrer (Tecnal, TE-0851) for 30 min. The solution was then centrifuged at 129 x g for 5 min. Subsequently, a 25 mL aliquot of the supernatant was placed in a petri dish and dried in an oven at 105 °C for 5 h. All analyses were performed in triplicate. The solubility was calculated by weight difference and expressed in percentage[9].

For hygroscopicity, the microcapsules (1 g, W1) were kept in a desiccator containing a saturated sodium chloride solution (75.3% relative humidity) at 25 °C for 1 week. The samples were then weighed (W2), and the hygroscopicity was calculated according to Equation 1[4]. All analyses were performed in triplicate.

$$\text{Hygroscopicity}(\%) = (W2-W1)/W1 \times 100 \quad (1)$$

2.7 Encapsulation efficiency (EE)

To determine the total phenolic content, 100 mg of microcapsules were dissolved in 1 mL of distilled water; 9 mL of ethanol (pure) was then added, and the solution was mixed for 5 min. The solution was then filtered through a 0.22 μm membrane filter. The total phenolic content of the microcapsule wall (the surface phenolic content) was determined by mixing 100 mg of microcapsules with 10 mL of ethanol. The dispersion was vortexed for 10 s and centrifuged at 129 x g for 3 min. The supernatant was filtered through a 0.22 μm membrane filter. The total and surface phenolic contents were quantified as described in the next section, and the EE was then determined using Equation 2[4].

$$EE\ (\%) = (TPC-SPC)/(TPC) \times 100 \quad (2)$$

where TPC is the total phenolic content, and SPC is the surface phenolic content.

2.8 Determination of the total phenolic content

The total phenolic content was determined using Folin-Ciocalteu reagent[10]. Samples of the supernatant (0.5 mL) were mixed with 0.5 mL of 95% ethanol solution, 2.5 mL of distilled water and 0.25 mL of 1 N Folin-Ciocalteu reagent, and the solution was then homogenised. Then, 0.5 mL of 5% (w/v) sodium carbonate solution was added. The samples were kept in a dark room for 60 min and then homogenised. The spectrophotometric absorbance was measured at 725 nm. Ethanol was used as the blank control. All analyses were performed in triplicate, and the values were calculated from the gallic acid calibration curve (0-150 mg/L) ($y = 0.0108x - 0.0185$; $R^2 = 0.9961$). The results were expressed as g of gallic acid equivalents (GAE) per g of microcapsules (GAE/g of microcapsules).

2.9 Antioxidant activity (AA)

The AA of microcapsules was performed using the ABTS, DPPH[11], and FRAP (Ferric Reducing Antioxidant Power)[12] methods. For DPPH, the sample (250 μL) was mixed with DPPH reagent (1.25 mL), and after 5 min, the absorbance was read at 517 nm using a spectrophotometer (Spectrum SP-2000 UV, Tucumán, Argentina). Ethanol was used as the blank control. Different concentrations of Trolox, between

50 and 250 µmol of Trolox mL^{-1}, were used to construct the calibration curve ($y = -0.0037x + 1.6425$; $R^2 = 0.9984$), and the results were expressed in µmol Trolox/g of microcapsules. For ABTS, the sample (30 µL) was mixed with 3.0 mL of the ABTS radical, homogenised in a vortex, and kept at rest for 6 min. The absorbance value was read at 734 nm using a spectrophotometer. Different concentrations of Trolox, varying from 100 to 1,600 µmol Trolox mL^{-1}, were used to obtain the calibration curve ($y = -0.0004x + 0.6082$; $R^2 = 0.9862$). The results were expressed in µmol of Trolox/g of microcapsules. The FRAP assay was performed by mixing 90 µL of sample with 270 µL of distilled water and 2.7 mL of FRAP reagent. This solution was homogenised in a vortex and kept in a water bath at 37 °C for 30 min. The absorbance value was read at 595 nm. Concentrations of Trolox ranging from 100 to 1,200 µmol Trolox mL^{-1} were used to obtain the calibration curve ($y = 0.0016x - 0.019$; $R^2 = 0.9962$) and the results were expressed in µmol Trolox g of microcapsules^{-1}.

2.10 Statistical analysis

All experiments were performed in triplicate, and the results were expressed as mean ± standard deviation.

3. Results and Discussion

3.1 EE and morphology

The EE for the microcapsules produced using extract:gum arabic ratios of 1:4, 1:6 and 1:10 were 95.70%, 94.99%, and 89.75%, respectively. This indicates that lower extract:gum arabic ratio results in higher EE. The high EE may be related to the freeze-drying technique, which uses vacuum and low temperatures, contributing to a reduction in compound degradation[13]. Dadi et al.[4] and Vonghirundecha et al.[6] obtained lower EE values of 71.44% and 91.2% for microcapsules of *M. stenopetala* and *M. oleifera* leaf extracts obtained by freeze-drying and spray-drying, respectively. The microencapsulation efficiency (ME) of the bioactive compounds can be quite varied, since during the sublimation of water in the freeze-drying process, the encapsulated component can be degraded[9].

In relation to morphology, all microcapsules had an irregular, brittle, flake-like appearance, characteristic of microcapsules obtained by the freeze-drying process (Figure 1)[4,5,14,15]. The microcapsules without extract (Figures 1A and 1B) also showed a porous structure, probably due to the ice sublimation process during freeze-drying and a reduced thickness, possibly due to the lack of material in their core[16]. Structures with cracks or fractures, a vitreous appearance and a granule structure were observed in the microcapsules containing extract[14,17,18] (Figures 1C-H). The glassy appearance is associated with the protection of encapsulated compounds against exposure to heat and oxygen[17]. Larger particle sizes were also observed for the microcapsules containing a higher concentration of extract (1E:4GA) (Figures 1G and 1H). Similar structures were also observed in microcapsules of *M. stenopetala* and

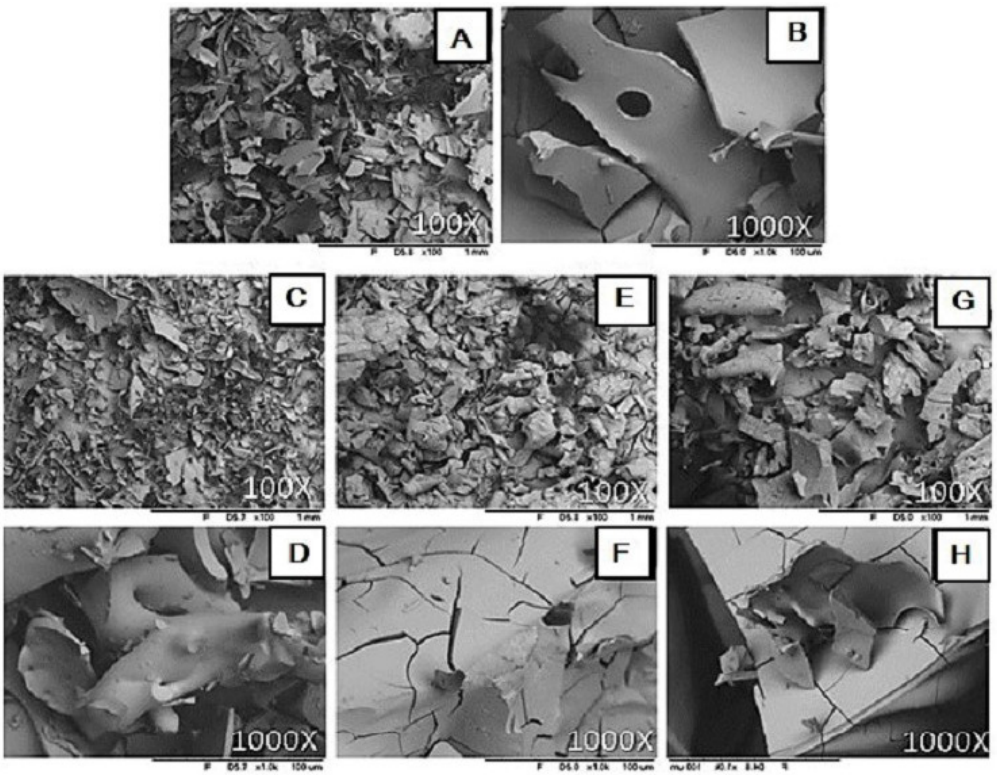

Figure 1. Scanning electron micrographs (SEM) of microcapsules at magnifications of 100× and 1000×. (A, B) Gum arabic microcapsules without extract. Microcapsules with extract:gum arabic proportions (C, D) 1E:10GA, (E, F) 1E:6GA and (G, H) 1E:4GA.

M. oleifera leaf extracts encapsulated with maltodextrin by freeze-drying[4,5].

3.2 Physicochemical properties of the microcapsules

The microcapsules were evaluated for moisture, water activity, hygroscopicity and solubility. At time 0, the moisture content of the microcapsules varied between 3.1% and 3.5%, (Figure 2A). After 10 months of storage, the moisture was reduced to between 1.8% and 2.2% with lower values in the microcapsules containing a smaller amount of gum arabic (1E:6GA and 1E:4GA). This result was probably due to the microcapsules being stored in a desiccator in room temperature, which may have led to the gradual removal of moisture over time. Moisture values below 3% can prevent chemical or microbiological changes in the microcapsules[4]. The results indicated that prolonged storage of these microcapsules does not cause deterioration related to high moisture content.

The water activity values varied between 0.11 and 0.28 at time 0, and it was lower in the 1E:4GA microcapsules (Figure 2A). This result is within the recommended Aw value for atomised products (< 0.30) and guarantees the stability of the microcapsules[19]. Vonghirundecha et al.[6] obtained microcapsules of *M. oleifera* leaf extract in maltodextrin by spray-drying, with Aw values between 0.22 and 0.29, which is higher than the values obtained in the present study. After storage, there was a slight increase in water activity, ranging from 0.33 to 0.39, with a higher increase in the microcapsules with a higher extract concentration (1E:6GA and 1E:4GA). This result indicates the presence of available water, which can cause biochemical reactions, resulting in a shorter shelf life of the microcapsules[20].

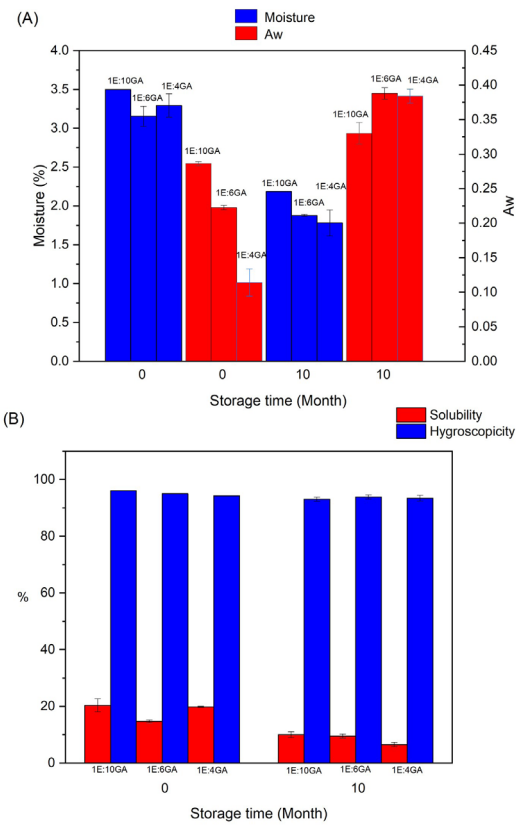

Figure 2. Physicochemical parameters of microcapsule formulations analysed at time 0 and after 10 months of storage. 01E:10GA (1 g of extract and 10 mL of gum arabic solution); 01E:06GA (1 g of extract and 6 mL of gum arabic solution); 01E:04GA (1 g of extract and 4 mL of gum arabic solution).

Another significant quality parameter of microcapsules is hygroscopicity, which indicates the ability to absorb water from the environment. Microcapsules with low hygroscopicity are ideal[4]. The hygroscopicity of the microcapsules varied between 20% and 30% at time 0 and decreased to values between 6.5% and 10.0% after 10 months of storage, representing a reduction of about 78% (Figure 2B). The storage, therefore, improved this property. Vonghirundecha et al.[6] have obtained hygroscopicity values between 6.5% and 8.65% (w/w) in microcapsules of maltodextrin and *M. oleifera* leaf extract obtained by spray-drying; George et al.[5] obtained hygroscopicity values of 14.35% in microcapsules of gum arabic and *M. oleifera* leaf extract obtained by freeze-drying, and Dadi et al.[4] obtained hygroscopicity values of 16.77% and 18.12% for microcapsules of *M. stenopetala* leaf extract obtained by freeze-drying, using maltodextrin and a mixture of maltodextrin and pectin as encapsulants, respectively. These values were close to those obtained in this work.

The higher the aqueous solubility (close to 100%) the better the solubility of these microcapsules, for example, in food systems[9]. The solubility of the microcapsules varied between 94% and 96%, being higher in the microcapsules with a higher amount of gum arabic (1E:10GA). This result can be attributed to the gum arabic, which is known for its high solubility and low viscosity in aqueous media[21,22]. After storage, the solubility of the microcapsule formulations was still good, only decreasing to around 93% (Figure 2B). These results were similar to those obtained by other researchers, such as Dadi et al.[4], who obtained a solubility of 94.65% for microcapsules of *M. stenopetala* leaf extract with maltodextrin obtained by freeze-drying, Vonghirundecha et al.[6], who obtained a solubility between 91.2% and 92.2% for maltodextrin microcapsules with *M. oleifera* leaf extract obtained by spray-drying, and George et al.[5], who obtained values between 86.35% and 98.74% for microcapsules of gum arabic, maltodextrin, and moringa leaf extract obtained by freeze-drying.

3.3 Total phenolic content and AA of the microcapsules

The total phenolic content of the microcapsules was determined at time 0 and after 10 months of storage at 25 °C (Figure 3A). The lower the amount of gum arabic used in the microcapsules (1E:4GA), the greater the total phenolic content due to the higher concentration of extract. There was an increase in total phenolic content of 28% and 36% in the 1E:6GA and 1E:4GA microcapsule formulations, respectively, after storage. This result may be due to the higher concentration of compounds resulting from the lower moisture content obtained in the microcapsules after storage.

The results obtained in this work were better than those obtained by other researchers. Vonghirundecha et al.[6] observed no significant change in phenolic content during 90 days

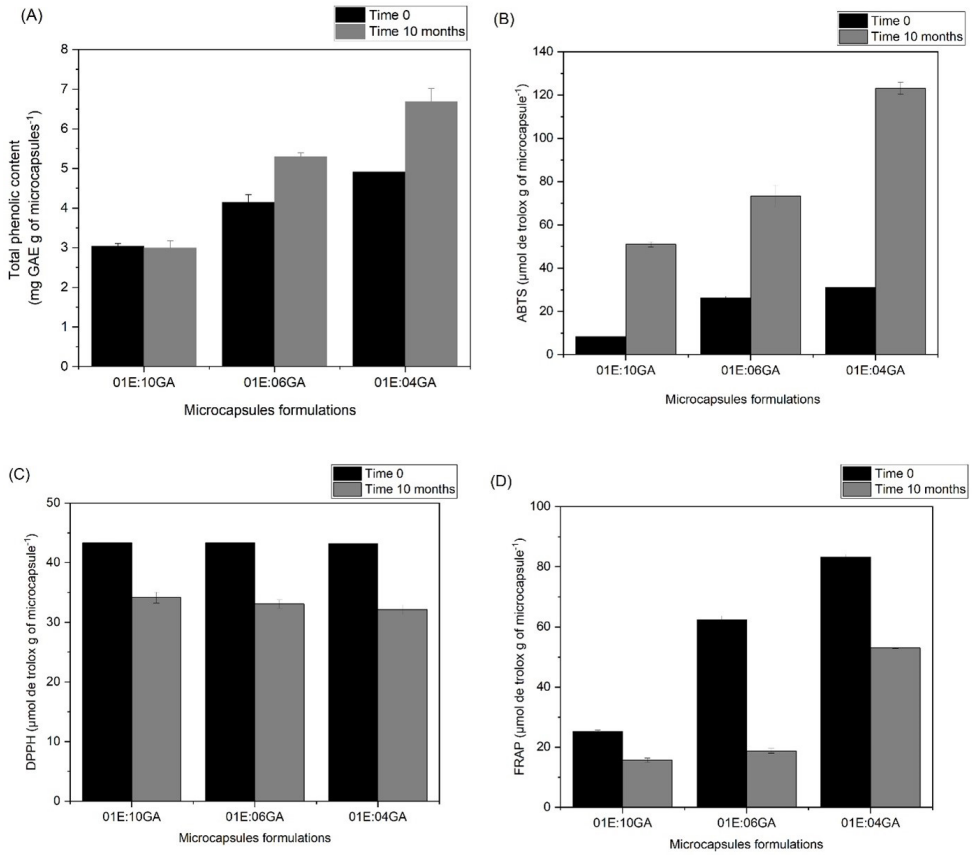

Figure 3. Total phenolic content and AA of microcapsule formulations analysed at time 0 and after 10 months of storage. 01E:10GA (1 g of extract and 10 mL of gum arabic solution); 01E:06GA (1 g of extract and 6 mL of gum arabic solution); 01E:04GA (1 g of extract and 4 mL of gum arabic solution).

of storage of *M. oleifera* leaf extract microcapsules at 4 °C and 37 °C in the dark in UV-protective airtight containers, and George et al.[5] obtained a stable total phenolic content in *M. oleifera* leaf microcapsules during storage at 4 °C for 28 days. On the other hand, Dadi et al.[4] found a decrease in the total phenolic content of *M. stenopetala* microcapsules after 60 days of storage at 4 °C, 25 °C and 45 °C.

With regards to the AA using the ABTS method (Figure 3B), there was a significant increase (between 140% and 307%) after 10 months of storage, being greater (123 µmol of Trolox/g of microcapsules) in the microcapsules with the lowest amount of gum arabic (1E:4GA), which also had a higher total phenolic content. This result suggests the presence of phenolic compounds with the potential to react with the ABTS radical. The AA of the microcapsules may be due to the presence of antioxidant compounds such as gallic acid, epicatechin gallate, catechin and coumarin in the *M. oleifera* leaf extract[7]. Vonghirundecha et al.[6] verified that there was no significant difference in the AA using the ABTS method for *M. oleifera* leaf microcapsules after 3 months of storage in the dark at 4 °C and 37 °C. At time 0, the AA using the DPPH method for the different microcapsules was 43.0 µmol of Trolox/g of microcapsules (Figure 3C). After storage, the values decreased to between 32 and 34 µmol of Trolox/g of microcapsules. For the FRAP method, the AA varied between 25.0 and 83.0 µmol of Trolox/g of microcapsules at time 0 and between 15.7 and 53.0 µmol of Trolox/g of microcapsules after 10 months of storage, being higher for the microcapsule 1E:4GA, indicating the presence of antioxidant compounds in the extract with the ability to reduce iron (Figure 3D). The decrease after storage was probably due to the degradation of antioxidant compounds. It is worth noting that the results may differ between antioxidant methods due to the peculiarities of each assay and the complexity of the extract[23].

4. Conclusions

In this study, the physicochemical characteristics, total phenolic content and AA of different microcapsule formulations containing moringa leaf extract were evaluated before and after storage for 10 months at 25 °C. All microcapsules (at 0 time) had a good EE (89.75 to 95.70%), high solubility (94 to 96%), favourable hygroscopicity (20 to 30%), low moisture (3.1% to 3.5%) and water activity (0.11 to 0.28). After 10 months of storage, the moisture and hygroscopicity decreased (reduction of about 39 and 78%, respectively), the solubility was kept at 94%, and the water activity increased (about 89%) in the microcapsules. Notably, the total phenolic

content and the AA (by the ABTS method) also increased after storage by 28-36% and 140-307%, respectively, with the highest values being observed in the microcapsules with higher concentration of extract. Microcapsules of *M. oleifera* leaf extract showed excellent bioactive potential after storage and could be a promising alternative for future application in the food, cosmetic, or pharmaceutical sectors.

5. Author's Contribution

• **Conceptualization** – Luciana Cristina Lins de Aquino Santana; Rosely de Jesus Nascimento; Paula Ribeiro Buarque.

• **Data curation** – Luciana Cristina Lins de Aquino Santana; Rosely de Jesus Nascimento; Carlos Renato Vieira Nascimento; Paula Ribeiro Buarque.

• **Formal analysis** – Luciana Cristina Lins de Aquino Santana; Rosely de Jesus Nascimento; Paula Ribeiro Buarque; Carlos Renato Vieira Nascimento.

• **Funding acquisition** - Luciana Cristina Lins de Aquino Santana.

• **Investigation** – Rosely de Jesus Nascimento; Carlos Renato Vieira Nascimento.

• **Methodology** – NA.

• **Project administration** – Luciana Cristina Lins de Aquino Santana.

• **Resources** – Luciana Cristina Lins de Aquino Santana.

• **Software** – NA.

• **Supervision** – Luciana Cristina Lins de Aquino Santana; Paula Ribeiro Buarque.

• **Validation** – NA.

• **Visualization** – Luciana Cristina Lins de Aquino Santana.

• **Writing – original draft** – Luciana Cristina Lins de Aquino Santana; Rosely de Jesus Nascimento.

• **Writing – review & editing** – Luciana Cristina Lins de Aquino Santana; Paula Ribeiro Buarque.

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