Influence of shell material on vitamin C content, total phenolic compounds, sorption isotherms and particle size of spray-dried camu-camu juice

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Abstract – Introduction. Camu-camu is a native Amazonian fruit mainly known for its high vitamin C content. Its composition confers high antioxidant capacity on this fruit and makes it a potential source of antioxidant products. The use of spray-drying with the aid of a carrier agent is a technique that has been applied for the preservation of important components of foods and drugs. The objective of our work was to evaluate the influence of those agents used as shell material on the vitamin content and total phenolic compounds of camu-camu powder juice obtained by spray-drying. **Materials and methods**. A commercial frozen camu-camu pulp was the raw material; maltodextrin and gum arabic were the selected carrier agents. Processes were performed in a mini-spray-dryer with inlet and outlet air temperatures of 180 °C and 85 °C, respectively, and a drying air flow rate of 700 $L \cdot h^{-1}$. Laser diffraction was used to determine the particle size distribution of the samples, and sorption isotherms of spray-dried camu-camu were measured using a static gravimetric method. Total phenolic compounds and vitamin C were determined in the raw pulp and in the powders obtained. **Results**. When using gum arabic and maltodextrin as the carrier agents, the moisture results obtained for the spray-dried camu-camu powders were 2.8% and 3.2%, respectively; the process yield was 84% and 72%, respectively. The spray-dried powder produced using gum arabic presented higher contents of vitamin C [(15,363 ± 226) $mg \cdot 100\ g^{-1}$] and phenolic compounds [(6,654 ± 596) mg GAE·100 g^{-1}] than the powder obtained with maltodextrin, respectively (11,258 ± 298) $mg \cdot 100\ g^{-1}$ and (5,912 ± 582) mg GAE·100 g^{-1}. **Conclusions**. The concentration factors for the vitamin C and phenolic compounds in camu-camu powder reveal the effectiveness of spray-drying to preserve the antioxidant capacity of this fruit. Gum arabic was a more effective barrier than maltodextrin for bioactive compound retention.

Brazil / *Myrciaria dubia* / fruits / food processing / spray-drying / microencapsulation / ascorbic acid

Influence du matériau utilisé pour l'enrobage de jus de camu-camu atomisé sur la teneur en vitamine C, les composés phénoliques totaux, les isothermes de sorption et la granulométrie du produit.

Résumé – **Introduction**. Le camu-camu est un fruit originaire d'Amazonie qui est surtout connu du fait de sa haute teneur en vitamine C. Une telle composition confère à ce fruit une capacité antioxydante élevée et en fait une source potentielle de produits antioxydants. L'utilisation d'un séchage de la pulpe par pulvérisation à l'aide d'un agent de support est une technique qui est appliquée à la conservation de composantes importantes d'aliments et de médicaments. L'objectif de nos travaux a été d'évaluer l'influence de ces agents, utilisés comme matériel d'enrobage, sur la teneur en vitamines et les composés phénoliques totaux de jus de camu-camu en poudre obtenu par séchage par atomisation. **Matériel et méthodes**. De la pulpe de camu-camu congelée du commerce a été utilisée comme matière première ; la maltodextrine et la gomme arabique ont été les agents d'enrobage sélectionnés. Les traitements ont été réalisés dans un mini sécheur par pulvérisation avec une température d'air d'admission de 180 °C et de sortie de 85 °C, et un flux d'air séchant de 700 $L \cdot h^{-1}$. La diffraction au laser a été utilisée pour déterminer la distribution granulométrique des échantillons ; les isothermes de sorption du jus de camu-camu atomisé ont été déterminés par une méthode gravimétrique statique. Les composés phénoliques totaux et la vitamine C ont été déterminés dans la pulpe brute et dans les poudres obtenues. **Résultats**. Lors de l'utilisation de la gomme arabique et de la maltodextrine comme agent d'enrobage, l'humidité obtenue pour les poudres de camu-camu séché par pulvérisation a été de 2,8 % et 3,2 %, respectivement ; le rendement du procédé a été de 84 % et 72 %, respectivement. La poudre séchée par pulvérisation produite à l'aide de gomme arabique a présenté des teneurs plus élevées en vitamine C [(15,363 ± 226) $mg \cdot 100\ g^{-1}$] et en composés phénoliques [(6,654 ± 596) mg GAE·100 g^{-1}] que la poudre obtenue avec de la maltodextrine, respectivement (11,258 ± 298) $mg \cdot 100\ g^{-1}$ and (5,912 ± 582) mg GAE·100 g^{-1}. **Conclusions**. Le facteur de concentration de la vitamine C et des composés phénoliques dans la poudre de camu-camu révèle l'efficacité du séchage par atomisation pour préserver la capacité antioxydante de ce fruit. La gomme arabique est une barrière plus efficace que la maltodextrine pour la rétention des composés bioactifs.

Brésil / *Myrciaria dubia* / fruits / traitement des aliments / séchage par pulvérisation / microencapsulation / acide ascorbique

1. Introduction

Camu-camu is a small acid fruit, native to the Amazon, known for its nutritional and functional characteristics due to its high ascorbic acid content and the presence of carotenoids and phenolic compounds [1].

The bioactive compounds and antioxidant capacities of polyphenolic extracts of fresh and dry native non-traditional Brazilian fruits were compared using different methods [2]. The results showed potential perspectives for industrial application of fruits such as camu-camu, acerola and puçá-preto. However, the stability of vitamin C and polyphenols is severely affected by temperature, pH, metal ions, and exposure to light and oxygen.

Chirinos *et al.* showed that at full maturity (red stage) Peruvian camu-camu presented the highest total phenolic content (1,320 mg GAE·100 g^{-1}) [3] in comparison with contents reported for cherry, plum and strawberry, as well as for other berry types, such as blueberry, cranberry and lingonberry [4]. Those authors also verified that at the green stage, camu-camu presented a higher content of vitamin C (2,280 mg·100 g^{-1}) than fully matured fruits (2,010 mg·100 g^{-1}), unlike the results obtained by Mattietto *et al.* [5] when analyzing different progenies and maturity stages of Brazilian camu-camu. They found that the vitamin C content, although presenting a large variation among progenies, showed an average increase with maturation.

Camu-camu antioxidant compounds were fractionated by Chirinos *et al.* into two fractions: an ascorbic acid-rich fraction and a phenolic-rich fraction [3]. According to this study, the ascorbic acid-rich fraction was the major contributor to the antioxidant capacity measured by the DPPH (2, 2-diphenyl-1-picrylhydrazil) method.

Using high-performance liquid chromatography (HPLC), these authors detected a total of 30 different phenolic compounds in camu-camu, such as flavan-3-ols (catechin and its derivatives), ellagic acid derivatives, anthocyanins (delphinidin 3-glucoside and cyanidin 3-glucoside), flavonols (rutin and its derivatives) and flavanones (naringenin and eriodictyol derivatives). These results suggest that camu-camu is an important source of health-promoting phytochemicals.

The inclusion of tropical and native Amazon fruits in functional food formulations has been the focus of research which associates their consumption with the prevention of non-communicable diseases. Particularly, camu-camu (*Myrciaria dubia* (HBK) McVaugh) berries present a very high content of vitamin C and other bioactive components such as carotenoids and phenolic compounds. The study of antioxidative and anti-inflammatory properties of camu-camu in humans carried out by Inoue *et al.* suggests that camu-camu juice is more powerful when compared with vitamin C tablets containing equivalent vitamin C content [6]. Although the mechanism is not yet elucidated, the high level of potassium, considered to accelerate intestinal absorption of vitamin C, can partially explain this result.

In spite of all these characteristics and potential health benefits, camu-camu is still an unknown fruit for most of the Brazilian population, with little commercial cultivation and few products available.

Microencapsulation by spray-drying using a coating material, widely applied by the food industry, is a less costly method to ensure the microbiological stability of products, reduce the core reactivity with environmental factors, mask the unpleasant taste, avoid the risk of chemical and/or biological degradation, and reduce storage and shipping costs [7]. Nowadays, microencapsulation is recommended for the controlled release of bioactive compounds at the right place and time to improve the effectiveness of food additives [8].

Among numerous shell materials, gum arabic and maltodextrin are commonly used as coating agents for food application due to their high solubility in water, low viscosity and low cost. There are several reports on microencapsulation of vitamin C-rich fruits by spray-drying, regarding the formulation of a functional product [7]. However, despite its very high vitamin C content, microencapsulation of camu-camu by spray-drying has few reports in the literature. Thus, the objective of our work was to evaluate the effect of carrier agents on the vitamin C content and total phenolic compounds of spray-dried camu-camu juice. Furthermore, the powders were also characterized with respect to particle size distribution and sorption isotherms as tools for understanding their stability.

2. Materials and methods

Frozen camu-camu pulp was purchased from a commercial fruit pulp supplier in the Northern region of Brazil, and stored at –18 °C prior to processing.

Maltodextrin (Cromaline) and gum arabic (Proquimios, Rio de Janeiro, Brazil) were used as carrier agents. Folin-Ciocalteu reagent and gallic acid were purchased from Sigma Aldrich (USA). Sodium carbonate, sodium hydroxide and potassium biphthalate were purchased from Vetec Química Fina (Brazil).

2.1. Sample preparation

The carrier agents were added to the thawed camu-camu pulp at a concentration of 15% (w/w), according to the procedure recommended by Dib Taxi [9], submitted to homogenization in a blender and then filtered in a sieve with 0.4 mm pore diameter in order to reduce the suspended solids. The proportion of the carrier agent to the total solids of the filtered juice was about 3:1.

2.2. Processing conditions

The spray-drying process was performed in a laboratory-scale spray-dryer (Büchi Labortechnik AG, Switzerland), with a 1.5-mm-diameter nozzle and a main spray chamber of 70 cm × 65 cm × 110 cm. The mixture was kept under magnetic agitation, at room temperature, and fed into the main chamber through a peristaltic pump. The process was carried out with a drying air flow rate of 700 $L{\cdot}h^{-1}$ and a compressor air pressure of 8.3 bar. The feed flow rate used was 14.1 $mL{\cdot}min^{-1}$ for gum arabic and 18.1 $mL{\cdot}min^{-1}$ for maltodextrin. The inlet and outlet air temperatures were (180 ± 5) °C and (85 ± 5) °C, respectively. The process yield was estimated as the relationship between the total solids (g) in the final powder and the total solids in the feed mixture (g).

2.3. Analytical methods

Samples of camu-camu pulp and powder were collected for the determination of the following parameters (all determinations were performed in triplicate):

– pH was measured using an automatic titrator (Metrohm® model 785 DMP – Titrino, Switzerland), after instrument calibration with 4.00 and 7.00 pH buffers;

– total acidity was determined in the same titrator using sodium hydroxide solution and was expressed as mg of citric acid per 100 g of sample;

– total solids were measured gravimetrically by determining the dry weight in a vacuum oven at 65 °C. The pulp soluble solids content was determined using a lab refractometer (Bellingham + Stanley Ltd., England) with temperature correction (20 °C), and the results were expressed in °Brix;

– the spectrophotometric quantification of phenolic compounds in camu-camu pulp and powder was performed using Folin-Ciocalteu reagent according to Singleton and Rossi's methodology [10], adapted by Georgé *et al.* [11], using gallic acid as a standard; absorbance was measured at 760 nm using a UV–Vis spectrophotometer (Hewlett-Packard). Total phenolics were expressed as mg of gallic acid equivalent (GAE) per 100 g of sample;

– vitamin C was determined according to Benassi and Antunes [12]; the DCFI (2,6 dichlorine phenol indophenol) titration method was carried out with ascorbic acid as a standard.

2.4. Statistical analysis

Results were evaluated using one-way variance analysis (ANOVA). Fisher's test was performed to evaluate differences among

Table I.
Water activity (*Aw*) of oversaturated saline solutions at 25 °C [13].

Solution	Water activity (A_w) (25 °C)
LiCl	0.113
$MgCl_2$	0.328
K_2CO_3	0.432
KI	0.689
NaCL	0.743
KCl	0.843
$BaCl_2$	0.903
Distilled water	1.000

treatments. Differences at $p < 0.05$ were considered significant. The Statistica software for Windows version 9.0 was used to perform all statistical analyses.

2.5. Isotherm measurement

Sorption isotherms were measured using a static gravimetric method, where about 1 g of camu-camu powder was kept at constant relative humidity for two weeks. The resolution of the analytical balance for moisture sorption was 0.01 mg. Water activity results ranging from 0.093 to 1 at 25 °C were obtained by placing oversaturated salt solutions in the bottom of the desiccators (*table I*). The initial moisture contents in the powders were determined under vacuum at 65 °C up to constant weight.

The GAB (Guggenheim-Anderson-de Boer) models (equations 1) were selected to fit water sorption data [14]:

$$X_{eq} = \frac{X_m C_{GAB} f a_W}{(1 - f a_W)^*(1 + (C_{GAB} - 1) f a_W)}$$

where X_{eq} is the moisture content at equilibrium (on a dry basis), a_w is the water activity, X_m is the monolayer moisture content, and C_{GAB} and f are the model constants. The GAB parameters were evaluated by non-linear regression applying the Levenberg-Marquardt algorithm (STATISTICA, version 9.0).

2.6. Particle size measurement

Laser diffraction was used to determine the particle size distribution of the samples. This technique is important for many different research areas. Examples include spray characterization, foods and consumer products, and analysis and control of particulate [15]. The measurements were performed using a particle size analyzer (Horiba LB-550, Germany) in a nanometer range. Prior to measurement, about 10 mg of samples were diluted with 10 mL of isopropanol at 250 rpm for 10 min. The particle size was determined by the Fourier-transform technique. Each measurement was carried out in triplicate and the Sauter mean diameter (diameter of a sphere with an equivalent surface to volume ratio to all the particles in the size distribution) was determined.

2.7. Rheological behavior of emulsions

The evaluation of apparent viscosities of gum arabic and maltodextrin water emulsions (15% w/w) was carried out in a concentric cylinder rheometer (ARES, TA Instruments, USA). The tests were performed at 85 °C under steady-state conditions with the shear rate varying from 0 s^{-1} to 2000 s^{-1}.

3. Results and discussion

The camu-camu commercial pulp used in the present study, from the Brazilian Amazon region, presented 5.0 °Brix of soluble solids and around 5.8% of total solids. These results were slightly lower than those reported by Silva *et al.* for camu-camu pulp (6.0 °Brix and 6.6%, respectively) prepared on a lab scale using a domestic centrifuge and fruits grown in the state of São Paulo [16]. The titratable acidity of the pulp (2.9 g citric acid·100 g^{-1}) was higher than that determined by those

Table II.
Vitamin C content and total phenolic compounds in camu-camu pulp and spray-dried powders obtained with maltodextrin and gum arabic.

Product analysed	Vitamin C (mg·100 g^{-1})	Total phenolic compounds (mg GAE·100 g^{-1})
Camu-camu pulp	1,523 a to 1,603 a	575 a to 671 a
Camu-camu powder (gum arabic)	15,363 b ± 226	6,654 b ± 596
Camu-camu powder (maltodextrin)	11,258 c ± 298	5,912 b ± 582

Means with different letters in the same column indicate significant difference at $p < 0.05$.
GAE: gallic acid equivalent.

authors (2.3 g citric acid·100 g^{-1}). The pH of camu-camu commercial pulp (3.04 to 3.30) was inferior, as expected, to the pH of the spray-dried camu-camu (3.47 and 3.61).

Gum arabic and maltodextrin water emulsions (15% w/w) exhibited dilatant behavior. When the shear rate ranged from 20 s^{-1} to 2000 s^{-1} at 85 °C, the apparent viscosity of the gum arabic and maltodextrin emulsions increased, respectively, from (4.56 to 13.2) mPa·s^{-1} and from (1.77 to 6.59) mPa·s^{-1}.

When using gum arabic and maltodextrin as the carrier agents, the moisture results obtained for the spray-dried camu-camu powders were 2.8% and 3.2%, respectively; the process yield was 84% and 72%, respectively. A comparative study, using scanning electron microscopy, carried out by Krishnan *et al.* showed that microcapsules of cardamom oleoresin using gum arabic as wall material were nearly spherical, whereas the microcapsules using maltodextrin were broken [17]. Thus, gum arabic offered greater protection for oleoresin encapsulation than maltodextrin. Mosquera *et al.* also recommended gum arabic as a more effective wall material than maltodextrin to improve the handling of freeze-dried strawberry powder [18]. The higher apparent viscosity of gum arabic water emulsions imparts a positive effect on microencapsulation efficiency.

Camu-camu commercial pulp presented, on average, total phenolic compounds and vitamin C contents ranging, respectively, from (574 to 671) mg GAE·100 g^{-1} and from (1,523 to 1,603) mg ascorbic acid·100 g^{-1}. These figures are lower than those reported by Chirinos *et al.* for fresh Peruvian camu-camu red fruit (1,320 mg GAE·100 g^{-1} and 2,010 mg ascorbic acid·100 g^{-1}, respectively) [3], although the vitamin C contents were close to those reported by Dib Taxi for fresh camu-camu juice (1,432 mg·100 g^{-1}) [9], also obtained for the Brazilian Amazon fruits.

The spray-dried powder produced using gum arabic presented higher vitamin C and phenolic compounds than the powder obtained with maltodextrin (*table II*), suggesting that gum arabic was more effective than maltodextrin in preserving the bioactive compounds, although the concentration factor for the phenolic compounds was a little higher (10.3 *versus* 9.9) for the maltodextrin product.

The study carried out by Chirinos *et al.* provided information concerning the antioxidant potential of camu-camu fruit, expressed as the DPPH level value, which is not only related to its high ascorbic acid content but also to its phenolic content; most (68% to 79%) of the overall antioxidant capacity of camu-camu fresh fruit was attributed to its vitamin C content [3]. Based on these results, it is possible to consider that the antioxidant capacity of camu-camu powder was preserved after spray-drying, although it was not determined in this work.

When analyzing the vitamin C content of microencapsulated camu-camu powders on a dry basis, the contents of 158 mg·g^{-1} and 116 mg·g^{-1}, for gum arabic and maltodextrin, respectively, were higher than reported data (41 mg·g^{-1} to 108 mg·g^{-1}) for

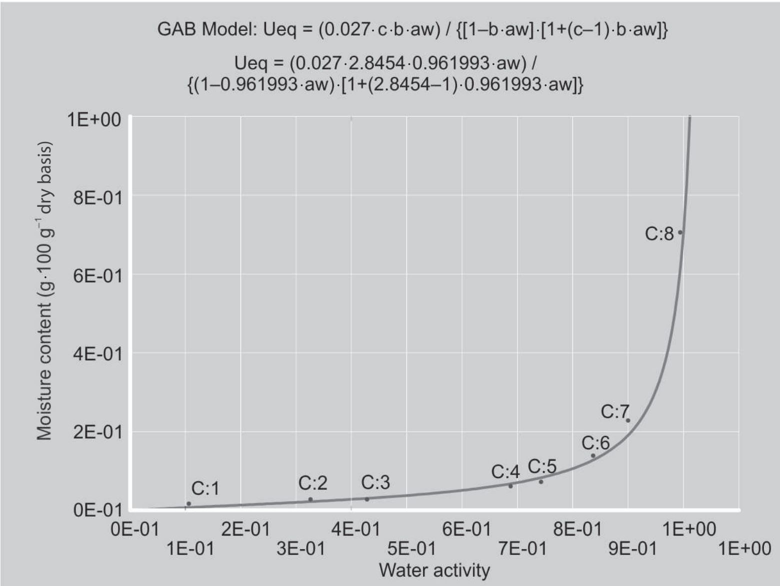

Figure 1.
Moisture sorption isotherms at 25 °C for camu-camu powder encapsulated with maltodextrin.

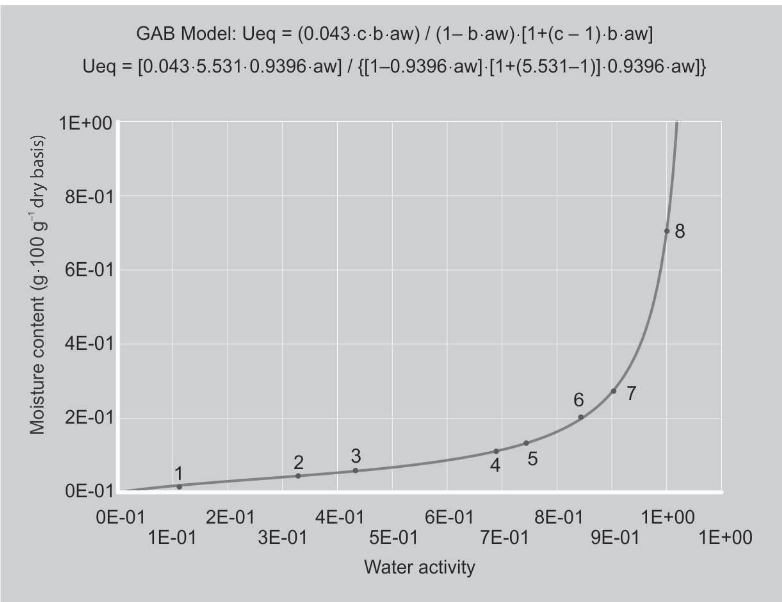

Figure 2.
Moisture sorption isotherms at 25 °C for camu-camu powder encapsulated with gum arabic.

concentrated camu-camu juice obtained by osmotic evaporation [19]. Among several concentration techniques, osmotic evaporation is considered to be the reference process for preserving the bioactive compounds.

3.1. Adsorption isotherms

The sorption isotherms of spray-dried camu-camu with maltodextrin and gum arabic at 25 °C show that there is good agreement between the experimental data and GAB model (*figures 1, 2*). The GAB equation has been recommended to represent experimental data of water sorption of food materials with water activity ranging from 0.10 to 0.90 [14]. The curves exhibit sigmoid-shaped (type II) isotherms, typically found for biological materials. It can be observed that, at the same water activities, the spray-dried camu-camu powder prepared with gum arabic adsorbed more water.

The results obtained using the model parameters estimated by non-linear regression ($R^2 > 0.97$) were dependent on the carrier agents (*table III*): C_{GAB} was 2.845 and 5.531 for the powders dried with maltodextrin and gum arabic, respectively. A relevant difference was observed for the monolayer moisture content, Xm = (0.0274 and 0.0438) g $H_2O{\cdot}g^{-1}$ (dry basis), for the spray-dried pulp with maltodextrin and gum arabic, respectively. Camu-camu powder liquefaction could be observed when water activity was above 0.8, for the camu-camu powder dried with gum arabic. Regarding the camu-camu powder with maltodextrin, this behavior occurred when water activity was above 0.9. These results indicate the higher water affinity of gum arabic when compared with maltodextrin and, consequently, the higher solubility of camu-camu powder, which is an important feature when judging the physical characteristics of juice powders.

3.2. Particle size measurement

Both samples showed unimodal particle size distributions; Sauter average diameters were 1.68 µm and 2.30 µm, respectively, for maltodextrin and gum arabic, with a standard deviation of 0.43 µm (*figures 3, 4*). When compared with a spray-dried açaí powder obtained by Tonon *et al.* [20], the camu-camu powder obtained in this work was more homogeneous and presented a smaller mean diameter, which is a desired

Table III.
Parameters of fitted experimental water sorption data by the GAB (Guggenheim-Anderson-de Boer) model.

Carrier agent	X_m	C_{GAB}	f	R^2
Gum arabic	0.0438	5.531	0.940	0.971
Maltodextrin	0.0274	2.845	0.962	0.997

X_m is the monolayer moisture content, and C_{GAB} and f are the constants of models.

result since, in general, water solubility is enhanced as particle size is reduced.

4. Conclusions

Gum arabic and maltodextrin were tested as carrier agents for camu-camu microencapsulation by spray-drying. The gum arabic was more effective than maltodextrin in preserving the bioactive compounds of the fruit. Furthermore, the gum arabic presented a higher water affinity when compared with maltodextrin. Neither the vitamin C nor the total phenolic contents presented significant loss during spray-drying. Thus, this technique can be employed for the processing of camu-camu pulp, preserving its heat-sensitive compounds.

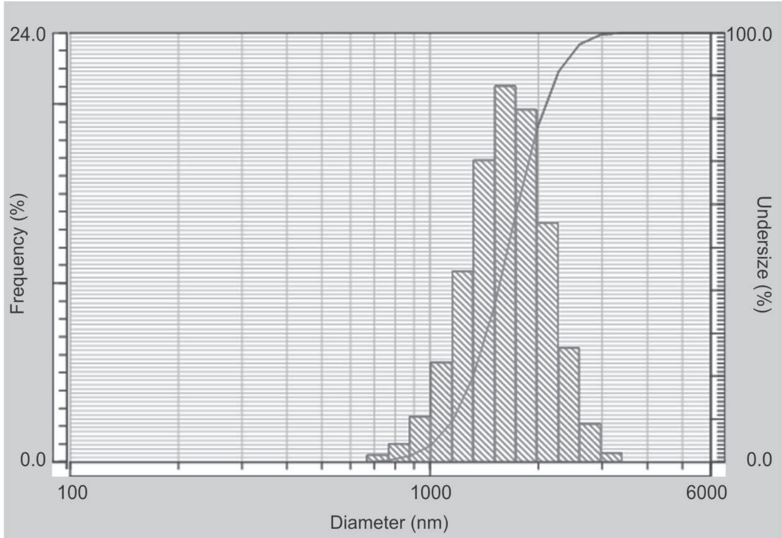

Figure 3.
Particle size distribution for camu-camu powder encapsulated with maltodextrin.

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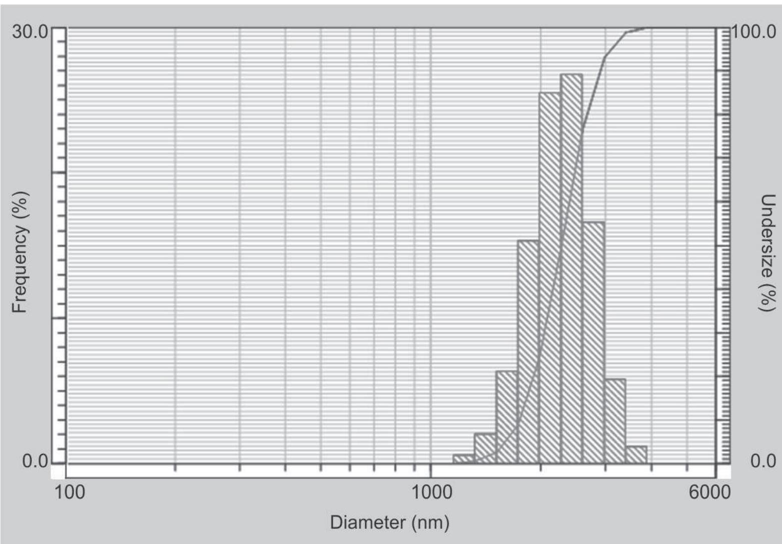

Figure 4.
Particle size distribution for camu-camu powder encapsulated with gum arabic.

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Influencia del material utilizado para el revestimiento del zumo de camu camu atomizado en la proporción de vitamina C, los compuestos fenólicos totales, las isotermas de sorción y la granulometría del producto.

Resumen – Introducción. El camu camu es una fruta originaria de la Amazonia, conocida, sobre todo, por su alto contenido en vitamina C. Dicha composición otorga a esta fruta una alta capacidad antioxidante y la convierte en una fuente potencial de productos antioxidantes. El secado de la pulpa mediante pulverización con la ayuda de un agente de apoyo es una técnica que se aplica para la conservación de importantes componentes de alimentos y medicamentos. Nuestro trabajo ha estado encaminado a evaluar la influencia de dichos agentes, utilizados como material de revestimiento, en la proporción de vitaminas y compuestos fenólicos totales del zumo de camu camu en polvo obtenido mediante el secado por atomizacion. **Material y métodos**. Como materia prima se utilizó pulpa de camu camu congelada comercial; la maltodextrina y la goma arábiga fueron los agentes de revestimiento elegidos. Los procesos se realizaron en un mini secador por pulverización con una temperatura del aire de admisión de 180 °C y del aire de salida de 85 °C, y un flujo del aire de secado de 700 $L \cdot h^{-1}$. Se usó la difracción por láser para determinar la distribución granulométrica de las muestras; las isotermas de sorción del zumo de camu camu atomizado se determinaron mediante un método gravimétrico estático. Se determinaron los compuestos fenólicos totales y la vitamina C tanto en la pulpa en bruto como en el polvo obtenido. **Resultados**. Cuando se utilizaron goma arábiga y maltodextrina como agentes de revestimiento, la humedad obtenida en el polvo de camu camu secado mediante pulverización fue del 2,8 % y el 3,2 %, respectivamente; el rendimiento del proceso fue del 84 % y el 72 %, respectivamente. El polvo secado mediante pulverización obtenido con ayuda de la goma arábiga presentó proporciones más elevadas de vitamina C [(15,363 ± 226) mg·100 g^{-1}] y de compuestos fenólicos [(6,654 ± 596) mg GAE·100 g^{-1}] que el polvo obtenido con maltodextrina, respectivamente (11,258 ± 298) mg·100 g^{-1} y (5,912 ± 582) mg GAE·100 g^{-1}. **Conclusion**. El factor de concentración de la vitamina C y de los compuestos fenólicos en el polvo de camu camu demuestra la eficacia del secado por atomización para preservar la capacidad antioxidante de esta fruta. La goma arábiga constituye una barrera más eficaz que la maltodextrina para la retención de los compuestos bioactivos.

Brasil / *Myrciaria dubia* / frutas / procesamiento de alimentos / secado por pulverización / microencapsulación / ácido ascórbico