PAB
Pesquisa Agropecuária Brasileira

ISSN 1678-3921

Journal homepage: www.embrapa.br/pab

For manuscript submission and journal contents, access: www.scielo.br/pab

Food Technology/ Original Article

Hygroscopic behavior of bacuri powders

Abstract – The objective of this work was to evaluate the hygroscopic behavior and physical properties of powders from bacuri pulp freeze-dried with different drying adjuvants. Three samples were prepared by adding the maltodextrin, gum Arabic, and albumin adjuvants, at a concentration of 20% (w/w), to the pulp. The powders were analyzed for moisture content, hygroscopicity, and particle morphology. The adsorption isotherms were obtained using the models of Brunauer-Emmett-Teller (BET), Guggeheim-Anderson-de Bôer (GAB), Henderson, and Oswin at the temperatures of 25 and 40ºC. Moisture contents ranged from 2.12 to 2.90%, and the lowest value was observed for the powder containing albumin. Hygroscopicity varied from 3.64 to 6.02%, with the lowest value for the powder containing maltodextrin. The powder particles showed non-spherical and irregular shapes, being more agglomerated in the powder with maltodextrin. For the isotherm, the best fit was obtained with the BET model and a type-III behavior was observed, which is typical of foods rich in soluble sugars. Therefore, the adjuvants contribute to the physicochemical properties of the powders, making them less hygroscopic and with irregular-shaped particles.

Index terms: adjuvants, adsorption, drying, moisture content, tropical fruits.

Comportamento higroscópico de pós de bacuri

Resumo – O objetivo deste trabalho foi avaliar o comportamento higroscópico e as características físicas de pós da polpa de bacuri liofilizada com diferentes adjuvantes de secagem. Foram preparadas três amostras, tendo-se adicionado os adjuvantes maltodextrina, goma arábica e albumina, na concentração de 20% (m/m), à polpa. Os pós foram analisados quanto a teores de umidade, higroscopicidade e morfologia das partículas. As isotermas de adsorção foram obtidas com uso dos modelos de Brunauer-Emmett-Teller (BET), Guggeheim-Anderson-de Bôer (GAB), Henderson e Oswin, nas temperaturas de 25 e 40ºC. Os teores de umidade variaram de 2,12 a 2,90%, e o menor valor foi observado para o pó contendo albumina. A higroscopicidade dos pós variou de 3,64 a 6,02%, com o menor valor para o pó com maltodextrina. As partículas dos pós apresentaram formatos não esféricos e irregulares, tendo sido mais aglomeradas no pó contendo maltodextrina. Para a isoterma, o melhor ajuste matemático foi obtido com o modelo BET e o comportamento observado foi do tipo III, característico de alimentos ricos em açúcares solúveis. Portanto, os adjuvantes contribuem para as características físico-químicas dos pós, tornando-os menos higroscópicos e com partículas irregulares.

Termos para indexação: adjuvantes, adsorção, secagem, teor de umidade, frutas tropicais.

Tháila Pimentel Albuquerque Moura[(1)], Andréa Cardoso de Aquino[(1)], Sueli Rodrigues[(1)], and Marcos Rodrigues Amorim Afonso[(1 ✉)]

[(1)] Universidade Federal do Ceará, Departamento de Engenharia de Alimentos, Campus do Pici, Bloco 858, CEP 60440-554 Fortaleza, CE, Brazil. E-mail: thaila.pim@gmail.com, andreacdeaquino@gmail.com, sueli@ufc.br, mafonso@ufc.br

✉ Corresponding author

Received
September 1, 2023

Accepted
May 10, 2024

How to cite
MOURA, T.P.A.; AQUINO, A.C. de; RODRIGUES, S.; AFONSO, M.R.A. Hygroscopic behavior of bacuri powders. **Pesquisa Agropecuária Brasileira**, v.59, e03503, 2024. DOI: https://doi.org/10.1590/S1678-3921.pab2024.v59.03503.

Introduction

Bacuri (*Platonia insignis* Mart.) is a tropical fruit known for its significant research potential and high levels of ascorbic acid, fiber, and bioactive compounds (Teixeira et al., 2019). The species is primarily found in the Amazon biomes of Brazil, specifically in the states of Piauí and Maranhão, with limited production areas in Mato Grosso and Tocantins (Chitarra & Chitarra, 2006).

Although bacuri can be used for diverse products, its perishable nature and seasonal availability pose challenges for its distribution to other regions of the country. Therefore, the application of suitable technologies is necessary to ensure a continuous supply of the fruit, such as pulp drying for long-term storage and a facilitated transportation (Ribeiro et al., 2016).

Several drying processes can be used to convert foods into dry-powder products, whose particle size varies depending on the intended purpose and food type. Fruit powders, in particular, often contain high sugar levels, which can lead to undesirable effects such as an increased hygroscopicity and agglomeration at temperatures above the glass transition temperature (Ribeiro et al., 2016). Therefore, certain physicochemical properties are used as references for powder handling and processing (Maciel et al., 2020).

In the design and analysis of the drying process, the study of isotherms plays a key role. The behavior of sorption isotherms can be predicted based on the structural, dynamic, and thermodynamic aspects of water through developed mathematical models (Park et al., 2008), such as those of Brunauer-Emmett-Teller (BET), Guggehneim-Anderson-de Bôer (GAB), Henderson, and Oswin.

The objective of this work was to evaluate the hygroscopic behavior and physical properties of powders from bacuri pulp freeze-dried with different drying adjuvants.

Materials and Methods

The used bacuri pulps were obtained from a fruit processing company located in the northern region of the state of Piauí, Brazil. The pulps were stored in a freezer at -18ºC, being thawed prior to the drying process. Three different samples were prepared by adding the following drying adjuvants at a concentration of 20% (w/w): maltodextrin (Maltogill 20DE, Cargill, Minneapolis, MN, USA), gum Arabic (CAS9000-01-5, Êxodo Científica, Sumaré, SP, Brazil), and 80% albumin (Proteína Pura, Netto Alimentos, São Paulo, SP, Brazil). The samples were homogenized using the 200 W Versatile Black mixer (Mondial, Barueri, SP, Brazil) and then frozen at -38ºC, for 24 hours, in the CL90-40V ultra-freezer (Terroni Equipamentos Científicos, São Carlos, SP, Brazil). The frozen samples were transferred to the LS 3000 freeze dryer (Terroni Equipamentos Científicos, São Carlos, SP, Brazil) and dried for 24 hours until reaching final pressures of 20–30 Pa. After drying, the samples were crushed using the 800 W ECO industrial blender, with a 2 L stainless steel cup (Spolu, Itajobi, SP, Brazil), at 18,000 rpm, for 1 min and then stored in laminated packaging until further analysis.

The moisture content and hygroscopicity of the powder samples were determined according to the method described by Instituto Adolfo Lutz (Procedimentos..., 2008) and to Goula & Adamopoulos (2008), respectively. Samples with each adjuvant were analyzed in three replicates, and the results were expressed as mean ± standard deviation. The assumptions of normality, homoscedasticity, and independence of errors were checked using the tests of Shapiro-Wilk, Bartlett, and Durbin-Watson, respectively, at 5% probability. All assumptions were met. The normality of data was tested by the Statistica software (TIBCO Software Inc., Palo Alto, CA, USA). The results were analyzed using the one-way analysis of variance, followed by Tukey's post-hoc test, also using the Statistica software (TIBCO Software Inc., Palo Alto, CA, USA), with differences considered significant at a 95% confidence level.

The powder particles were examined using the Quanta FEG 540 scanning electron microscope (FEI Company, Thermo Fisher Scientific, Waltham, MA, USA). For this, the powder samples were placed on double-sided adhesive tape and fixed onto a metallic support. Subsequently, the metallic plate with the powders was coated with platinum and gold using the Q1550TES sputter coater (Quorum, Laughton, East Sussex, United Kingdom). Micrographs of the powders were captured at magnifications of 1,000 and 2,000x.

The gravimetric method described by Spiess & Wolf (1987) was used to determine the adsorption isotherms of the samples. Triplicate samples of 1.0 g powder were pre-weighed into aluminum crucibles, which were placed in closed reservoirs containing saturated saline solutions of CH_3COOK, K_2CO_3, NaBr, $SnCl_2$, KCl, and $BaCl_2$. The reservoirs were then transferred to the MA415 BOD oven (Marconi Equipamentos para Laboratório Ltda, Piracicaba, SP, Brazil) with temperature control (25 and 40ºC), where they were kept until reaching a constant weight. Weight was measured every 24 hours until a variation of less than 1% was achieved. After reaching equilibrium at each temperature, the water activities of the samples were determined using the 4TE AquaLab meter (Decagon Devices, Meter Group, Pullman, WA, USA). The equilibrium moisture (X_0) of each sample was calculated through the following equation (Moreira et al., 2013):

$$X_0 = \frac{W_{eq} - W_s}{W_s},$$

where X_0 is the equilibrium moisture content (g/g), W_{eq} is sample weight at equilibrium (g), and W_s is the weight of the dried sample (g).

When analyzing the isotherms of the samples, mathematical adjustments were performed using the BET, GAB, Henderson, and Oswin models (Table 1), whose parameters were determined using the Statistica software (TIBCO Software Inc., Palo Alto, CA, USA). The evaluation of the models took into account the values of the adjusted coefficient of determination and the mean relative error, calculated according to the following equation:

$$E = \frac{100}{n}\sum_{i-1}^{n}\frac{\left|\left(M_i - Mp_i\right)\right|}{M_i},$$

where E is the mean relative error (%), Mi are the values obtained experimentally, Mpi are the values predicted by the model, and n is the number of experimental data.

Table 1. Mathematical models used for fitting the adsorption isotherms of freeze-dried bacuri (*Platonia insignis*) pulp powders.

Model[(1)]	Equation[(2)]
GAB	$X_e = \frac{X_m.C.K.a_w}{(1-K.a_w).(1-K.a_w+C.K.a_w)}$
BET	$X_e = \frac{X_m.C.a_w}{(1-a_w)}.\left[\frac{1-(n+1).(a_w)^n + n.(a_w)^{n-1}}{1-(1-C).a_w{}^n - C.(a_w)^{n+1}}\right]$
Henderson	$Xe = \left[\frac{-1n(1-a_w)}{b}\right]^{\frac{1}{a}}$
Oswin	$Xe = a.\left[\frac{a_w}{1-a_w}\right]^b$

(1)BET, Brunauer-Emmett-Teller; and GAB, Guggehneim-Anderson-de Bôer (GAB). (2)X_e, equilibrium moisture content (g/g); X_m, water content in the molecular monolayer (g/g); a_w, water activity; n, number of molecular layers; C, K, constants of sorption; and a, b, model fitting parameters.

Results and Discussion

The powder containing albumin showed the lowest value, which differed significantly ($p<0.05$) from that of the others (Table 2). The moisture content of the powders from all three samples was below 5%, which is considered microbiologically safe according to Fazaeli et al. (2012), since a low moisture content contributes to a better fluidity and storage stability of the product. Similar moisture contents were reported by Maciel et al. (2020) for powdered guava (*Psidium guajava* L.) pulp freeze-dried with 3.01 to 3.34% maltodextrin and by Poornima & Sinthiya (2017) for beet (*Beta vulgaris* L.) extract powder containing 1.0 to 3.4% maltodextrin and gum Arabic. According to Poornima & Sinthiya (2017) and Goula & Adamopoulos (2008), the moisture content of different products may vary depending on their composition and on the used drying methods, in which drying temperature and adjuvant concentration directly influence the moisture content of the powder.

According to the hygroscopicity results, bacuri pulp powders are classified as non-hygroscopic (Table 2),

Table 2. Mean values of the physicochemical characterization of freeze-dried bacuri (*Platonia insignis*) pulp powders containing 20% (w/w) maltodextrin, gum Arabic, and albumin.

Analysis	Powder samples[(1)]		
	Maltodextrin	Gum Arabic	Albumin
Moisture (%)	2.90±0.10a	2.60±0.05a	2.12±0.17b
Hygroscopicity (%)	3.64±0.25b	6.02±0.58a	5.07±0.27a

(1)Means followed by equal letters, in the same row, do not present a statistical difference between the samples ($p<0.05$).

DOI: 10.1590/S1678-3921.pab2024.v59.03503

i.e., with values lower than 10% (GEA Niro Research Laboratory, 2003). The sample containing maltodextrin showed the lowest hygroscopicity and differed significantly from the others ($p<0.05$). Ruengdech & Siripatrawan (2022) reported similar findings in their study for catechin nanoemulsion powders freeze-dried using foam mat containing maltodextrin and gum Arabic, observing that the samples with maltodextrin presented lower hygroscopicity values of 8.2 and 10.1%. Oliveira et al. (2014) found a hygroscopicity of 8.51% for the pulp powder of yellow mombin (*Spondias mombin* L.) freeze-dried with maltodextrin. Conversely, Braga et al. (2019) observed higher hygroscopicity values of 13.06 and 16.58% for powders from blackberry (*Rubus* spp.) freeze-dried with maltodextrin, concentrated powdered milk, and Capsul. These authors explained that the low hygroscopicity values of the freeze-dried powders may be attributed to their particle sizes, as larger particles result in a smaller total surface area and lower water absorption.

Gum Arabic facilitated moisture absorption from the environment, which can be attributed to its branched structure containing hydrophilic groups. According to Ruengdech & Siripatrawan (2022) and Ganaie (2021), the hygroscopicity of powders is influenced by the number of hydrophilic groups present in the structure of each drying adjuvant, which determines the interactions between the hydrogen in the water molecules and the hydroxyl groups in the amorphous regions of the matrix or substrate.

Overall, the micrographs of the powders containing 20% (w/w) drying adjuvants showed that the three samples exhibited non-spherical and irregular shapes (Figure 1). This characteristic is commonly observed in fruit-based products, as they often contain a high content of fiber, sugar, and fat, leading to the formation of complex granules and agglomerates (Zea et al., 2013). Similarly, Braga et al. (2019) found that powders from blackberry freeze-dried with maltodextrin, concentrated milk, and Capsul presented irregular and porous particles. According to Ozkan et al. (2019), the non-uniformity in particle size often occurs during the grinding process after drying, indicating a lack of control over the size of the particles. Additionally, in the present study, the powder particles showed small depressions and porous surfaces, which can be due to ice sublimation during the freeze-drying process.

The particle sizes were smaller for the samples containing gum Arabic (Figure 1 C and D), when compared with those with albumin (Figure 1 E and F). Moreover, the sample containing maltodextrin showed more agglomerated particles than those produced with gum Arabic and albumin (Figure 1A). Ganaie et al. (2021) also observed particle agglomeration in honey powders freeze-dried with maltodextrin, attributing this behavior to the glass transition of amorphous carbohydrate matrices. Therefore, products with a high sugar content tend to have a greater tendency for particle agglomeration.

The fitting of the mathematical models varied for the powders from bacuri pulp freeze-dried with 20% (w/w) maltodextrin, gum Arabic, and albumin (Table 3). According to Cavalcante et al. (2018), the appropriate model should be selected based on the lowest mean relative error, which should be below 10%. Based on this criterion, the GAB model should have been considered the best model; however, a physical inconsistency was observed in the K-value, which exceeded 1.0 for maltodextrin at 40ºC (Chirife et al., 1992), implying an infinite sorption. Consequently, the BET model, with the second lowest mean relative error, was considered the best fit to represent the isotherm of bacuri pulp powder. Ribeiro et al. (2016) also selected the BET model, with a mean relative error of 6.95%, for acerola (*Malpighia glabra* L.) powder freeze-dried with 19.1% maltodextrin. Furthermore, Oliveira et al. (2014) found that BET, with a mean relative error of 9.14%, was the most suitable mathematical model for yellow mombin powder freeze-dried with 17% (w/w) maltodextrin.

According to Moreira et al. (2013), the GAB and BET models, which are the most used for food products, are fit to evaluate the moisture content of the primary layer (monolayer, X_m) of foods, providing insights into their adsorption capacity. The water content of the molecular monolayer plays a crucial role in determining the hygroscopicity and affinity of molecules for water, as well as the overall stability of the food (Celestino, 2010).

Based on the BET parameters (Table 3), the moisture content of X_m increased with rising temperature. This temperature-induced change is often associated with alterations in the physical structure of the powdered product, leading to a greater number of active sites with affinity for water molecules, which results in an increased monolayer content (Moreira et al., 2013).

DOI: 10.1590/S1678-3921.pab2024.v59.03503

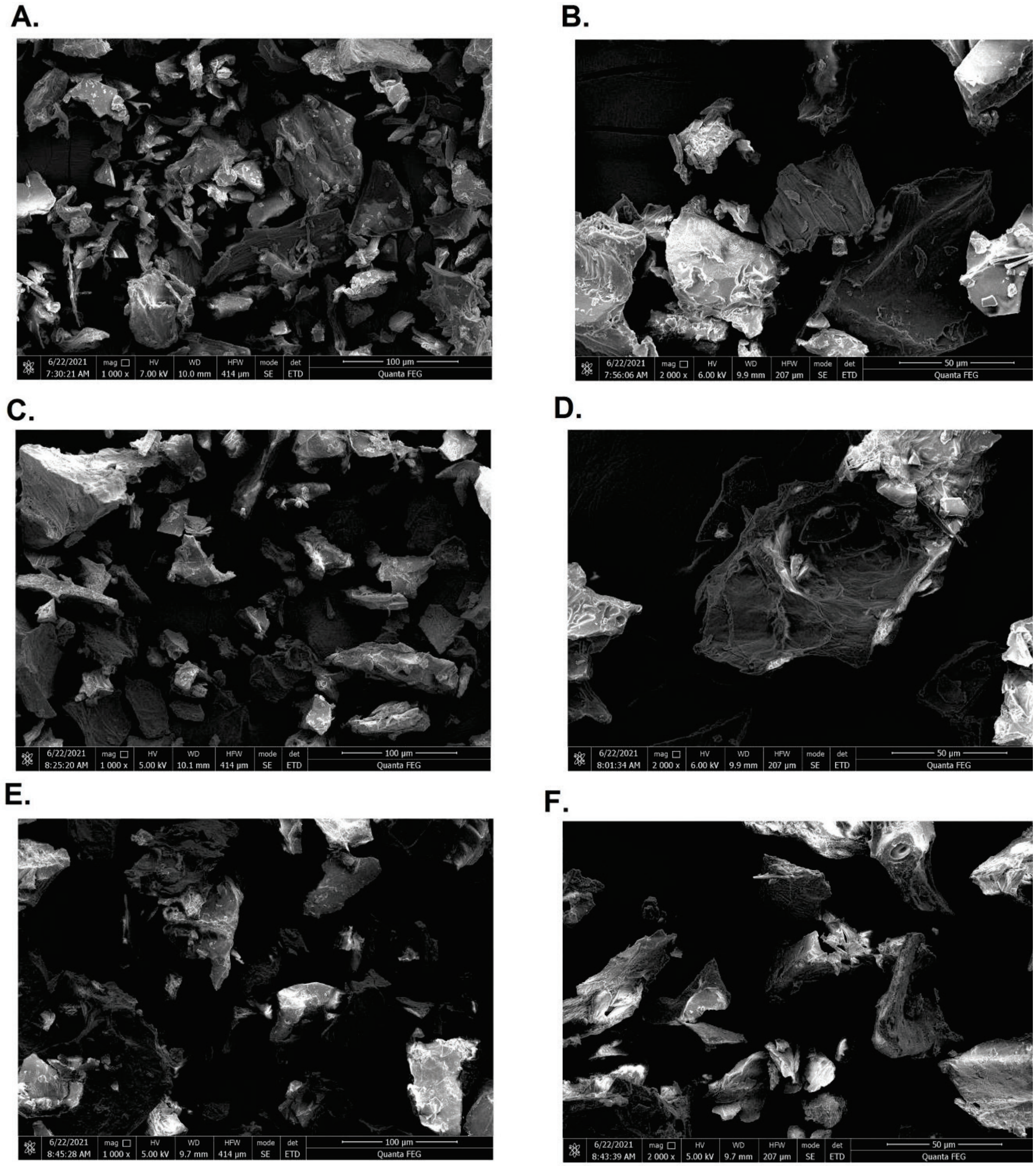

Figure 1. Micrographs (at magnifications of 1,000 and 2,000x) of particles of freeze-dried bacuri (*Platonia insignis*) pulp powders containing 20% (w/w) maltodextrin (A and B), gum Arabic (C and D), and albumin (E and F).

Retaining a higher amount of water in the monolayer at a given temperature contributes to a greater stability and lower quality loss in the product (Goula & Adamopoulos, 2008).

In the BET model, the X_m values observed for the powders containing maltodextrin, gum Arabic, and albumin (Table 3) were similar to those from 0.030 to 0.065 g water per g solids obtained at 25°C by Stępień et al. (2020a) for avocado (*Persea americana* Mill.) powders with inulin and maltodextrin. However, Maciel et al. (2020) found higher X_m values, from 0.3808 to 0.5411 g water per g solids, at 35 and 45ºC, for guava powders with 4.0 and 8.0% albumin. Using the BET model, Ribeiro et al. (2016) described a similar behavior for X_m in acerola powder freeze-dried at different temperature ranges (25, 35, and 45ºC). Stępień et al. (2020a) added that increasing inulin and maltodextrin concentrations in freeze-dried avocado powders resulted in a higher moisture content in the monolayer, at 25°C, in the GAB and BET models. In these last two studies, both temperature and the type/concentration of the adjuvant influenced the increase observed in the monolayer. The objective is usually to reduce water absorption, considering that the high sugar content in fruits can lead to an increased solubility at high temperatures during prolonged storage. Although this effect can be mitigated by the use of carrier agents, the efficacy of this process depends on the combination of carrier agent and raw material.

The sorption constants of the molecular layer decreased with increasing temperature (Table 3), indicating the influence of temperature on the equilibrium moisture content between samples. The C and K constants indicate the type of isotherm and the bonding behavior of the water molecules on the surface of the obtained product. In the present study, the isotherms showed a type III classification (Figure 2), characterized by a J shape, which is typical for foods

Table 3. Results for the fitting of the adsorption isotherm of freeze-dried bacuri (*Platonia insignis*) pulp powders containing 20% (w/w) maltodextrin, gum Arabic, and albumin at 25ºC and 40ºC.

Model[(1)]	Parameter[(2)]	Powder sample					
		Maltodextrin		Gum Arabic		Albumin	
		25ºC	40ºC	25ºC	40ºC	25ºC	40ºC
GAB	X_m	0.0767	0.0570	0.117	0.102	0.0876	0.0950
	C	1.13	1.21	1.19	0.789	1.24	0.565
	K	0.968	1.05	0.862	0.940	0.909	0.956
	R^2	0.996	0.999	0.997	0.999	0.998	0.998
	E (%)	4.79	5.29	2.92	5.25	3.21	9.25
BET	X_m	0.0604	0.0968	0.0664	0.0741	0.0582	0.0730
	C	1.89	0.496	2.61	1.12	2.25	0.731
	n	168	172	16.6	22.1	19.9	24.9
	R^2	0.998	0.998	0.998	0.999	0.999	0.998
	E (%)	4.16	10.48	1.76	4.48	1.78	8.90
Henderson	a	0.650	0.486	0.821	0.625	0.743	0.551
	b	3.74	2.77	3.92	3.46	4.52	3.32
	R^2	0.988	0.991	0.996	0.997	0.997	0.983
	E (%)	9.80	19.7	3.92	10.19	5.91	15.34
Oswin	a	0.0774	0.0651	0.102	0.0820	0.0854	0.0653
	b	0.896	1.15	0.684	0.882	0.742	0.976
	R^2	0.997	0.998	0.991	0.998	0.995	0.997
	E (%)	4.03	10.0	6.10	3.87	2.29	6.66

[(1)]BET, Brunauer-Emmett-Teller; and GAB, Guggeheim-Anderson-de Bôer (GAB). [(2)]X_m, moisture content in the molecular monolayer (g/g); C, K, sorption constants; n, number of molecular layers; a, b, fitting parameters; R^2, coefficient of determination; and E, mean relative error (%).

rich in soluble components according to Brunauer et al. (1940); this classification may deviate from typical isotherm analyses but can vary depending on the product. Similar isotherm types and shapes were reported by Maciel et al. (2020), Ribeiro et al. (2016), and Bernal et al. (2015) for powders of guava dried in tray dryers, freeze-dried acerola pulp, and borojó (*Borojoa patinoi* Cuatrec.) freeze-dried with gum Arabic, respectively.

There was a substantial increase in equilibrium moisture for the samples within the water activity range of 0.6 to 0.7, at 25ºC, and of 0.5 to 0.6, at 40ºC (Figure 2 A and B). Therefore, the samples tend to be more stable when exposed to environments with a relative humidity below 50%. However, within the water activity range of 0.5 to 0.6, corresponding to a relative humidity between 50 and 60%, there was an increased water adsorption by the powder particles, resulting in a higher moisture content and compromised integrity. Among the powder samples, those containing maltodextrin showed lower equilibrium moisture values at low water activities, but an increased moisture content at water activities of 0.8 and from 0.7 to 0.8 in the 25 and 40ºC isotherms, respectively. Other authors (Rodríguez-Bernal et al., 2015; Silva et al., 2018; Stępień et al., 2020b) also found that fruit-based products often show a water activity proportional to the increase in relative humidity at a constant temperature (Figure 2).

No significant differences were observed when bacuri powder was exposed to higher temperatures (Figure 2B). Despite this, according to Al-Muhtaseb et al. (2010), foods whose constituents have a low molecular weight, such as organic acids, salts, and sugars, may become more hygroscopic at higher temperatures due to solubilization. In the case of the present study, the adjuvants contributed to mitigating these effects at a temperature of 40ºC, with albumin showing a greater stability than the other adjuvants.

Cavalcante et al. (2018), Stępień et al. (2020b), and Silva et al. (2018) also observed an increase in relative moisture beyond a water activity of 0.6 in the powders of soursop (*Annona muricata* L.) spray-dried with maltodextrin, pumpkin (*Cucurbita maxima* Duchesne) freeze-dried with maltodextrin, and tucumã (*Astrocaryum aculeatum* G.Mey.) freeze-dried with gum Arabic, maltodextrin, dextrin, and modified starch, respectively. Silva et al. (2018) concluded that an increased water adsorption leads to particle agglomeration and a reduction in the glass transition temperature of the adjuvant, potentially causing physicochemical changes in the product and reducing its stability.

Considering the moisture gain starting at water activities of 0.5 to 0.6, it is recommended to store bacuri powder under conditions that do not exceed these limits in order to prevent potential product alterations due to an increased hygroscopicity.

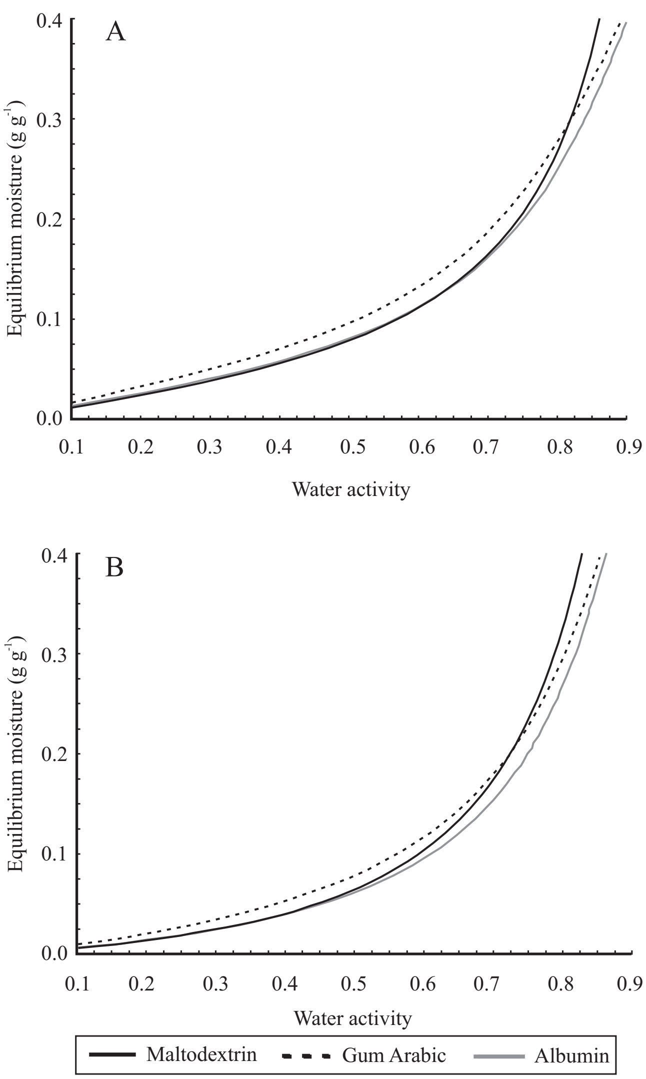

Figure 2. Adsorption isotherms of freeze-dried bacuri (*Platonia insignis*) pulp powders containing 20% (w/w) maltodextrin, gum Arabic, and albumin at 25ºC (A) and 40ºC (B) in the best-fitting model of Brunauer-Emmett-Teller.

Conclusions

1. The addition of drying adjuvants results in a low moisture content in freeze-dried bacuri (*Platonia insignis*) powders.
2. The inclusion of maltodextrin reduces the hygroscopicity of the powder.
3. The inclusion of albumin improves the stability of the powder in environments with a high relative humidity.
4. The particles of the powder show sharp and irregular shapes.
5. For the isotherms of bacuri pulp powder, classified as type III, the best-fit model is that of Brunauer-Emmett-Teller.

Acknowledgments

To Coordenação de Aperfeiçoamento de Pessoal de Nível Superior (CAPES), for financing, in part, this study (Finance Code 001); and to Central Analítica of Universidade Federal do Ceará (funding programs Finep-CT-INFRA, Pró-Equipamentos CAPES, and MCTI-CNPq-SisNano2.0), for the microscopic measurements.

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