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RESEARCH ARTICLE

Moisture Sorption Behavior of Cupuassu Powder

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Abstract:

Introduction:

In this research, the hygroscopic behavior of cupuassu (*Theobroma grandiflorum*) powder containing 40% maltodextrin was studied via the moisture adsorption and desorption isotherms at 25°C.

Methods:

The experimental sorption data of the cupuassu powder were fitted using the Halsey, Henderson, Oswin, GAB and Peleg models. In addition, the powder morphology was assessed using scanning electron microscopy.

Results:

Moisture sorption isotherms curves showed type III behavior, typical of foods rich in soluble components, such as sugars, present in the samples. The adsorption curve indicated that the product requires greater attention when stored and handled in environments with relative humidity above 50%. The microbiological stability of the product is assured up to 11.5% moisture content.

Conclusion:

During the product storage, it is recommended to use packaging with water vapor and air impermeability, due to the presence of porous microspheres that affect the protection of the active material and facilitate the moisture gain. Among the models evaluated, the Peleg and GAB models presented good suitability on predicting the product's sorption isotherms.

Keywords: Maltodextrin, Mathematical modeling, Moisture sorption isotherm, Storage, GAB, *Theobroma grandiflorum*.

1. INTRODUCTION

Cupuassu (*Theobroma grandiflorum*) is a tropical fruit native of the Amazon region, which presents citrus pulp with high fiber content and strong aroma. This fruit is considered an excellent raw material to be used in the food industry due to its variety of food applications and nutritional benefits attributed to the presence of ascorbic acid and total phenolics [1, 2]. Thus, the cupuassu has a high economic potential and it can be used in the development of new functional ingredients for food enrichment [3]. An alternative for the elaboration of new products is the use of specific processes such as spray drying to obtain powdered fruit, which guarantees the product's stability by the reduction of the water activity, a_w [4] and practically

does not compromise its sensorial and physicochemical characteristics, leading to the production of good quality foods [5].

However, powdered products obtained from fruits are generally very hygroscopic [4], which may compromise their physical, chemical and microbiological stability. The knowledge of the hygroscopic behavior of powdered products can be carried out using the Moisture Sorption Isotherms (MSI) [6], that describe the relationship between the equilibrium moisture content of the food material and its a_w, at a given temperature [7]. The adsorption curve is related to the moisture gain, while the desorption represents the moisture losses of a product [5]. The difference between the curves is called hysteresis and its effect may occur due to several factors, such as capillary condensation, changes in the physical structure of the material, surface impurities and phase change [8]. Therefore, moisture sorption isotherms are considered important thermodynamic

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tools and provided useful information for food processing operations such as drying, packaging and storage [9]. The isotherms are used to calculate the drying time, to predict ingredients behavior in mixtures, for packaging selection, for modeling the moisture changes during storage and to estimate shelf life stability; these as particularly important factors mainly for powdered foods [10].

Numerous mathematical models have been proposed to describe food moisture sorption behaviors. Some of these models are based on theories of the sorption mechanism; others are semi-empirical or purely empirical. The theoretical models of BET [11] (based on monolayer) and GAB (based on a multilayer and condensed film) [12]; semi-empirical of Halsey [13] and Henderson [14]; and empirical of Oswin [15] and Peleg [16], are commonly used for MSI prediction of powdered products.

The GAB model has a theoretical background since it is a refinement of the physical adsorption theory of Langmuir and BET. With three parameters, the GAB model can represent the experimental moisture sorption data of foods in the water activity range of greater practical interest [17]. The GAB model has been recommended by the European Project Group COST 90 on Physical Properties of Foods as the fundamental equation to characterize the water sorption of food materials [17]. On the other hand, the Peleg model is a four-parameter model, which is a purely empirical equation without a theoretical background. However, those models have shown goodness fit for the prediction of the moisture sorption isotherms of fruits powders [18 - 21].

Despite the availability of the substantial number of equations, no single equation has the ability to describe accurately the moisture sorption behavior of food products over the entire range of Relative Humidity (RH) and that's because water is associated with the food matrix by different mechanisms for different values of a_w [7]. According to Lomauro *et al.* [22], detailed research in the literature showed that MSI of foods could be described by more than one mathematical model. Therefore, it is necessary to select the model that best fits the moisture sorption data for each specific product.

Studies about the hygroscopic behavior of cupuassu powder containing maltodextrin as drying agent are not available in the literature, which indicates the need of studies to verify the behavior of this product when maltodextrin is added in its composition. In this context, the aim of this work was to determine the adsorption and desorption isotherms of the product, choosing the appropriate packaging and storage conditions; as well as fitting different mathematical models to the experimental data, to select the model that best describes the moisture sorption phenomenon of the powder product.

2. MATERIALS AND METHODS

2.1. Materials

The cupuassu pulp was supplied by the Mixed Agricultural Cooperative of Tomé-Açu (CAMTA), located in the city of Tomé-Açu, Pará, Brazil (02° 25' 08" S and 48° 09' 08" W). The pulp was stored in polyethylene bags (1 kg), frozen at -18°C, and transported to the Federal University of Pará (UFPA) (01° 27' 21" S and 48° 30' 16" W). For the experiments, the pulp was thawed under refrigeration (≈ 4°C) and then brought to room temperature (≈ 25°C). Maltodextrin with a dextrose equivalent of 20 (Maltogill 20, Cargill Agrícola SA, Uberlândia, Brazil) was used as a drying agent.

2.2. Preparation of Cupuassu Powder

The cupuassu powder was obtained in a mini spray dryer (Büchi B-290, Büchi Labortechnik AG, Flawil, Switzerland) at the drying laboratory of the Federal University of Pará (UFPA) (01° 27' 21" S and 48° 30' 16" W). The mixture (pulp:water, ratio 1:1 w/w) was homogenized in a colloidal mill (Brasil 56-RC-6332, São Paulo, Brazil) for 3 min and filtrated through 0.30 mm mesh. Then, 40% maltodextrin was added under the 6.84% total solids content of the filtered mixture. The spray dryer was fed with the formulation into the chamber by a peristaltic pump, using a feed flow rate of 7.5 mL/min and feed temperature of 25°C. The equipment was programmed to operate with *nozzle* internal diameter of 0.7 mm, parallel current flow, aspiration air flow in 100%, compressed air pressure of 0.8 MPa, drying air flow rate of 35 m³/h and inlet and outlet air temperatures of 150 and 93°C, respectively.

2.3. Powder Analysis

The physicochemical properties of cupuassu powder were determined according to the methods proposed by the AOAC [23]: moisture in a vacuum oven at 70°C (Marconi MA030/12, São Paulo, Brazil) (method 920.151); ashes (method 940.26); protein (method 920.152) (nitrogen-protein conversion factor of 6.25); lipids (method 963.15); total and reducing sugars (method 920.183b); total titratable acidity (method 942.15A); pH in a potentiometer (Hanna HI 2221, Woonsocket, USA) (method 981.12); and Ascorbic Acid content (AA) (method 967.21). Other analyses were also performed, such as: total phenolic compounds (TPC) according to the methodology described by Singleton and Rossi [24], using a spectrophotometer (Pharmacia Biotech Ultrospec 2000, Michigan, USA) at a wave-length of 760 nm and the result was expressed as mg of gallic acid equivalents (GAE) per gram of sample; total soluble solids by direct reading in a digital refractometer (Quimis Q767BD, Diadema, São Paulo, Brazil); water activity (a_w) by direct reading in a digital thermohygrometer (Aqualab 4TEV, Decagon, Pullman, USA) at 25°C; and color parameter for L* (lightness), a* (redness\greenness), b* (yellowness\blueness), C* (chroma – color intensity), and h° (hue angle – values of 0°, 90°, 180°, and 270° denote pure red, pure yellow, pure green, and pure blue colors, respectively) was measured using a digital colorimeter (Konica Minolta CR-400, Osaka, Japan) calibrated with white plate. All analyses were carried out in triplicate.

2.4. Powder Morphology

Powder morphology was evaluated by Scanning Electron Microscopy (SEM) in the Nanomanipulation Laboratory (PPGF/UFPA). The samples were fixated onto stubs with conventional conductive double-sided adhesive tape and metallized with a gold/palladium allow in a mini sputter coater (Quorum Technologies SC7620, Kent, UK) using 5 mA current

for 120s. Next, the samples were analyzed in a scanning electron microscope (Tescan Vega3, Brno, Czech Republic) using electron beam current of 85-90 µA, 5 Kv acceleration voltage, approximately 15 mm working distance (WD), and magnification of 2000x.

2.5. Determination of Moisture Sorption Isotherms

The adsorption and desorption isotherms were obtained at 25°C in a Vapor Sorption Analyzer (VSA) equipment, using the DVS (*Dynamic Vapor Sorption*) method, which is based on the readings of sample mass and a_w successively until the pre-established equilibrium condition is reached. For the const-ruction of the isotherms, a sample of cupuassu powder was initially stored in a desiccator with silica gel under vacuum and at 25°C for 24 hours to ensure $a_w < 0.1$ in the sample [25]. After this step, approximately 1 g of the sample was weighed in a stainless steel capsule, in the analytical microbalance of the VSA. The data were obtained in an adsorption-desorption cycle, for a range of 0.1 to 0.9 a_w. The equilibrium condition was determined when the mass change and the time variation ($\Delta m/\Delta t$), between consecutive readings, reached a value lower than 0.05. After the analysis, the dry mass of the sample was determined in a vacuum oven at 70°C [23].

2.6. Determination of Monolayer Moisture Content

The monolayer moisture content (m_o) was calculated using the BET linear equation (Eq. **1**) [11].

$$\frac{a_w}{(1-a_w)m} = \frac{1}{m_o C} + \frac{(C-1)}{m_o C}.a_w \qquad (1)$$

where m = equilibrium moisture content (g H_2O/100 g d.b.); a_w = water activity (dimensionless), m_o = monolayer

moisture content (g H_2O/100 g d.b.); and C = constant related to the sorption heat.

2.7. Mathematical Modeling of the Sorption Isotherms

The mathematical models proposed by Halsey [13], Henderson [14], Oswin [15], Peleg [16] and GAB described by Maroulis *et al.* [12] (Table **1**) were fitted by nonlinear regression analysis to the experimental sorption data of the product, using the software Statistica 7.0.

The goodness of the fit of each model was evaluated in terms of maximum coefficient of determination (R^2), minimum standard error of estimate (SEE) (Equation 2) and minimum mean absolute percentage error (P) (Equation 3). According to Lomauro *et al.* [22], if the P value is less than 10%, the model is acceptable.

$$SEE = \sqrt{\sum_{i=1}^{N} \frac{(m_e - m_p)}{F}} \qquad (2)$$

$$P = \frac{100}{N} \sum_{i=1}^{N} \left(\frac{m_e - m_p}{m_e} \right) \qquad (3)$$

where N = number of observations; m_e = experimental value of equilibrium moisture content (g H_2O/100 g d.b.); m_p = predicted value of equilibrium moisture content (g H_2O/100 g d.b.); and F = degree of freedom of the regression model.

3. RESULTS AND DISCUSSION

3.1. Characterization of Cupuassu Powder

The cupuassu powder composition, physicochemical characterization and color parameters are presented in Table **2**.

Table 1. Mathematical models used to fit the sorption isotherms of cupuassu powder at 25°C.

Model Name	Model Equation	Parameters Number
Halsey	$m = \left[\dfrac{-a}{\ln a_w} \right]^{\frac{1}{b}}$	2
Henderson	$m = \left[\dfrac{-\ln(1-a_w)}{1-a_w} \right]^{\frac{1}{b}}$	2
Oswin	$m = a.\left[\dfrac{a_w}{1-a_w} \right]^{b}$	2
GAB	$m = \dfrac{m_o.c.k.a_w}{[(1-k.a_w).(1+(c-1).k.a_w]}$	3
Peleg	$m = k_1.a_w^{n_1} + k_2.a_w^{n_2}$	4

m = equilibrium moisture content (g H_2O/100 g d.b.); m_o= monolayer moisture content (g H_2O/100 g d.b.); a_w = water activity (dimensionless); a, b, c, k, k_1, k_2, n_1 and n_2 = model constants.

The powder presented high acidity, low moisture and low a_w, which allows the product to present greater stability to degradation processes, since, the lower availability of water and the pH limit the microbial growth [26], and consequently, prevent the deterioration of the product during the storage. Additionally, the low fat and high protein contents; besides, the presence of vitamin C and phenolic compounds are important attributes in the product. The major constituent of the product was sugar, which is attributed to the addition of maltodextrin. In turn, despite the addition of maltodextrin, the characteristic yellowish-white color of the pulp was maintained in the powdered cupuassu. In other words, the white coloration of the added maltodextrin did not promote the depreciation of the characteristic color of the product. Similar behavior was observed for Gac fruit aril powder by Kha *et al*. [27].

The scanning electromicrograph of the cupuassu powder (Fig. **1**) shows that the product presents spherical particles with some small orifices (pore). Porous microspheres affect protection of the active material and facilitate the moisture gain since the water can penetrate the pores more easily [28].

Table 2. Composition, physicochemical characterization and color parameters of cupuassu powder.

Properties	Cupuassu Powder
Moisture (%)	2.66±<0.01
Ashes (%)	1.53±0.01
Lipids (%)	0.89±0.01
Proteins (%)	5.45±0.06
Total sugars (%)	41.2±0.4
Reducing sugars (%)	24.05±0.06
AA (mg/100 g)	105.84±0.01
TPC (mg GAE/100 g)	133.8±0.3
a_w (at 25°C)	0.16±<0.01
pH	3.60±0.01
Total titratable acidity (g citric acid/100 g)	2.1±0.1
Total soluble solids (°Brix)	43.8±0.2
Color parameters	
$L*$	86.1±0.4
$a*$	-4.73±0.08
$b*$	24.5±0.2
$C*$	24.6±0.4
$h*$	100.8±0.2

AA = ascorbic acid content; TPC = total phenolic compounds; $L*$ = lightness; $a*$ = redness\greenness; $b*$ = yellowness\blueness; $C*$ = chroma; $h*$ = hue angle.
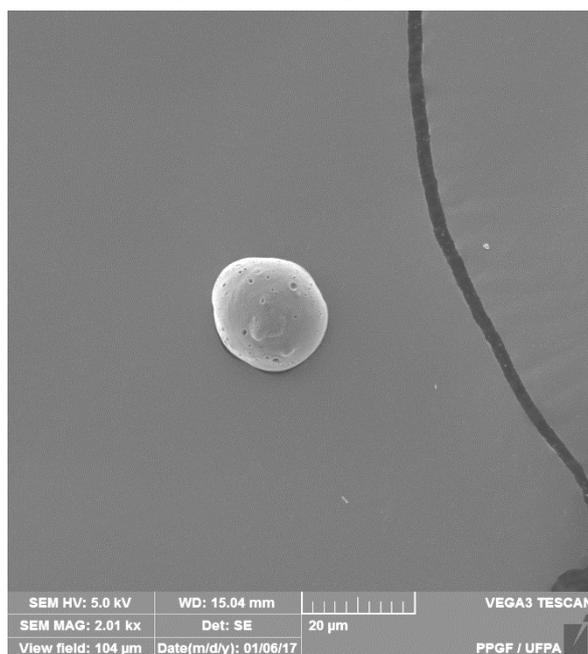
Fig. (1). Scanning electron microscopy images of cupuassu powder containing 40% maltodextrin.
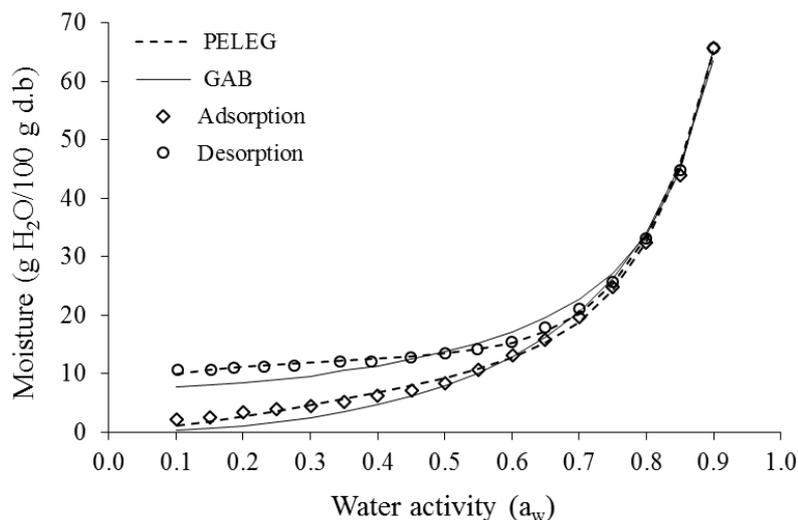
Fig. (2). Sorption isotherms of cupuassu powder at 25°C and the models of Peleg and GAB fitted to the adsorption and desorption data.

3.2. Moisture Sorption Isotherms

Fig. (**2**) shows the adsorption and desorption data for the cupuassu powder at 25°C. According to the adsorption data, the product will exhibit microbiological stability ($a_w < 0.6$) [29] if its moisture content remains below 13 g H_2O/100 g d.b., which corresponds to product moisture of 11.5 g H_2O/100 g. According to the BET equation, the moisture values equivalent to the monolayer (m_o) were 4.3 g H_2O/100 g d.b. for adsorption and 7.6 g H_2O/100 g d.b. for desorption ($R^2 > 0.98$). The m_o adsorption value indicates the highest stability moisture level of the powder product. On the other hand, based on the m_o value for the desorption process, it is recommended not to dry the cupuassu pulp added of 40% maltodextrin at moisture levels below 7.6 g H_2O/100 g d.b, to avoid unnecessary energy loss. Since, at this moisture level, the a_w of the product will be less than 0.6, so its microbiological stability will be ensured.

The sorption isotherm presented type III behavior, which is characteristic of products rich in sugars, according to the classification of Brunauer *et al*. [11]. This is attributed to the presence of total sugars as the major constituent of the product, which is especially related to the addition of maltodextrin. Similar behavior was reported previously in atomized products as syrup powder [30], strawberry powder [31] and orange juice powder [32], all using the maltodextrin as a drying agent. According to Ghorab *et al*. [33], MSI of amorphous materials such as maltodextrin often show type II or type III profiles.

The adsorption isotherm showed a linear behavior until 0.5 a_w level, and from this point, it assumed an exponential behavior with a progressive increase of the product's moisture content. According to this behavior, the product requires greater care, when stored and/or handled in environments with RH above 50%, since it is more susceptible to humidification, and therefore, more favorable to degradation processes (caused by undesirable reactions) and to microorganisms' growth. Thus, to avoid excessive moisture gain, it is necessary the use of impermeable packaging or packaging with low water vapor permeability.

Another feature of the product is the presence of bioactive compounds such as vitamin C (105.84 mg/100 g) and total phenolics (133.8 mg GAE/100 g), which are very susceptible to oxidative processes. Therefore, to minimize such processes, it is strongly indicated that the packages also have imperme-ability to air and does not allow light to pass through. In this context, Polyethylene Terephthalate with Aluminum foil and Low-Density Polyethylene (PET/Al/LDPE) film presents as an ideal packaging material for the product [34].

A type of packaging used to store powder juice presents 70 mm × 70 mm dimensions and holds 25 g of the product. Using these parameters, the equation proposed by Costa *et al*. [35] was used to estimate the storage time required for the product to reach a critical moisture level (13 g H_2O/100 g d.b. at a_w = 0.6). It was considered the minimum value of water vapor permeability of 0.01 g H_2O/m^2.day for a PET/Al/LDPE film with 86 μm thickness, being 11 μm of PET, 9 μm of Al and 66 μm of LDPE, when exposed to an environment at 38°C and RH of 90% [34]. On the regarded conditions, the estimated storage time for the cupuassu powder with 3% moisture is over two years.

Fig. (**2**) also shows the beginning of hysteresis effect at the capillary condensation region ($a_w \approx 0.7$), which extends to the monolayer region ($a_w \approx 0.1$). However, the hysteresis loop did not close in this region, which can be attributed to loss of flowability and crystallinity of the product during the adsorption process. The addition of maltodextrin increases the glass transition temperature (T_g) of the product and generally tends to form amorphous solids. On the other hand, the increase in moisture content decreases the T_g of the product due to the water plasticizing effect. The amorphous solids can undergo physical changes such as stickiness, collapse, caking, tackiness, agglomeration, crystallization and compaction dur-ing processing, handling and storage. These physical changes in dehydrated products are related to their T_g. Below the T_g, the food is expected to be stable, whereas above this temperature, the rate of physical, chemical and biological changes of the product increases with the storage temperature [33, 36, 37].

Table 3. Estimated parameters and performance criteria of the models fitting to the moisture sorption data of cupuassu powder at 25°C.

Isotherm	Model	Parameters	R^2	SEE	P (%)
Adsorption	Halsey	a = 6.08	0.99	0.91	11.4
		b = 0.96			
	Henderson	a = 0.21	0.99	1.58	25.9
		b = 0.58			
	Oswin	a = 9.24	0.99	0.46	7.9
		b = 0.89			
	GAB	m_o = 7.99	0.99	0.46	8.3
		c = 1.44			
		k = 0.99			
	Peleg	k_1 = 23.17	0.99	0.70	10.1
		n_1 = 1.34			
		k_2 = 118.32			
		n_2 = 9.15			
Desorption	Halsey	a = 20.49	0.96	2.75	16.8
		b = 1.27			
	Henderson	a = 0.08	0.86	5.51	32.8
		b = 0.83			
	Oswin	a = 14.33	0.93	4.49	23.5
		b = 0.66			
	GAB	m_o = 6.48	0.99	0.93	5.9
		c = -22.39			
		k = 1.0			
	Peleg	k_1 = 14.83	0.99	0.53	2.3
		n_1 = 0.18			
		k_2 = 120.58			
		n_2 = 8.23			

3.3. Moisture Sorption Isotherms Modeling

The coefficient of determination (R^2), standard error of estimate (SEE) and mean absolute percentage error (P), as well as the coefficients of the mathematical models fitted to the moisture sorption data of the powdered product at 25°C are shown in Table **3**. According to the results, the Peleg and GAB models can predict with good precision the MSI of the cupuassu powder (Fig. **2**), since the R^2 maximum, SEE minimum and P ≤ 10% indicated the best fit to both adsorption and desorption data of the product. Comparable results were observed by Farahnaky *et al*. [30] for MSI of the powdered syrup. Several studies report the higher efficiency of the GAB model on predicting the MSI of powdered fruits, for example, a commercial cupuassu powder without drying agent [38], strawberry pulp powder [31], mango pulp powder [39], yellow mombin pulp powder [40], tamarind pulp powder [9], among others. In turn, there are few reports of the Peleg model application in the MSI prediction of powdered fruits. According to Silva *et al*. [41], the Peleg model resulted in better fits to the experimental data of umbu-cajá powder.

CONCLUSION

The cupuassu powder containing 40% maltodextrin presents type III sorption isotherms. The hygroscopic evaluation indicated that the product should preferably be stored in an environment with an RH below 50% and while the product moisture remains below 11.5%, it presents microbiological stability. Additionally, the monolayer values, for adsorption, indicate that the moisture level of greater product degradation stability is 4.3%, while the desorption isotherm indicates that the cupuassu pulp should not be dried at moisture content lower than 7.6%, to avoid unnecessary energy loss. It is recommended that the cupuassu powder should be stored in a packaging impermeable to water vapor and air, which does not allow the light to pass through; preferably under vacuum or nitrogen atmosphere to minimize the moisture gain and oxidation of the bioactive compounds. The Peleg and GAB models can accurately predict the adsorption and desorption isotherms of the powder product studied.

LIST OF ABBREVIATIONS

SEM	=	Scanning Electron Microscopy
PET	=	Polyethylene Terephthalate
LDPE	=	Low-Density Polyethylene
SEE	=	Standard Error of Estimate

ETHICS APPROVAL AND CONSENT TO PARTICIPATE

Not applicable.

HUMAN AND ANIMAL RIGHTS

No animals/humans were used for studies that are the basis of this research.

CONSENT FOR PUBLICATION

Not applicable.

CONFLICT OF INTEREST

The authors declare no conflict of interest, financial or otherwise.

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