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Hygroscopic Behavior of Lyophilized Powder of Grugru Palm (*Acrocomia aculeata*)

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ABSTRACT

The objective of this study was to determine adsorption isotherms and hygroscopic behavior of lyophilized powder from grugru palm. The powders of grugru palm were obtained by lyophilization process without maltodextrin (T1) and with 8% matodextrin (T2). The experimental data were obtained through the static gravimetric method at temperatures (25˚C, 30˚C, 35˚C and 40˚C), with different saturated solutions of salts. The models of GAB, BET, Henderson and Oswin were fitted to experimental data. The values obtained for hygroscopicity were 7.68% and 6.86% and the degrees of caking were 0.33% and 0.09% for T1 and T2, respectively. Mathematical models of adsorption isotherms for grugru palm powders can be classified as Type III. The GAB and Oswin models represented better the behavior of isotherms for T1 and T2. Grugru palm powder showed an increase in the humidity of the monolayer X_m along with increasing temperature. The grugru palm powder demonstrated to be a non-hygroscopic product, non-caking features.

Keywords: *Acrocomia aculeate*; Adsorption Isotherms; Hygroscopicity; Degree of Caking

1. Introduction

Grugru palm (*Acrocomia aculeata* (Jacq.) Lodd. ex-Mart.) is native to the grasslands, savannas and open forests of Tropical America and occurs in dense populations in some localities [1].

It presents a fresh pulp, which is consumed fresh or used in the production of flour that can be utilized in different foods; it has a sweet taste and is mucilaginous. This fruit can be eaten fresh or cooked, and used in soft drinks and ice creams [2].

Ramos *et al.* [3] demonstrated the potential of grugru palm pulp as a nourishing food, able to contribute to the enrichment of the regional diet in supplemental feeding programs, and a natural source of β-carotene, vitamin A and minerals as copper, potassium and zinc.

Dehydration of fruits and food is used as a method of preservation and storage, providing a new product on the market, which has motivated the investments in agricultural production and processing [4,5].

According to Toneli *et al.* [6], foods in storage period are exposed to various relative humidities and to different conditions of temperature; so it is necessary to adjust the moisture in order to reach equilibrium with the environment. These authors observed physical changes (caking) in inulin powder when there is humidity variation.

Mathlouthi and Rogé [7] stated that to explain the behavior of different insoluble food products, several mathematical models were proposed for sorption isotherms. Studies by Kaymak-Ertekin and Gedik [8] for sorption isotherms in apples and potatoes showed an increase in temperature, resulting in decrease of equilibrium for moisture content.

The understanding of moisture sorption isotherms in food has great importance in food science and technology for many uses, such as design and optimization of industrial processes, e.g., drying, assessing the problems of packaging, modeling of moisture changes that occur during drying, forecasting the stability of shelf life and predicting mixtures of ingredients [9].

Obtaining grugru palm powder is still handmade, and studies are necessary to show the best techniques for obtaining and storing this product. Given the above, the objective of this work was to evaluate the hygroscopic

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behavior and the application of mathematical models in predicting the adsorption isotherms of lyophilized powders from grugru palm.

2. Material and Methods

2.1. Raw Material

Grugru palm fruits were harvested in Araripe Plateau in the Cariri Region, Ceará State, Brazil, and taken to the Laboratory of Food Quality Control and Drying (UFCE), which were selected and sanitized and subsequently peeled for pulp separation. The pulp was stored at −20˚C until the lyophilization process.

Two lyophilized powders of grugru palm were used: one treatment without addition of drying adjuvant and other with maltodextrin of dextrose equivalent (DE) 20.

Control treatment (T1)—powder without maltodextrin Treatment with maltodextrin (T2)—powder with addition of maltodextrin 8%.

2.2. Lyophilization

The pulp was unfrozen and triturated; after this process the pulp of T1 and T2 were spread on stainless steel trays with diameter of 15 cm. The trays were placed in ultra-freezer (CL 90 - 40 V, Terroni) at −40˚C for 24 hours; after this period the pulp was taken to a bench lyophilizer (LS 3000, Terroni) for 25 hours. Right after the lyophilization, the dried product was taken to a rotating-knives mill (MA 048, Marconi) in order to get a powder with homogeneous granularity.

2.3. Modeling of Adsorption Isotherms

In determining the moisture adsorption isotherms, a static gravimetric method described by Wolf *et al.* [10] was applied using saturated solutions of salts according to Greenspan [11], at room temperature of 25˚C ± 2˚C. The solutions of salts were prepared and put in containers of tempered glass closed with silicone.

Measurements of adsorption isotherms were performed in triplicate. For each cell about 0.20 g of each treatment were weighed in pre-librated aluminum crucibles. Afterward, the crucibles were taken to cells, which contained the saturated solutions of salts.

The process was monitored by weighing of samples on analytical balance every 24 hours to obtain equilibrium. After balance the water activity of the samples was determined at temperatures of 25˚C, 30˚C, 35˚C and 40˚C, using a_w meter, model AQUALab 4TEV. Then they were taken to a lab chamber with air circulation at 105˚C for determination of moisture content.

The equilibrium moisture (X_{eq}, Equation (1)) was calculated as the difference between the mass of balanced sample and the dried mass:

$$X_{eq} = \frac{m_{eq} - m_s}{m_s} \quad (1)$$

where: X_{eq} = equilibrium moisture (db); m_{eq} = mass of balanced sample (g); m_s = mass of dried sample (g).

For adjusting of the experimental data from adsorption isotherms of grugru palm powder, mathematical models of GAB (Equation (2)), BET (Equation (3)), Henderson (Equation (4)) and Oswin (Equation (5)) were used, represented respectively by the equations in **Table 1**. The calculations of parameters of each model were performed using the software Statistic version 7.0 [12].

The quality of adjusting different models was evaluated through the best values of the determination coefficient (R^2) and the relative mean deviation (E%, Equation (6)), defined by Iglesias and Chirife [17]:

$$E\% = \frac{100}{n}\sum_{i-1}^{n}\frac{\left|Xeq_e - Xeq_p\right|}{Xeq_e} \quad (6)$$

where: E% = relative mean error; Xeq_e = experimental

Table 1. Mathematical models used to adjust the experimental data of adsorption isotherms.

Models	Equations	
GAB (Guggenheim-Andersen-de Boer) [10]	$X_{eq} = \frac{X_m \cdot C \cdot K \cdot a_w}{(1-)K \cdot a_w \cdot (1 - K \cdot a_w + C \cdot K \cdot a_w)}$	(2)
BET [11]	$X_{eq} = \frac{X_m \cdot C \cdot a_w}{1 - a_w}\left[\frac{1-(n+1)\cdot(a_w)^2 + n\cdot(a_w)^{n+1}}{1-(1-C)\cdot a_w - C\cdot(a_w)^{n+1}}\right]$	(3)
Henderson [12]	$X_{eq} = \left[\frac{-\ln(1-a_w)}{b}\right]^{\frac{1}{a}}$	(4)
Oswin [13]	$X_{eq} = a \cdot \left[\frac{a_w}{1-a_w}\right]^b$	(5)

*a_w = water activity; X_m = moisture content in the molecular monolayer (g $H_2O \cdot g^{-1}$ dry basis); X_{eq} = equilibrium moisture content (g $H_2O \cdot g^{-1}$ dry basis); C = constant related to heat of sorption of the molecular layer; a, b, K = adjusting parameters.

values; Xeq_p = values predicted by the model; n = number of experimental data.

2.4. Hygroscopicity

The analysis was performed using the methodology A14a, described by GEA Niro Research Laboratory [18], consisting in powder exposure to air relative humidity (RH) of 79.5%, which was adsorbed through the powder sample until a constant weight is reached. The calculation of hygroscopicity is given by Equation (7). The parameters used to characterize the hygroscopicity of grugru palm powder were obtained according GEA Niro Research Laboratory [18].

$$\%\ \text{Hygroscopicity} = \frac{(\%WI + \%FW)\times 100}{100 + \%WI} \quad (7)$$

where: $\%FW$ = % free water;
$\%WI = ((c-b)/(b-a))\times 100$; a = weight of plate (g); b = weight of plate + powder (g); c = weight of plate + powder in equilibrium (g).

2.5. Degree of Caking

The analysis was performed through the methodology A15a, described by GEA Niro Research Laboratory [18], which consists of exposing the powder to absorb moisture from the air (79.5% relative humidity) until reaching equilibrium. Afterward, the powder was dried and sieved under standard conditions (sieve mesh modified to 1200 μm). What was left in the sieve was expressed as degree of caking. The calculation of the degree of caking is given by Equation (8). The parameters used to characterize the degree of caking of grugru palm powder were obtained according to GEA Niro Research Laboratory [18].

$$\%Degree\ of\ Caking = b\times 100/a \quad (8)$$

where: a = grams of powder used; b = grams of powder retained in the sieve.

2.6. Statistical Analysis

Data were analyzed statistically through analysis of variance (ANOVA), and differences between the averages for hygroscopicity and degree of caking were determined by Tukey test at 5% probability using the software *Statistic* version 7.0 [12].

3. Results and Discussion

3.1. Adsorption Isotherms

The adsorption isotherms of lyophilized powders from grugru palm fruits showed a behavior characteristic to Type III, according to Brunauer classification [19]. It is can be observed in **Figures 1** and **2** that the values of the experimental data for equilibrium moisture (X_{eq}) of lyophilized grugru palm powders raised in accordance to increasing water activity, under conditions of constant temperature, e.g. at 25˚C shown in Figures cited.

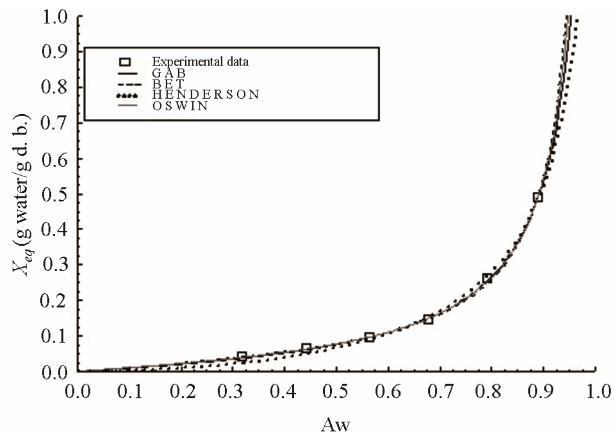

Figure 1. Adsorption isotherms at 25˚C of lyophilized powder of grugru palm without addition of maltodextrin (T1).

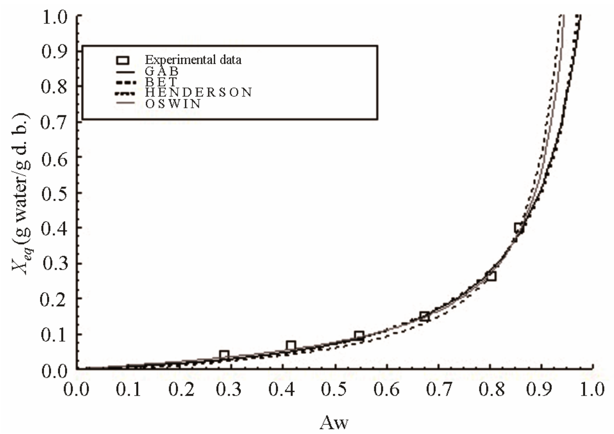

Figure 2. Adsorption isotherms at 25˚C of lyophilized powder of grugru palm with addition of 8% maltodextrin (T2).

The parameters of the models applied to experimental data of adsorption isotherms of lyophilized powders of grugru palm, the determination coefficients (R^2) and the relative average deviations (E%) used as evaluation criteria for the representation of isotherms are shown in **Table 2**.

According to the results presented in **Table 2**, one notes that among the models studied (GAB, BET, Henderson and Oswin) all can represent the behavior of grugru palm powders because they showed representative R^2 e E%, respectively, from 0.9843% to 0.9996% and 0.23% to 1.57%, values below the criteria recommended by Aguerre *et al.* [20], where E% less than 10% indicates reasonable representative models, and by Labuza *et al.* [21], in which the representation of isotherms is considered extremely good for E% less than 5%. However, the GAB model (triparametric) represents more accurately the T1 treatment and the Oswin model (bi-parametric)

Table 2. Adjustment results of experimental data of adsorption isotherms at 25˚C, 30˚C, 35˚C and 40˚C for lyophilized powders of grugru palm.

Models	Parameters[c]	T1[a]				Models	Parameters[c]	T2[b]			
		25˚C	30˚C	35˚C	40˚C			25˚C	30˚C	35˚C	40˚C
GAB	X_m	0.0705	0.0709	0.0825	0.1027	GAB	X_m	0.1122	0.1166	0.1156	0.0981
	C	1.2663	1.2072	0.9623	0.6949		*C*	0.5982	0.5626	0.5596	0.7402
	K	0.9791	0.9925	0.9886	0.9796		*K*	0.9278	0.9369	0.9459	0.9822
	R^2	0.9996	0.9993	0.9992	0.9996		R^2	0.9897	0.9900	0.9904	0.9965
	E%	0.23	0.31	0.40	0.29		*E*%	1.13	1.24	1.16	0.69
BET	X_m	0.0755	0.2301	0.0690	0.1579	BET	X_m	0.2241	0.1259	0.1387	0.1524
	C	1.4186	0.2770	1.3286	0.4451		*C*	0.3314	0.6404	0.5478	0.4968
	n	1.3371	-0.6423	1.0522	1.1523		*n*	-0.7423	1.3501	1.3105	1.2144
	R^2	0.9988	0.9916	0.9991	0.9994		R^2	0.9859	0.9961	0.9954	0.9976
	E%	0.39	1.39	0.40	0.34		*E%*	1.39	0.76	0.79	0.58
Henderson	*a*	0.5836	0.5591	0.5533	0.5450	Henderson	*a*	0.6155	0.6047	0.5942	0.5873
	b	3.3685	3.1554	3.0070	2.8519		*b*	3.5360	3.3701	3.2653	3.0546
	R^2	0.9951	0.9940	0.9950	0.9966		R^2	0.9843	0.9833	0.9828	0.9883
	E%	1.01	1.08	0.93	0.78		*E*%	1.45	1.62	1.57	1.29
Oswin	*a*	0.0773	0.0766	0.0801	0.0827	Oswin	*a*	0.0756	0.0775	0.0780	0.0837
	b	0.8859	0.9421	0.9687	1.0099		*b*	0.9120	0.9417	0.9647	0.9962
	R^2	0.9995	0.9993	0.9991	0.9993		R^2	0.9961	0.9958	0.9954	0.9979
	E%	0.26	0.32	0.40	0.31		*E*%	0.63	0.79	0.79	0.54

[a]T1: lyophilized powder without addition of maltodextrin; [b]T2: lyophilized powder with addition of 8% maltodextrin. [c]Abbreviations: R^2 = detemination coefficient. *E*% = relative average error. X_m = moisture content in the monolayer (g $H_2O \cdot g^{-1}$ dry basis). C = constant related to the heat of sorption in the molecular layer. K = GAB constant related to multi-layers. n = BET constant related to multi-layers. *a e b* = adjusting parameters of the Henderson and Oswin models.

the T2. Oliveira *et al.* [22] also obtained better adjustments of GAB model and Oswin model for lyophilized sapodilla.

The GAB model is reported by several researchers in forecasting adsorption isotherms for various dehydrated foods, as Ferreira and Pena [23] in obtaining adjustment of the model for peach palm powder; Silva *et al.* [24], for yellow mombin powder; Kaymak-Ertekin and Gedik [8] for dehydrated grapes and potatoes; and Goula *et al.* [25], who obtained the best prediction for tomato powder.

The Oswin model did not fit to mathematic modeling in predicting the behavior of foods studied by Goula *et al.* [25] and Al-Muataseb *et al.* [26]. This report does not fit to that observed in T2 in this study, which showed R^2 = 0.9961 to 0.9979 and *E*% = 0.63 to 0.79. Kaymak-Ertekin and Gedik [8] also observed that the Oswin model presented data well correlated for apples and potatoes. Lomouro *et al.* (1985), quoted by Al-Muataseb *et al.* [27], reported that the Oswin model accounted for only 57% of the isotherms described for foods.

The values of moisture in the monolayer (X_m) in the GAB model for T1, between the temperatures of 25 and 40˚C, has raised with increasing temperature (**Table 2**). Similar behavior was observed in the determination of adsorption isotherms of flour of peach palm (*Bactris gasipaes*) at 15˚C and 35˚C (23) for Surinam cherry powder at temperatures of 20˚C and 40˚C [28].

The values of water content in the monolayer with increasing temperature of T1 varied from 0.07 to 0.10 g water g^{-1} dry basis, and in T2 it was 0.07 to 0.08 g water g^{-1} dry basis; values of T2 has the same trend observed in the range presented by Rahman (1995), quoted by Cova *et al.* [29], which is from 0.05 to 0.08 g water g^{-1} dry basis for water content in the monolayer of starch rich products. The addition of maltodextrin in T2 may be the explanation for this treatment because it stands in the profile of starch rich products. However, Talla *et al.* [30] found values for banana, mango, pineapple between 0.080 and 0.185 g water g^{-1} dry basis, values higher than previously reported.

The values of *K* in this study were inferior to 1.0 at all temperatures, presenting little variation between 0.97 and 0.99. Vieira *et al.* [28], quoting Fernandez (1995), reported that values of *K* lower than 1.0 show a characteristic indicating that the isotherm for food products tends to an asymptote when activity equals to 1.0.

One can observe that increasing temperature from 25˚C to 40˚C leads a reduction in values of the constant *C*; a compatible behavior was observed by Gabas *et al.* [31] for pineapple powder with addition of maltodextrin, which demonstrates temperature is dependent to the constant *C*. These same authors reported that strong adsorbate-adsorbent interactions are favored by lower temperatures, reflecting on *C* increase and showing that this variation along with temperature may be the result of a mathematic compensation between *C* and *K*.

In **Figures 3** and **4**, there is a graphical representation of the adsorption isotherms of moisture of grugru palm powder, for all temperatures studied, adjusted by GAB and Oswin model for T1 and T2, respectively.

There is little influence of temperature on equilibrium moisture content of grugru palm powder for water activity below 0.55 in T1, being observed from this point that for the same a_w there is an increase in the equilibrium moisture content along with rising temperatures (**Figure 3**). In T2, this effect was observed from water activity above 0.50 (**Figure 4**). In studies carried out with Surinam cherry powder, it was observed an influence of temperature on the equilibrium moisture content from a_w above 0.30 [28]; this shows that grugru palm powder presents a hygroscopicity below that observed in Surinam cherry powder.

As may be observed, the curves of **Figures 3** and **4** do not intersect themselves with the increasing temperature, a fact also reported by Kaymak-Ertekin and Gedik [8] for apples with high content of pectin and sugar, and for potatoes with high content of starch. This similar behavior of grugru palm powder to data of the previously cited authors is due to its sweet fruit with approximately 35% carbohydrates [32], being 13.3% glucose [33].

3.2. Hygroscopicity and Degree of Caking

Table 3 shows the values of hygroscopicity and degree of caking for T1 and T2. Treatment T1 presented greater caking in relation to T2. This fact may be related to the addition of drying adjuvant, maltodextrin, in which its use entails a reduction in the powder hygroscopicity; this fact is also evidenced in adsorption isotherms of this work.

Powder caking is an undesirable phenomenon, which initially consists of transformation of powder into solid and sticky material, resulting in decreased functionality and fluidity, so quality loss, and the main cause of agglomeration is the presence of water induced by plasticization of the surface of particles [34].

In accordance with GEA Niro Research Laboratory [18], in which is characterized the hygroscopic behavior and the degree of caking, respectively, the grugru palm powder is classified as a non-hygroscopic and non-caking product, Oliveira *et al.* [35] showed similar response about this classification (hygroscopicity 5.17% and 6.39%; degree of caking 0.03% and 3.11%) for powder grugru palm obtained by oven dryer.

In studies carried out with sucrose, Mathlouth and Rogé [7] reported the formation of agglomerates is related to the solid bridges created during the process, which turns out sucrose more hygroscopic. The same can be observed in T1, where the powder formed the greatest caking and consequently making the grugru palm powder more hygroscopic.

Costa *et al.* [36] stated that different variations in the hygroscopic behavior of food powders are also attributed to their size, as finer particles have higher contact surface and therefore higher number of active sites. This report may explain the non-hygroscopicity behavior of grugru palm powder when compared to that of fruits found in the literature, which showed high hygroscopicity, as the particle size of grugru palm powder is 1200 μm and the particle size of most fruit powders is in the range from 500 to 600 μm.

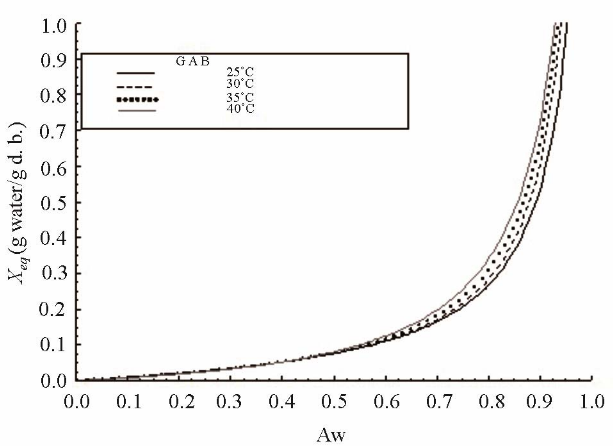

Figure 3. Best model (GAB) for adsorption isotherms at different temperatures of lyophilized grugru palm powder without maltodextrin (T1).

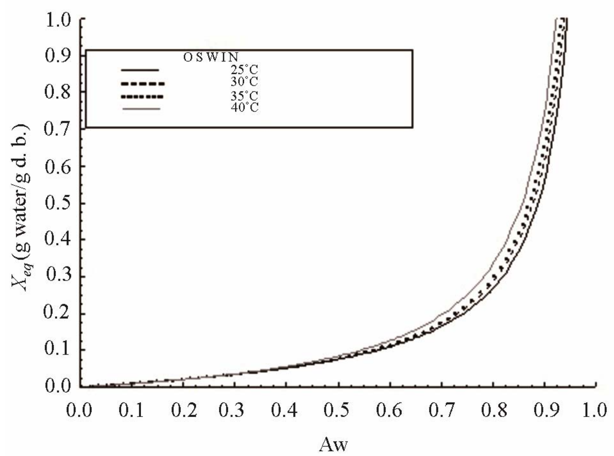

Figure 4. Best model (Oswin) for adsorption isotherm at different temperatures of lyophilized grugru palm powder with 8% maltodextrin (T2).

Table 3. Hygroscopicity and degree of caking of lyophilized powders from grugru palm.

Treatment[a]	Hygroscopicity (%)	Degree of caking (%)
T1	7.68 a[*]	0.33 a
T2	6.86 a	0.09 b

[a]T1 = lyophilized powder without maltodextrin; T2 = lyophilized powder with 8% maltodextrin. [*]Equal lower-case letters in the same column do not differ by Tukey test at 5% probability.

4. Conclusions

Mathematical models of adsorption isotherms for grugru palm powders can be classified as Type III, according to Brunauer classification.

All models studied adjust themselves to lyophilized powder of grugru palm, and GAB and Oswin models represent best the behavior of the adsorption isotherms for T1 and T2, respectively.

Grugru palm powder showed an increase in the humidity of the monolayer X_m along with increasing temperature. The values of C were reduced along with increasing temperature. The constant K suffered small variations in function to temperature.

Lyophilized powders of grugru palm were characterized as non-hygroscopic and non-caking product.

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