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Hygroscopic behavior of lyophilized acerola pulp powder

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A B S T R A C T

Powder products are characterized by their practicality and long life. However, fruit powders have high hygroscopicity and tend to agglomerate due to its hydrophilic nature. The isotherms of equilibrium moisture content apply to the study of dehydrated food preservation potential. Acerola is a nutritionally rich fruit, with great economic and industrial potential. The objective of this study was to analyse acerola powder adsorption isotherms obtained by lyophilization and characterize the powder obtained from lyophilized acerola pulp. Analysis of hygroscopicity, solubility and degree of caking were performed. Isotherms were represented by the mathematical models of GAB, BET, Henderson and Oswin, at temperatures of 25, 35 and 45 °C. According to the results, the obtained powder showed hygroscopicity of 5.96 g of absorbed water $100g^{-1}$ of solids, solubility of 95.08% and caking of 14.12%. The BET model showed the best fit to the adsorption isotherms of the acerola pulp powder obtained by lyophilization. The obtained isotherm was of type III, with a "J" shape. There was an inversion of the effect of temperature on the isotherms of acerola powders.

Palavras-chave:
Malpighia glabra
higroscopicidade
secagem

Comportamento higroscópico do pó da polpa de acerola liofilizada

R E S U M O

Produtos em pó são caracterizados pela sua praticidade e vida útil longa; no entanto, os pós de fruta apresentam alta higroscopicidade e tendência à aglomeração devido à sua natureza hidrofílica. As isotermas de equilíbrio higroscópico se aplicam para estudo do potencial de conservação de alimentos desidratados. A acerola, por sua vez, é um fruto rico nutricionalmente e de grande potencial econômico e industrial. O objetivo do presente trabalho foi analisar as isotermas de adsorção do pó de acerola obtido por liofilização e caracterizar o pó obtido a partir de polpa de acerola liofilizada. Foram feitas as análises de higroscopicidade, do grau de caking e da solubilidade. Para representação das isotermas foram utilizados os modelos matemáticos de GAB, BET, Henderson e Oswin nas temperaturas de 25, 35 e 45 °C cujos resultados mostraram que o pó obtido apresentou higroscopicidade de 5,96 g de água absorvida $100g^{-1}$ de sólidos, solubilidade de 95,08% e caking de 14,12%. O modelo de BET foi o que melhor se ajustou às isotermas de adsorção do pó de polpa de acerola obtido por liofilização. A isoterma obtida foi do tipo III em formato de "J"; observou-se a ocorrência de uma inversão do efeito da temperatura sobre as isotermas dos pós de acerola.

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Introduction

Acerola (*Malpighia glabra*) is a tropical fruit with high contents of vitamin C, besides carotenoids and anthocyanins, which are compounds that capture free radicals in the human organism, standing out as a functional food with great economic and nutritional potential (Mesquita & Vigoa, 2000). On the other hand, acerola has easy cultivation, pleasant taste and aroma and allows wide industrial use, making it possible the manufacturing of many products (Freitas et al., 2006).

The problem in the acerola sector is the high perishability of the fruits after harvest and during the commercialization. Thus, drying is one of the processes available for the application in the industry of fruit pulps, concentrating the principles of raw material and making it possible to store the product for long periods under conditions of transport, storage and adequate package, in order to maintain physical and chemical characteristics of the product obtained by the drying (Gomes et al., 2004).

Drying is the removal of water from the material promoted by the difference of water vapor pressure between the drying medium and the surface of the product, causing the transfer of mass of the product (Park et al., 2008). Most methods of food drying involve the application of heat; however, in lyophilization, water is removed through sublimation.

According to Gomes et al. (2002), the powder produced by the drying of fruit pulps results in a material with characteristics different from those of the pulp, requiring the analysis of its properties. These products are characterized by having high contents of soluble solids and low-molecular-weight sugars. These sugars have hydrophilic nature and, along with an amorphous state, may lead to undesirable effects such as high hygroscopicity and tendency to agglomerate at temperatures above the vitreous transition temperature. Hygroscopicity is the capacity of the food powder to absorb water from an environment with relative humidity superior to that of equilibrium (Jaya & Das, 2004; Carlos et al., 2005). The tendency for agglomeration is an effect attributed to the absorption of water on the surface of the particles, forming a saturated solution, thus making particles sticky and able to form hydrogen bridges, causing the phenomenon of caking (Goula & Adamopoulos, 2008). Hygroscopic equilibrium isotherms are one of the most important pieces of information for powder fruit products (Anselmo et al., 2006). The isotherm is a curve that describes, at certain temperature, the equilibrium relationship of an amount of water sorbed by components of the biological material and the vapor pressure or relative humidity. Many authors developed models for the adjustment of sorption isotherms aiming to predict the behavior of the isotherms through structural, dynamic and thermodynamic aspects of the water, translated into theories and equations that are mostly empirical, among which the BET theory is currently the most complete (Park et al., 2008).

One of the main applications of isotherm equations is establishing the bonding energy, adsorption energy and the molecular monolayer of water, which indicate the relationship of the water with the chemical reactions that determine the deterioration of biological materials, besides allowing the study of the potential of conservation of dehydrated foods, establishing an adequate project of permeable packages and defining the moisture content that results in longer shelf life etc. (Park et al., 2008).

This study aimed to analyse the adsorption isotherms of acerola powder obtained through lyophilization using the adjustment of mathematical models and characterize the powder obtained by the lyophilization of acerola pulp.

Material and Methods

The acerola pulps utilized in the experiments were obtained from the market in Fortaleza-CE, collected in the commercial package (polyethylene) containing 100 g and maintained at -18 °C until the beginning of the experiment.

Drying was performed in a lyophilizer (TERRONI®, Model LS3000), in which the samples, previously frozen until -38 °C for 24 h, were dehydrated for 24 h. The product obtained from the lyophilization was transferred to a plastic package, which was sealed and manually compressed in order to the powder product. The material was accommodated in structured packages made of Pet/Aluminum/Polyethylene materials with grammage of 122 g m^{-2}. Maltodextrin with dextrose equivalent (DE) of 20 in the proportion of 19.1% was used as a drying adjuvant in acerola pulp formulation.

The following analysis were performed in the characterization of the powders: hygroscopicity, determined according to Goula & Adamopoulos (2008) under conditions of 24 °C and 75% relative humidity, using saturated solution of NaCl, and degree of caking, calculated according to Goula & Adamopoulos (2008). Solubility was determined according to Cano-Chauca et al. (2005). All the analysis were performed in triplicate.

The adsorption isotherms of the powders were determined in triplicate, by weighing 1 g of powder samples in aluminum crucibles, previously tared in an oven at 105 °C. Then, the samples were placed in closed cells containing saline solutions in order to condition the internal environment with moisture content values, according to Greespan (1977). The cells containing saturated saline solutions and the determination of isotherms were subjected to temperatures of 25, 35 and 45 °C, as shown in Table 1.

The cells containing the samples were taken to the oven with temperature control, where they remained until constant mass, determined by weighing every 24 h until variation lower than 1%. After the equilibrium at each studied temperature, water activity in the samples was determined using a water activity meter (AQUALab 4 TEV).

The equilibrium moisture (X_0) was calculated according to Eq. (1), which was also used by Moreira et al. (2013):

Table 1. Saturated saline solution and water activity of the cell according to the temperature

Saturated saline solutions	Temperature (°C)		
	25	35	45
CH_3COOK	0.2598	0.2651	0.2730
K_2CO_3	0.4774	0.4556	0.4495
NaBr	0.5896	0.5248	0.4596
$SnCl_2$	0.7474	0.7396	0.7333
KCl	0.8429	0.8198	0.7482
$BaCl_2$	0.8853	0.8512	0.8310

$$X_0 = \frac{M_{eq} - M_s}{M_s} \tag{1}$$

where:

X_0 - equilibrium moisture, g g^{-1};
M_{eq} - mass of sample in equilibrium, g; and
M_s - mass of dried sample, g.

The mathematical adjustment of the experimental data of the powder isotherms was performed using the models of GAB (Gugghenein, Anderson & De Bôer) BET (Brunauer-Emmett-Teller) Henderson and Oswin, represented by the Eqs. (2) to (5):

- GAB:

$$X_{eq} = \frac{X_m \cdot C \cdot K \cdot a_w}{(1 - K \cdot a_w) \cdot (1 - K \cdot a_w + C \cdot K \cdot a_w)} \tag{2}$$

- BET:

$$X_{eq} = \frac{X_m \cdot C \cdot a_w}{(1 - a_w)} \cdot \left[\frac{1 - (n+1) \cdot (a_w)^n + n \cdot (a_w)^{n+1}}{1 - (1 - C) \cdot a_w - C \cdot (a_w)^{n+1}}\right] \tag{3}$$

- Henderson:

$$_{eq} \quad \left[\frac{-\ln(1 - a\ \ )}{\ }\right]^{-} \tag{4}$$

- Oswin:

$$X_{eq} = a \cdot \left[\frac{a_w}{1 - a_w}\right]^b \tag{5}$$

where:

X_{eq} - equilibrium moisture, g H_2O g^{-1};
X_m - water content in the molecular monolayer, g H_2O g^{-1};
a_w - water activity;
n - number of molecular layers;
C, K - sorption constants; and
a, b - adjustment parameters.

The adjustments were performed using the program Statistica version 7.0. The models were evaluated considering the magnitude of the adjusted coefficient of determination (R^2) and mean relative error (E), predicted by the Eq. (6) used by Rodovalho (2008).

$$E = \frac{100}{n} \sum_{i=1}^{n} \frac{|(M_i - Mp_i)|}{M_i} \tag{6}$$

where:

E - mean relative error, %;
M_i - values obtained experimentally;
M_{pi} - values predicted by the model; and
n - number of experimental data.

Results and Discussion

The obtained results for the characterization of lyophilized acerola pulp powder are shown in Table 2. The hygroscopicity of the powder obtained by lyophilization showed values of 5.96%. Oliveira et al. (2014) obtained hygroscopicity of 12.53% for whole lyophilized yellow mombin and 8.51% when maltodextrin was added. Tonon et al. (2009) claim that maltodextrin reduces the hygroscopicity of dehydrated products because of its low hygroscopicity. The fruit powders obtained through lyophilization from juices and pulps are characterized by high hygroscopicity, which consists in the capacity of the food powder to absorb water from an environment with relative humidity higher than that of equilibrium (Jaya & Das, 2004; Carlos et al., 2005). This characteristic is due to the fact that fruit powders have high contents of soluble solids, which determine the hygroscopic character attributed to the amorphous state of the powder obtained by lyophilization (Canuto et al., 2014). Such state influences characteristics of the dehydrated material, such as tendency to form agglomerates and the phenomenon of caking (Barbosa, 2010).

The values obtained for the degree of caking of acerola pulp powder obtained through lyophilization were around 14.12% and are within the desirable range of up to 34% for food powders, according to Jaya & Das (2004). Oliveira et al. (2014) observed degree of caking of 6.64% for yellow mombin powder. Oliveira et al. (2013) obtained values of 0.03 and 0.09% for powders of grugru palm (*Acrocomia aculeata*) using a 1200-μm mesh sieve for the analysis of the powder obtained through lyophilization. In the present study, a 500 μm mesh sieve was used for the analysis of caking of acerola pulp powder, according to the methodology. This difference in particle size caused different capacities of absorption and agglomeration of the powders, producing different results in the degree of caking. Costa et al. (2003) claim that different variations in the hygroscopic behavior of food powders are attributed to their size, because finer particles have larger contact surface and, therefore, higher number of active sites.

As to the solubility, the lyophilization process generated acerola pulp powder with 94.08% of solubility. Cano-Chauca et al. (2005) obtained solubility higher than 90% in mango powder mixed with maltodextrin. The high solubility of the powder can be related to the process of freezing and the application of vacuum, generating amorphous products. In addition, they may have broken cell structure, causing larger amounts of solids to dissolve and become part of the supernatant. Cano-Chauca et al. (2005) cite that amorphous solids have high solubility and high dissolution velocity compared with the crystalline state.

Table 2. Characterization of the acerola pulp powder obtained through lyophilization

Parameters	Lyophilized powder
Hygroscopicity (%)	5.96 ± 0.15
Degree of Caking (%)	14.12 ± 9.10
Solubility (%)	94.08 ± 0.73

The results of the adjustments of mathematical models of GAB, BET, Henderson and Oswin to the experimental data of lyophilized acerola pulp powder are shown in Table 3. According to Lomauro et al. (1985), mean relative errors (E) lower than 10% indicate good adjustment of the model to the experimental data. Thus, the models applied at temperatures of 35 and 45 °C showed good adjustments to the experimental data, with error values between 3.60 and 7.15, except for the model of Henderson at 35 °C, which showed value of 14.03. For this study, at the temperature of 25 °C, the model of GAB showed error close to 10%; the mathematical models adjusted to the acerola pulp powder showed coefficients of determination (R^2) from 0.942 to 0.995.

Among the studied models, the BET model showed the best adjustment to the adsorption of acerola pulp powder obtained through lyophilization. The GAB model, despite its lower mean relative error (E) for the temperature of 25 ºC and higher values of coefficient of determination (R^2), cannot be the most representative, because K values are higher than 1, which makes it physically inconsistent, according to Chirife et al. (1992), since it indicates infinite sorption. The other representative models, in decreasing order, were Oswin and Henderson, with errors of 7.15 to 19.59% and 7.13 to 29.13%, respectively. Oliveira et al. (2014), working with lyophilized yellow mombin powder, observed that the BET model promoted the best adjustment for the whole yellow mombin pulp powder, while the model of Henderson was the best to represent the isotherm of the sample containing maltodextrin. Santos et al. (2014), for guava powder obtained through spray dryer, observed that the model of Henderson showed the best adjustment at all the evaluated temperatures. Oliveira et al. (2013), studying the hygroscopic behavior of lyophilized powder of grugru palm (*Acrocomia aculeata*), observed that the models of GAB and Oswin showed the best adjustment to the experimental data of isotherms of the powder without the addition of maltodextrin and mixed with 8% of maltodextrin, respectively.

Table 3. Results of the adjustments of adsorption isotherms for acerola pulp powder obtained through lyophilization

Model	Parameter	Temperature (ºC)		
		25	35	45
GAB	X_m	0.03626	0.04409	0.1787
	C	91.34	3.75	0.3334
	K	1.053	1.036	0.8831
	R^2	0.986	0.990	0.995
	E (%)	10.18	6.58	4.14
BET	X_m	0.05562	0.05770	0.06529
	C	1.754	1.712	1.148
	n	233.4	186.1	180.2
	R^2	0.972	0.988	0.995
	E (%)	12.23	5.01	3.60
Henderson	a	0.4963	0.6219	0.6024
	b	3.125	3.709	3.535
	R^2	0.942	0.977	0.994
	E (%)	29.13	14.03	7.13
Oswin	a	0.05996	0.07077	0.07059
	b	1.027	0.9190	0.9624
	R^2	0.964	0.987	0.993
	E (%)	19.59	6.59	7.15

X_m - Moisture content in the molecular monolayer (g of water per g of dried solids); R^2 - Coefficient of determination; E (%) - Mean relative error; C, K - Sorption constants of the molecular layer; n - Number of molecular layers; a, b - Adjustment parameters

According to Moreira et al. (2013), through the GAB and BET models adjusted for obtaining isotherms, it is possible to evaluate the moisture content of the monolayer (X_m) of foods, allowing a physical knowledge on the adsorption theory. The molecular monolayer is the primary layer of the food and its water content interferes with the hygroscopicity or affinity of the molecules to water. The amount of moisture in the monolayer allows reaching maximum food stability with minimal losses of quality. Below this value, the rates of deterioration reactions, especially in dehydrated food, are minimal, except for the reaction of oxidation of unsaturated fats (Celestino, 2010; Goula et al., 2008). For the acerola pulp powder obtained through lyophilization, there was an increment in the values of the monolayer with the increase in temperature from 25 to 35 and 45 °C (Table 3) for the models of GAB and BET. Although this behavior is not very common, Ferreira & Pena (2003) presented two alternative mechanisms to justify such behavior: temperature increase can cause modifications in the physical structure of product, making available a higher number of active sites with affinity to water molecules and also increase the solubility of solutes intrinsic to the product, causing a higher number of water molecules to remain retained in the monolayer.

Furthermore, there was a difference between the values of moisture content in the monolayer between the models; the BET model showed predominantly higher values. Similarly, Oliveira et al. (2011) observed higher moisture content in the molecular monolayer (Xm) for the model of BET, for the lyophilized sapodilla powder. Moreira et al. (2013) also observed increase in temperature as a predominant factor for the increment of moisture contents in the monolayer (Xm) through the models of GAB and BET, similar to the result of the present study.

The C values for the acerola pulp powder showed a constant decrease with the increase in temperature (Table 3). Moreira et al. (2013) claim that this reduction in C values is expected and cite that low temperatures favor the force of interaction between adsorbate-adsorbent, favoring an increment in the values of the constant C.

Figure 1 shows that the obtained isotherm is of type III, according to the classification of Braunauer (Andrade et al., 2011). The typical form of an isotherm reflects the form in which it connects to the system, so that the weaker interactions with water molecules generate higher water activity; thus, the product becomes more unstable (Andrade et al., 2011). The isotherms with a wider zone in their first part, i.e., in a "J" shape, are typical of food rich in soluble components, such as sugars (Al-Muhtaseb et al., 2004). This form the of curves is characteristic of food with high sugar contents and, at low water activity, the moisture content increases linearly with a_w, whereas, at high levels of water activity, the water content increases rapidly (Pedro et al., 2010).

The sorption isotherm of acerola pulp powder obtained through lyophilization (Figure 1) allows noting that there is an increase in a_w with the increment in equilibrium moisture (X_0) at constant temperature. In relative humidity (RH) values higher than 70%, there is an expressive increase in water absorption by the powder, evidenced by the increase

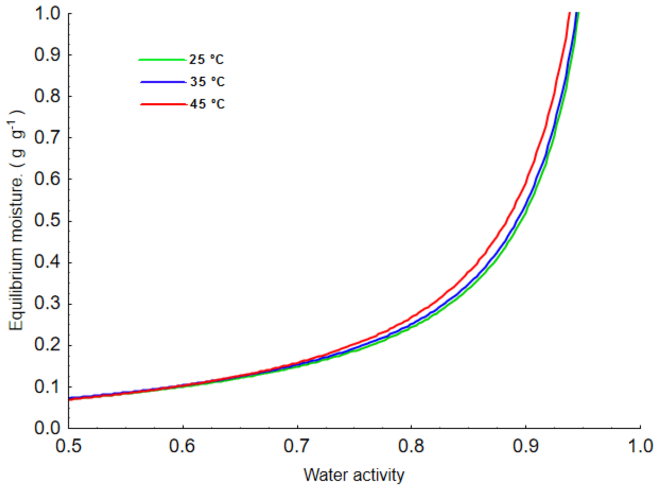

Figure 1. Sorption isotherms of acerola pulp powder obtained in lyophilizer at experimental temperatures of 25, 35 and 45 °C (lines) predicted by the model of BET

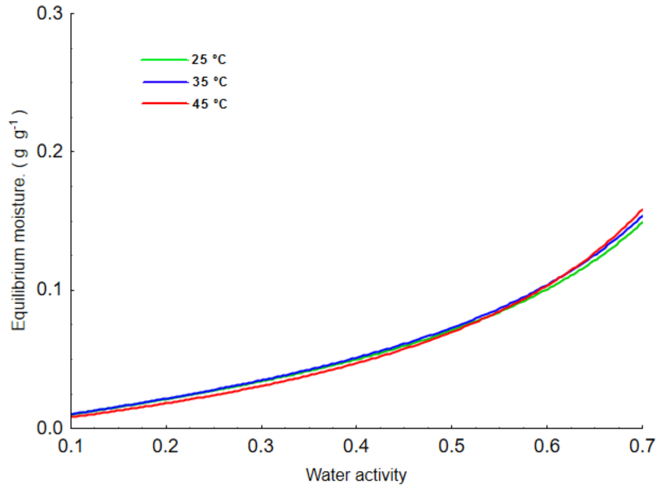

Figure 2. Inversion of the isotherm of lyophilized acerola pulp powder according to the model of BET, for the temperatures of 25, 35 and 45 °C

in equilibrium moisture. At constant temperature, there is a relationship between water activity of a food (a_w) and the equilibrium RH of the air in the closed environment; therefore, it is always 100 times higher than the value of water activity (a_w = RH/100).

For the studied temperatures, which simulate possible storage temperatures, the isotherms have tendency to greater water absorption at higher temperatures, 45 °C in this case, a common phenomenon for products rich in sugar, such as fruit powders.

The effect of temperature on the sorption isotherm is of great importance, because foods are exposed to various temperatures during storage and processing. Temperature affects the mobility of water molecules and the dynamic equilibrium between the vapor and the adsorbed phase (Al-Muhtasheb et al., 2004). According to Goula et al. (2008), the content of equilibrium moisture decreases with the increase in temperature. At a constant water activity, this tendency can be attributed to a reduction in the total number of active sites for bound water, as a result of physical and/or chemical change induced by temperature. However, there may be an inversion of the effect of temperature on the isotherms, which would result in increase of equilibrium moisture with the increase in temperature.

According to Figure 2, there was an inversion of the effect of temperature on the isotherms of the acerola pulp powders obtained through lyophilization. The inversion occurred between the isotherms of 45 and 25 °C in the water activity range of 0.52 to 0.60 and between the isotherms of 45 and 35 °C in the water activity range of 0.60 and 0.67.

The behavior of inversion of the temperature effect on the isotherm of adsorption observed in the present study (Figure 2) is in agreement with Telis-Romero et al. (2005). These authors claim that some studies reported such inversion of the temperature effect for water activity higher than 0.70 in products with high sugar contents, such as fruits, which can be explained by an increase in the solubility of sugar in the water. Pedro et al. (2010) constructed sorption isotherms for passion fruit powder obtained in spray dryer and demonstrated precisely an increase in the content of equilibrium moisture with the increment in water activity at constant temperature.

Conclusions

1. Drying through lyophilization generated an acerola pulp powder with low hygroscopicity, high solubility and a degree of agglomeration (caking) within the acceptable limits for food powders.

2. The model of BET adjusted best to the pulp powder of acerola lyophilized at the temperatures of 35 and 45 ºC; the model for the temperature of 25 °C showed the most acceptable results.

3. There were inversion of the temperature effect on the isotherms of the powder and expressive increase in water absorption by the powder in relative humidity values higher than 70%, especially at higher temperatures, which negatively affect its stability.

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