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Sorption isotherms of powdered cajá-manga pulp in different drying processes[1]

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ABSTRACT - High-sugar dehydrated foods have strong hygroscopicity, which can be avoided by using drying aids. One way to assess the hygroscopic behavior is through sorption isotherms. The objective of this work was to evaluate the influence of maltodextrin concentration on sorption isotherms of powdered cajá-manga pulp in spray-dryer, lyophilization and fluidized bed spray drying processes. The dryings were carried out in a spray-dryer, lyophilizer and fluidized bed, with 10%, 20% and 30% of maltodextrin. Adsorption isotherms were determined by the static gravimetric method using salt solutions for a relative humidity range of 21% to 90%. The GAB, BET, Henderson, and Oswin models were adequate to the experimental data. The relative deviation between the experimental values and the estimated values was calculated for each curve, in order to assess which equation best fit the experimental data. The mean relative error value of less than 10% was considered one of the criteria for selecting the models. The BET (A), BET (B) and GAB (C) models had the lowest error means, 3.35%, 3.35% and 5.28%, for cajá-manga powders obtained by spray-dryer, lyophilization and fluidized bed spray drying processes, respectively, and coefficients of determination above 0.99. It is concluded that the type of drying process did not influence the behavior of the adsorption isotherms, and that increases in maltodextrin concentration and temperature are significant in reducing equilibrium moisture contents.

Key words: Equilibrium curves. Dehydration. Isotherm models. Cajarana.

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INTRODUCTION

The biggest obstacles faced in the dehydration of high-sugar fruit pulp with low molecular weight acids, which have a low glass transition temperature, are the high hygroscopicity, low particle agglomeration, low glass transition temperature, and adherence of the final product to the dryer walls, giving the dry product characteristics such as handling difficulties, transport and storage, and low yield during the drying process (FREITAS *et al*., 2019). These problems can be avoided with the use of drying aids, making the powders less hygroscopic and easy to flow. According to Tonon *et al*. (2009), the incorporation of high molecular weight additives to the product before drying increases the glass transition temperature and prevents agglomeration of powder particles.

One of the most used materials as a drying aid is maltodextrin, due to its low cost, low hygroscopicity, high solubility, in addition to presenting an antioxidant effect and excellent retention of volatile substances, in the order of 65% to 80% (CEBALLOS; GIRALDO; ORREGO, 2012; FERRARI; GERMER; AGUIRRE, 2012).

With the aim of succeeding in the drying process, it is interesting and necessary to know the influence that the addition of adjuvants will exert on the elaborated formulations (FEITOSA *et al*., 2018). Knowledge of the physical properties of powders, such as hygroscopicity, is of great importance, as they directly affect the behavior of the product during storage, handling and processing.

Hygroscopicity is the ability of a material to absorb moisture from the environment and is linked to its physical, chemical and microbiological stability. Therefore, it is essential to know the hygroscopic behavior of dehydrated products (OLIVEIRA; CLEMENTE; COSTA, 2014).

One way to evaluate the hygroscopic behavior of foods is through their sorption isotherms. This procedure portrays the ability of food to absorb water when placed in certain atmospheric and temperature conditions, and is used to define processes, drying time, in addition to determining the shelf life of the product and the type of packaging most suitable for its storage (PICELLI; ARRIECHE; SARTORI, 2010; SOUSA *et al*., 2019).

In this context, the objective was to evaluate the influence of maltodextrin concentration on the sorption isotherms of powdered cajá-manga pulp in spray drying processes, such as spray-dryer, lyophilization and fluidized bed.

MATERIAL AND METHODS

The experiments were carried out from January 2019 to July 2020. Commercial cajá-manga pulps were used, with maltodextrin (DE20) being added in the proportions of 10%, 20% and 30%. The mixture was subjected to homogenization for five minutes at 6,000 rpm in a rotary homogenizer.

Spray drying was carried out with a spray-dryer, model LM MSD 1.0 (Labmaq® do Brasil), with a stainless steel chamber, a 1.2 mm diameter sprinkler nozzle, compressed air flow (3.0 L/min), feed flow (0.5 L/h), drying air flow (3.0 m^3/min) and drying air inlet temperature at 135 °C.

In lyophilization, the samples were placed (100 g) in stainless steel trays (18 cm in diameter) and frozen at -40 °C for 2.3 h in a vertical ultrafreezer from the company Terroni®. Then, they were dehydrated for 24 h in a lyophilizer model L101 from the company Liotop®. After each dehydration, the samples were sieved (500 µm) to homogenize the particles.

The fluidized bed drying was carried out in a spouted bed dryer, model FBD 3.0 Labmaq® Brasil, with a stainless-steel chamber, atomizing nozzle of 1.2 mm in diameter, feed flow of 4.5 mL/min, flow of 30 L/min compressed air, drying air temperature at 78.6 °C and inert polyethylene material (400 g).

The sorption isotherms were determined by the static gravimetric method. Samples weighing about 0.2 g were weighed in previously tared aluminum crucibles. The crucibles were placed on a support inside closed glass cells, containing saturated salt solutions (CH_3COOK – 21%, K_2CO_3 – 44%, NaBr – 58%, $SnCl_2$ – 76%, KCl – 84%, $BaCl_2$ – 90%) at the temperature of 25 °C ± 2 °C.

The process was monitored by weighing the samples every 24 hours until they reached a constant mass, which characterizes equilibrium moisture. After the equilibrium was detected, the water activity was measured, calibrating the equipment at temperatures of 25 ºC, 35 ºC and 45 ºC. Subsequently, the samples were weighed and taken to an oven at 105 °C, until constant weight, to determine the dry mass of each sample.

Equilibrium moisture (X_{eq}) was calculated by the difference between the mass the sample had at equilibrium and its initial dry mass, using the equation below:

$$Xeq = \frac{M_o - M_s}{M_s} \qquad (1)$$

Where:

X_{eq} = equilibrium moisture (g $100g^{-1}$ on a dry basis);

M_o= sample mass at equilibrium (g);

M_s= dry sample mass (g).

For the mathematical adjustment of the experimental data of the adsorption isotherms, the following mathematical models were used: Guggenheim, Anderson and Boer (GAB); Brunauer, Emmet and Teller (BET); Henderson; and Oswin, according to the following equations:

CAB: $$x_e = \frac{X_m \cdot C \cdot K \cdot a_w}{(1 - K \cdot a_w) \cdot (1 - K \cdot a_w + C \cdot K \cdot a_w)} \tag{2}$$

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

Henderson: $$x_e = \left[\frac{-\ln(1 - a_w)}{b} \right]^{\frac{1}{a}} \tag{4}$$

Oswin: $$x_e = a \cdot \left[\frac{a_w}{1 - a_w} \right]^b \tag{5}$$

Where:

X_e – equilibrium moisture content (g 100g-1 on a dry basis);

X_m – moisture content in the monolayer (kg water/kg dry sample);

a_w – water activity;

a, b, C, K, n – constant in the models in which they appear.

The mathematical models were adjusted through non-linear regression analysis using the Gauss Newton method. The models were selected considering the coefficient of determination (R^2) and the mean relative error (E) (Equation 6). We considered the value of the coefficient of determination greater than 95% and the mean relative error less than 10% as one of the criteria for model selection (LOMAURO; BAKSHI; LABUZA, 1985; OLIVEIRA; CLEMENTE; COSTA, 2014; OLIVEIRA; COSTA; AFONSO, 2014; ROCHA *et al*., 2014).

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

Where:

E - relative mean error;

M_i - values obtained experimentally;

Mp_i - values predicted by the model;

n - number of experimental data.

RESULTS AND DISCUSSION

The mathematical models were adjusted to the equilibrium moisture content to select the model that best represented the sorption isotherms, observing, therefore, the equilibrium moisture content as a function of temperature variation and maltodextrin concentration in each experiment.

It was observed that the determination coefficients presented satisfactory values for all models evaluated in this study, in all drying methods. Thus, to determine the best adjusted models, these coefficients were taken into account together with the smallest relative mean errors (E).

The BET model showed the lowest average of errors for the cajá-manga powders obtained by spray drying processes in spray-dryer, lyophilization and GAB in fluidized bed, with averages of 3.35%, 3.35% and 5.28%, respectively, and determination coefficient above 0.99. Adjustments made through the mathematical models of GAB and BET allowed a physical understanding of the adsorption theory (ANDRADE; LUMES; PÉREZ, 2011; MOREIRA *et al*., 2013).

The values of moisture content in the monolayer (X_m), in the BET model, for the powders obtained in spray-dryer, showed a minimum of 0.0614, a maximum of 0.0954 and an average of 0.0774 kg kg^{-1}. There was a reduction in the values of X_m, as the temperature of the evaluation increased, in all concentrations of maltodextrin. Silva *et al*. (2015) also found that with increasing temperature of the isotherms, the moisture content in the molecular monolayer decreased in powdered umbu-cajá pulp. Equilibrium moisture content generally decreases with increasing temperature at constant water activity. This decrease is explained by the fact that absorbed molecules gain kinetic energy, causing the moisture content of the monolayer to decrease with increasing temperature (DIOSADY *et al*., 1996). This trend can also be attributed to the reduction in the number of active sites for binding with water, as a result of physical efforts and/or chemical changes induced by temperature (PERDOMO *et al*., 2009). For the lyophilized powder, the same BET model showed a behavior contrary to the spray-dryer process. Increasing the temperature from 25 ºC to 45 ºC resulted in an increase in moisture values in the monolayer (X_m).

The GAB model provided the best fit for the powder obtained in a fluidized bed. The GAB is a simple model with parameters that have physical definition, in addition to adequately representing the experimental data in a wide range of water activity - 0.10 to 0.90 (PEDRO; TELIS-ROMERO; TELIS, 2010). The moisture content in the monolayer showed an average of 0.1427 kg kg^{-1} between the tests evaluated. The model presented an increase in X_m when the temperature ranged from 25 ºC to 45 °C.

Even though the increase in moisture content in the monolayer with rising temperature is not a behavior common to all foods, Ferreira and Pena (2003) justify this performance to changes in the physical structure of the food according to temperature, which makes available a greater number of active sites with affinity to water molecules. The same authors also mention that there is an increase in solubility of solutes, intrinsic to the product, causing greater retention of water molecules in the monolayer.

It was observed that the growth in maltodextrin content reduced the moisture content in the monolayer of powders obtained in spray-dryer. There was also a reduction in the X_m of the lyophilized samples with the increase in the concentration of maltodextrin. Canuto, Afonso and Costa (2014) also obtained a reduction in monolayer contents with the use of maltodextrin in freeze-dried papaya. In this context, the use of maltodextrin did not favor stability increase of the powders obtained by spray-drying and lyophilization, since there was a reduction in the moisture content of the monolayer, which decreases the relative humidity of the environment to which the powders can be exposed. When this occurs, it is recommended that they be stored in packages that offer resistance to moisture exchange.

There was moisture gain in the monolayer (X_m) in the GAB model for the powder obtained in a fluidized bed, between concentrations of 10% and 30%. Oliveira, Clemente and Costa (2014), and Oliveira, Costa and Afonso (2014) also reported increased moisture in the monolayer when using maltodextrin, up to a temperature of 35 °C, in macaúba coconut pulp and powdered cajá. The samples containing higher levels of maltodextrin were more stable, with higher levels of moisture in the monolayer, allowing exposure to environments with higher relative humidity when compared to samples with lower levels of maltodextrin.

The sorption constant (C), which represents the total heat of sorption of the first layer and has a physical meaning related to the effect of temperature, of the evaluated models, BET (A), BET (B) and GAB (C), of the powders obtained in spray-dryer, lyophilization and fluidized bed, respectively, demonstrated constant reduction, with increasing temperature from 25 °C to 45°C. A similar behavior was observed by Conegero *et al.* (2017) and Cavalcante *et al*. (2018) in their studies on the hygroscopic behavior of powdered mangaba and soursop pulps added with maltodextrin and obtained by drying in lyophilization and spray-dryer, in that order. This reduction can be explained by the strength of interaction between adsorbate-adsorbent, favored by low temperatures, causing an increase in C values (MOREIRA *et al*., 2013).

As for the GAB constant K, which is a measure of the interactions between water vapor molecules with the adsorbent (CATELAM; TRINDADE; ROMERO, 2011), Syamaladevi *et al*. (2009) reported that K values for foods generally range from 0.7 to 1.0. This constant increases with the strength of interaction between water vapor and the adsorbent, and values greater than 1 are physically inadequate, indicating infinite sorption (TIMMERMANN, 2003).

In the powders obtained in a fluidized bed, the K values in all ranges of maltodextrin concentration showed a reduction with the increase in temperature. In this work, the values of the constant K were in the range of $0.7 < K < 1.0$ at all temperatures and maltodextrin concentrations, varying between 0.81 and 0.96. It was also observed that the force of interaction between water vapor and the solid matrix of powdered cajá-manga pulp decreased with the addition of maltodextrin, registering higher K values in samples with 10% maltodextrin. Similar behavior was reported by Oliveira, Costa and Afonso (2014) for cajá pulp, with the K constant values reduced with the addition of maltodextrin.

Figures 1, 2 and 3 illustrate the experimental values of equilibrium moisture as a function of the water activity of powdered cajá-manga pulp obtained in spray-dryer, lyophilization and fluidized bed, at temperatures of 25 ºC, 35 ºC and 45 °C and with maltodextrin concentrations of 10%, 20% and 30%, respectively. In all isotherms, an increase in equilibrium moisture was observed along with an increase in water activity (a_w).

The sorption isotherms of foods with high concentrations of sugar, such as most fruits, generally resemble type II and type III isotherms (OLIVEIRA *et al*., 2013). According to figures 1, 2 and 3 (except 2C), the equilibrium isotherms of powdered cajá-manga pulp showed exponential curves, with characteristic behavior of type III isotherms, according to IUPAC (1985).

The type of isotherm is a reflection of how interactions with water occur. Weaker interactions favor greater water activity, which makes the food more unstable. Feitosa *et al*. (2017) reported that the curves of yacon potato mix powders and freeze-dried lime juice also showed exponential behavior.

In Figure 2C, the equilibrium isotherm at 25 °C for the lyophilized powder presented a curve with sigmoidal behavior, type II, according to the IUPAC classification (1985). The curves of the isotherms, except for Figure 2C, showed similar conditions of water activity and equilibrium humidity, even with the difference in evaluated temperatures. The same occurred in a study by Ribeiro, Costa and Afonso (2016) with the isotherms of powdered freeze-dried acerola pulp at temperatures between 15 and 45 °C.

The increase in temperature modifies the mobility of water molecules and the balance between the vapor and the adsorbed phase, which causes a reduction in the equilibrium moisture content (MOREIRA *et al*., 2013). According to Goula *et al*. (2008), the reduction in the number of active sites available for binding with water occurs for physical and/or chemical reasons, which causes a decrease in equilibrium moisture when increasing temperature.

In this work, it was noted that the equilibrium moisture content of the evaluated powders behaved proportionally to the temperature, that is, the increase in temperature increased the equilibrium moisture content of the isotherms. Conegero *et al*. (2017) stated that the moisture balance also increased with the rise in temperature of the powdered freeze-dried mangaba pulp isotherms. Within the studied temperatures, which aimed to simulate the possible temperatures of the food distribution chain, processing, transport, marketing and storage, the isotherms with the highest temperature tended to increase water absorption.

Figure 1 - Isotherms of adsorption of powdered cajá-manga pulp, obtained in spray-dryer, at temperatures of 25 ºC, 35 ºC and 45 °C, predicted by the BET model with 10% (A), 20% (B) and 30% (C) maltodextrin content

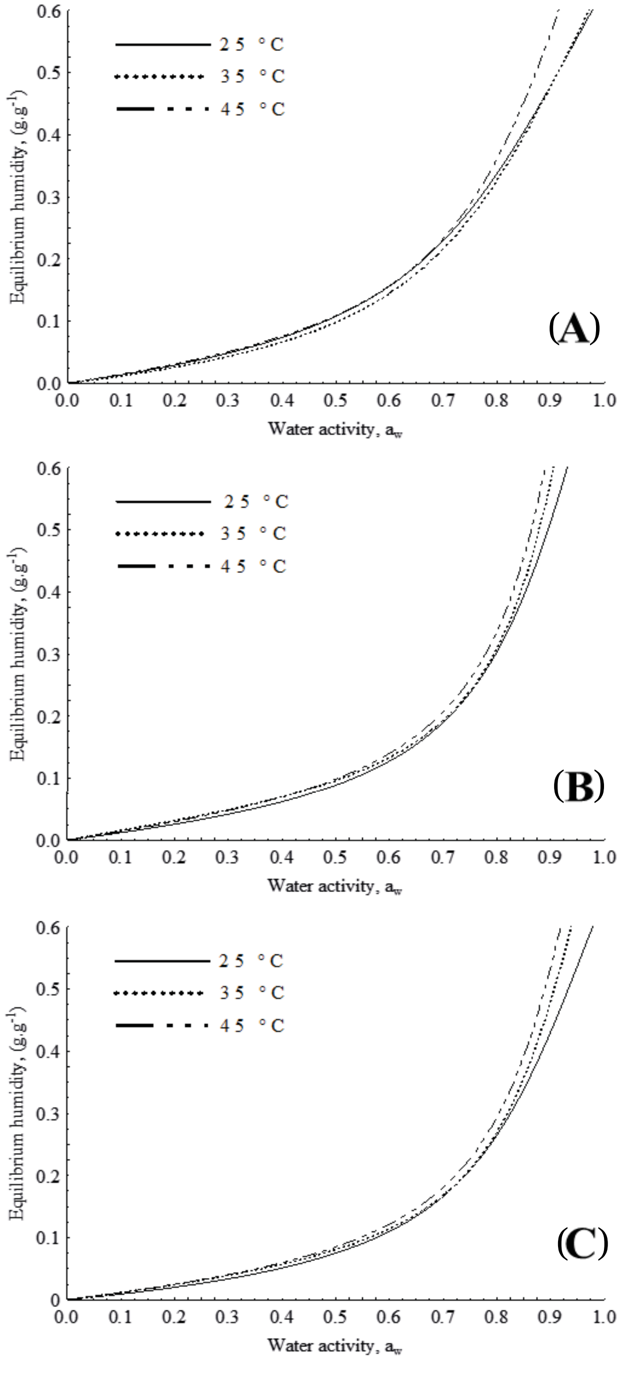


Figure 2 - Isotherms of adsorption of powdered cajá-manga pulp, obtained by lyophilization, at temperatures of 25 ºC, 35 ºC and 45 °C, predicted by the BET model with 10% (A), 20% (B) and 30% (C) maltodextrin content

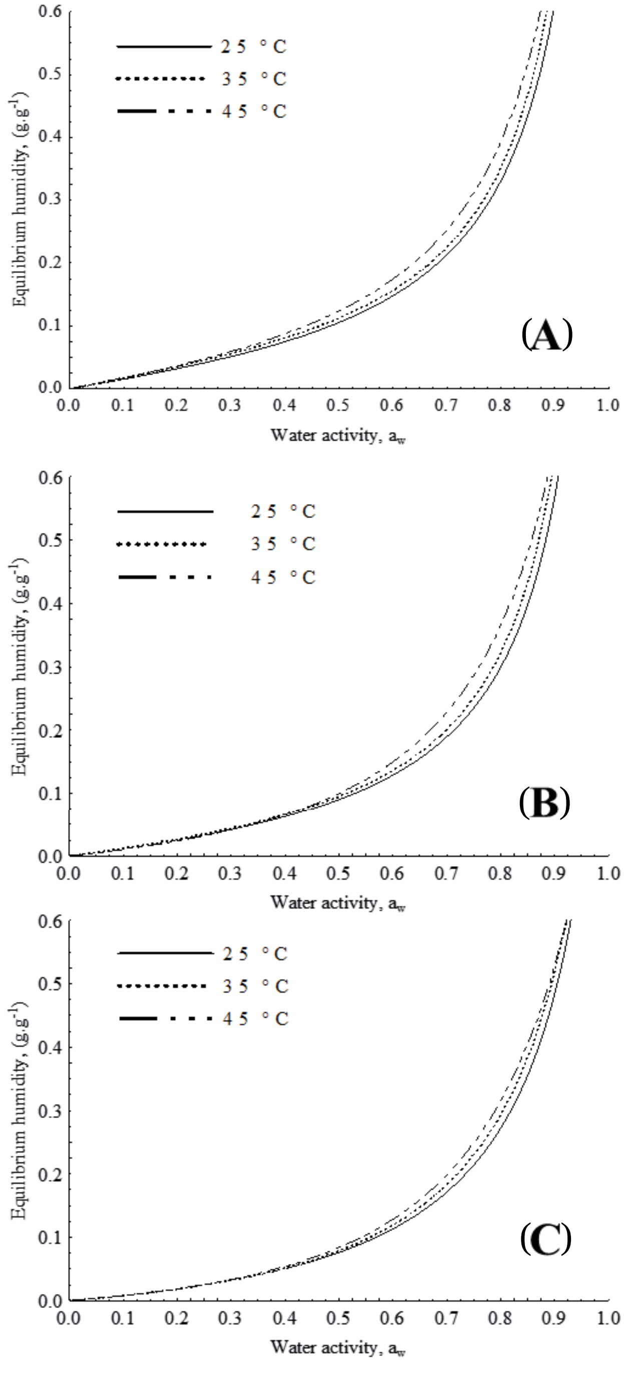
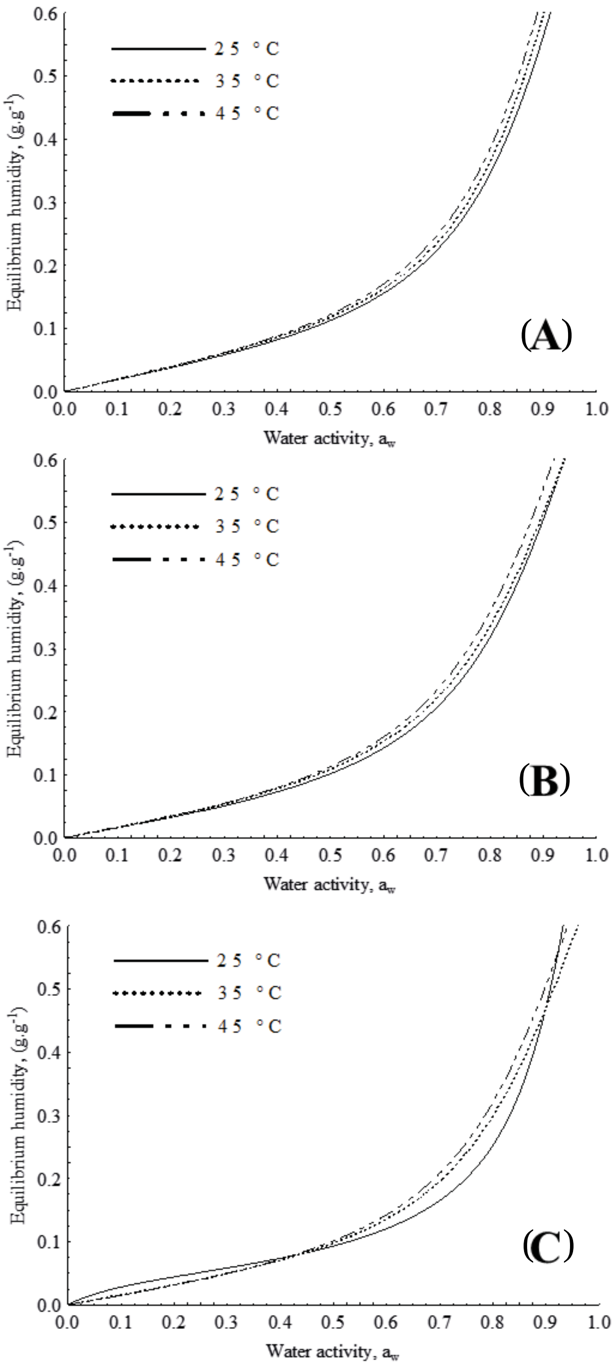


Figure 3 - Isotherms of adsorption of powdered cajá-manga pulp, obtained in a fluidized bed, at temperatures of 25 ºC; 35 ºC and 45 °C, predicted by the GAB model with 10% (A), 20% (B) and 30% (C) maltodextrin content



At constant temperature, in general, it was observed that there was an increase in a_w with an increase in equilibrium humidity. Significant water absorption was evidenced, which caused an increase in the equilibrium humidity of the powders when exposed to environments with relative humidity greater than 70%. According to Ribeiro, Costa and Afonso (2016), at constant temperature, there is a relationship between the a_w of a food and the relative humidity (RH) of the air. In a closed environment, the relative humidity is always 100 times greater than the activity value from water.

According to figures 1, 2 and 3, it was noted that in water activities up to 0.5 there was little variation in the equilibrium humidities between temperatures in the same water activity, that is, at low relative humidities the temperature does not exert much influence on the absorption of water from the environment. Within the range of water activity between 0.5 and 0.7 it was observed that the isotherm curves of the powders caused a small increase in equilibrium moisture at the same water activity. This behavior occurs due to the increase in solubility of sugars in water, according to temperature (PEDRO; TELIS-ROMERO; TELIS, 2010).

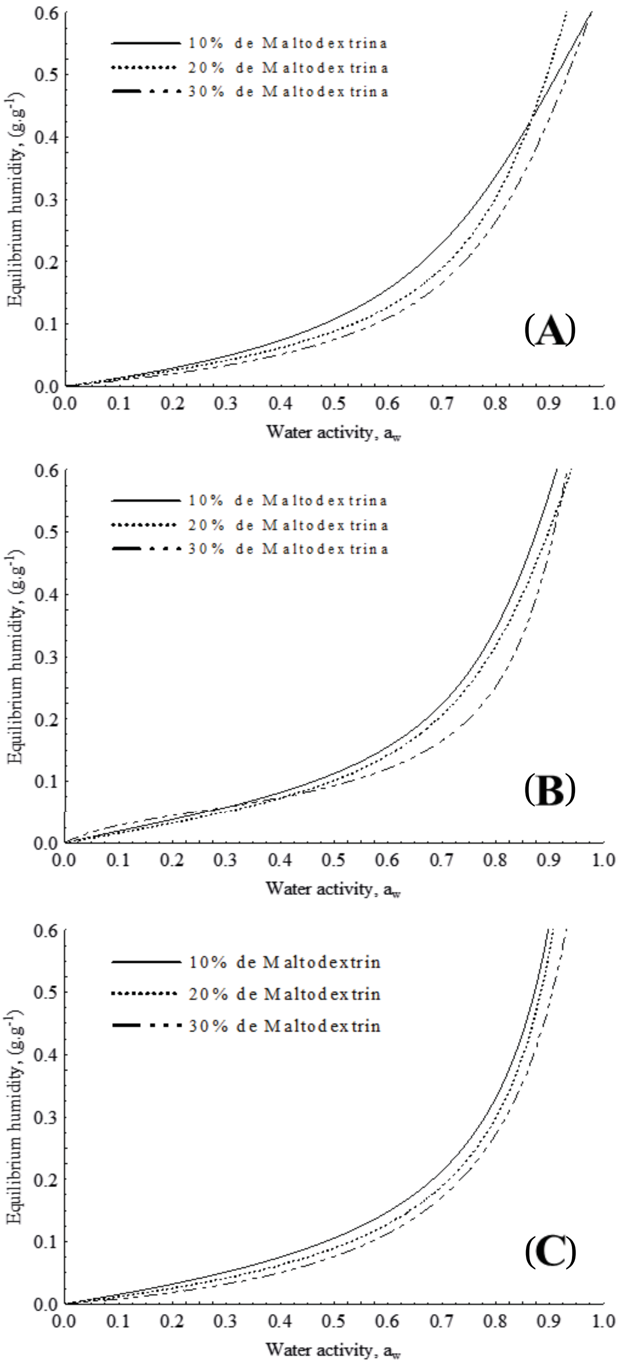
Temperature affected the mobility of water molecules and the dynamic equilibrium between steam and adsorbed gas. It was found that at a_w above 0.7, there was a drastic increase in the equilibrium moisture content of the isotherms.

Ribeiro, Costa and Afonso (2016), when studying the hygroscopic behavior of freeze-dried acerola, found that the isotherms tended towards greater water absorption at higher temperatures and relative humidity greater than 70%, demonstrating similarity with this work, a common phenomenon for high-sugar products like fruit powders. In the region with higher water activities, water exerted a strong influence on the stability of powdered cajá-manga pulp in both drying processes, since water activities greater than 0.6 can favor microbial growth and result in the acceleration of undesirable reactions.

Figure 4 depicts the experimental values of equilibrium moisture as a function of water activity in different concentrations of maltodextrin (10%, 20% and 30%) at 25 °C. The presented isotherms exhibited an increase in equilibrium moisture with increasing water activity (a_w).

The maltodextrin in powdered cajá-manga pulps proportionally decreased the equilibrium moisture of the isotherms (Figure 4). It was observed that, when increasing the concentration of maltodextrin in the pulp during the drying process, there were lower equilibrium moisture values for the same amount of water activity, demonstrating the influence of maltodextrin on the capacity to absorb water, portraying its lower hygroscopicity. This result was due to the fact that maltodextrin is an agent that reduces the hygroscopicity of the sample; therefore, the higher the concentration of maltodextrin, the greater the stability in relation to water absorption. This behavior can be observed by noting the positions of the isotherm curves. The curves of the samples that contain higher maltodextrin contents are presented in a lower position than the samples with lower maltodextrin contents.

Figure 4 - Isotherms of adsorption of powdered cajá-manga pulp, obtained in spray drying, lyophilization and fluidized bed, at a temperature of 25 °C, predicted by the BET (A), BET (B) and GAB (C) models, with 10%, 20% and 30% maltodextrin content



Oliveira, Costa and Afonso (2014), and Canuto, Afonso and Costa (2014) also reported that they obtained lyophilized powders of cajá and papaya with a decrease in the equilibrium moisture content with the use of maltodextrin, which demonstrates that the addition of maltodextrin amortizes the hygroscopicity of food powders.

It can be seen that the isotherms (Figure 4) in the initial regions up to a_w 0.6 did not show large variations in equilibrium moisture. However, in the region of water activity above 0.7, this behavior was modified, that is, any small variation in water activity represented a considerable gain in water by the evaluated powders. A similar behavior was observed in the isotherms studied by Oliveira, Costa and Afonso (2014) with lyophilized cajá pulp using maltodextrin. Canuto, Afonso and Costa (2014) found an exponential increase in equilibrium moisture with small variations in water activity, mainly at values above 0.7. These results demonstrate that these products should be avoided in environments with relative humidity greater than 70%.

The isotherms of powdered cajá-manga pulps obtained by the spray drying process in a spray-dryer (Figure 3C) showed an inversion of the 10% maltodextrin isotherms at the points where water activity is 0.83 and 0.90, with isotherms of 20% and 30%, respectively, which shifted the isotherm in question to the right. This behavior can be explained by the high equilibrium moisture content, increasing the sugar solubilization power, indicating that the sample in which the inversion occurred contains a lower maltodextrin content, therefore, a smaller amount of sugar to be solubilized.

The isotherms of the lyophilized powdered cajá-manga pulps (Figure 4B) also show two inversions of the sample with 30% maltodextrin in relation to other evaluated samples (10% and 20%), as the referred isotherms presented a sigmoid shape, type II, unlike the 10% and 20% maltodextrin samples, type III.

According to Pereira, Queiroz and Figueiredo (2006), the characteristic of adsorption of water in dehydrated foods is attributed by the chemical nature of its organic components, such as sugars, the existence of intermolecular forces of the Van der Waals type, the capacity of hydroxyl groups to form bonds with water molecules, and the type of drying process.

CONCLUSIONS

1. The hygroscopicity of the cajá-manga powders presents similar behavior and, in general, the type of drying process does not influence the behavior of the adsorption isotherms;

2. The increase in maltodextrin concentration and temperature reduces the equilibrium moisture content; therefore, this is a decisive factor to predict the behavior of cajá-manga pulp in the sorption isotherms;
3. From a technological point of view, maltodextrin can be recommended as a suitable drying aid for the production of powdered cajá-manga pulp and for reducing its hygroscopicity.

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