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Hygroscopic behavior of freeze-dried papaya pulp powder with maltodextrin

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ABSTRACT. The sorption isotherms describe the relationship between water activity and equilibrium moisture content of foods at a certain temperature. This work was carried out to study the hygroscopicity of freeze-dried papaya pulp powder with maltodextrin at different concentrations using sorption isotherms constructed at 25°C. Three formulations of freeze-dried powders were prepared, papaya pulp powder, papaya pulp powder with maltodextrin 5% (w w^{-1}) and papaya pulp powder with maltodextrin 15% (w w^{-1}). In order to adjust the adsorption isotherms data, mathematical models of GAB, BET, Henderson and Oswin were used. The GAB model showed the best fit for all samples. The addition of maltodextrin increased the amount of water in the monolayer and reduced the hygroscopicity of the powders.

Keywords: *Carica papaya* L., adsorption isotherms, stability.

Comportamento higroscópico do pó da polpa de mamão liofilizada contendo maltodextrina

RESUMO. As isotermas de sorção descrevem a relação entre a atividade de água e conteúdo de umidade de equilíbrio de um alimento em determinada temperatura. Neste trabalho foi realizado o estudo da higroscopicidade de pós de polpa de mamão liofilizada contendo maltodextrina, em duas concentrações, através da determinação das isotermas de sorção à 25ºC. Foram elaboradas três formulações antes da secagem por liofilização e obtenção dos pós: uma contendo apenas polpa de mamão, outras duas contendo 5 e 15% (p p^{-1}) de maltodextrina. Para ajuste dos dados de adsorção dos pós foram utilizados os modelos matemáticos de GAB, BET, Henderson e Oswin. O modelo de GAB proporcionou o melhor ajuste para todas as formulações estudadas. A adição da maltodextrina aumentou a quantidade de água na monocamada e reduziu a higroscopicidade dos pós.

Palavras-chave: *Carica papaya* L., isotermas de adsorção, estabilidade.

Introduction

Powdered food products are usually very hygroscopic. Hygroscopicity is the ability of food powder to absorb moisture from high relative humidity environment (JAYA; DAS, 2004). Powders obtained by freeze-drying process of fruit juices and pulps are characterized by high hygroscopicity that promotes the phenomenon known as caking and other undesirable effects (CARLOS et al., 2005).

The hygroscopicity of fruit powders with high levels of sugar is attributed to the amorphous state of sugars (SIMATOS; BLOND, 1975). During the freeze-dried process of fruit juice powders with a high degree of amorphous sugar are obtained. These amorphous sugars are responsible for the high hygroscopicity (CARLOS et al., 2005).

Sucrose, glucose and fructose, common in fruit products, are responsible for strong interactions with water molecule due to the polar terminals in these molecules. They are very hygroscopic in amorphous state and loose free flowing nature at high moisture content (JAYA; DAS, 2004).

Maltodextrin is an adjuvant widely used in drying processes, including freeze-drying process. Maltodextrins are obtained from starch hydrolysis, consisting of units of β-D-glucose and are generally classified by their dextrose equivalent (DE) (BEMILLER; WHISTLER, 1996). Maltodextrins are especially used in hard drying materials, such as fruit juices (REINECCIUS, 1991) in order to reduce the problems of agglomeration during storage and to improve product stability (BHANDARI et al., 1993; SILVA et al., 2006).

The sorption isotherms graphically describe the relationship between water activity and equilibrium moisture content of the material at a constant temperature (THYS et al., 2010).

In order to extend food shelf-life, it is interesting to keep its water activity below 0.6 since most microorganisms will not grow below this value. The sorption isotherm knowledge of intermediate moisture foods can predict the shelf life of packaged products susceptible to ambient humidity during storage and distribution (YAN et al., 2008).

Sorption isotherms can be obtained by adsorption or desorption. The adsorption and desorption processes are not fully reversible, so that a differentiation can be made between isotherms to determine whether the product moisture is increasing (adsorption) or decreasing (desorption) (AL-MUHTASEB et al., 2004).

Therefore, this study investigated the hygroscopic behavior of freeze-dried papaya pulp with maltodextrin through adsorption isotherms obtained by fitting the models of GAB (Gugghenheim, Anderson and DeBoer), BET (Brunauer-Emmett-Teller), Henderson and Oswin. In addition, the physicochemical characterization of papaya powders were also conducted and discussed.

Material and methods

Papaya pulp was purchased in the market of Fortaleza city, Ceará State, Brazil. The pulp was stored at -20°C. The pulp was thawed and spread on stainless steel trays with diameter of 15cm. Trays were placed in ultra-freezer (CL 90-40V, Terroni) at -40°C for 24 hours then were taken to a bench freeze-drier (LS 3000, Terroni) for 24 hours.

Three different powder formulations of freeze-dried papaya pulp were used: freeze-dried papaya pulp powder; freeze-dried papaya pulp powder with maltodextrin (dextrose equivalent, DE 20) 5% (w w^{-1}); freeze-dried papaya pulp powder with maltodextrin (dextrose equivalent, DE 20) 15% (w w^{-1}).

Physico-chemical characterization

Powders were characterized by the following analyses in triplicate: pH, total titratable acidity and moisture content according to the methodology from Adolfo Lutz Institute (BRASIL, 2005a); total soluble solids according to AOAC method # 932.12 (AOAC, 1997); reducing/non reducing sugars and soluble sugars according to Miller (1959); and ascorbic acid according to Strohecker and Henning (1967). Therefore, these characteristics were evaluated using descriptive statistical analysis, running the analysis of variance (ANOVA) and Tukey's test ($p < 0.05$).

Modeling of adsorption isotherms

Moisture adsorption isotherms were determined by using a static gravimetric method described by Wolf et al. (1985). Triplicates of 0.12 g samples were weighed in pre-weighed aluminum crucibles. These crucibles were then placed into seven tempered glass cells with 15 cm long and 15 cm high covered with a silicone lid 19 cm length. In order to obtain constant relative humidity in each cell (10, 21, 44, 58, 76, 84 and 90%) were used solutions with saturated salts (LiCl, CH_3CO_2K, K_2CO_3, NaBr, $SnCl_2$, KCl and $BaCl_2$) prepared according to Greenspan (1977).

The process was accomplished by sample weighing every 24 hours until the equilibrium. At this point, the water activity was determined at 25°C using a water activity meter, model AQUALab 4TEV by Decagon Devices, Inc. - EUA. Then, crucibles were taken to a vacuum oven at 60°C for moisture content determination.

The equilibrium moisture content of each sample was calculated by the difference between the balanced sample mass and its dry mass according to the Equation 1.

$$X_{eq} = \frac{m_{eq} - m_s}{m_s} \tag{1}$$

where:

X_{eq} = equilibrium moisture (g g^{-1});
m_{eq} = balanced sample mass (g);
m_s = dried sample mass (g).

For experimental data adjustment to obtain the isotherms of freeze-dried papaya powder, mathematical models of Guggenheim-Anderson-DeBoer (GAB), Brunauer-Emmett-Teller (BET), Henderson and Oswin were used, represented respectively by the equations 2, 3, 4 and 5. The models fitting were obtained using the software Statistica 7.0 (STATSOFT, 2005).

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

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

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

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

where:

a_w = water activity;

X_m = water content in the molecular monolayer (g g^{-1}) = X_{eq} equilibrium moisture (g g^{-1}),

n = number of molecular layers,

C, K = sorption constants,

a, b = adjustment parameters.

The models' fitting were evaluated based on the coefficient of determination (R^2) and the relative mean deviation (E) defined by Iglesias and Chirife (1976) (Equation 6).

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

where:

E = relative mean error;

M_i - = experimental value;

M_p = value predicted by the model;

n = number of experimental data.

Results and discussion

Physicochemical characterization

The Table 1 lists the physicochemical characterization of papaya powders. The pH of formulations ranged from 4.61 to 4.75, with statistical difference between formulations ($p < 0.05$). The addition of maltodextrin led to a gradual reduction in the pH ($p < 0.05$). Grizotto et al. (2005) found lower pH in papaya pulp, with value of 4.19. The values of total acidity expressed as citric acid ranged from 1.38 to 0.55 and also showed significant difference ($p < 0.05$) between formulations. The acidity values have decreased ($p < 0.05$) with the maltodextrin addition due to the reduction of organic acids percentage.

The powders presented high levels of ascorbic acid, characterized as excellent sources of vitamin C, since the recommended daily intake (RDI) is 45 mg (BRASIL, 2005b). There was a significant difference ($p < 0.05$) between formulations. The maltodextrin addition decreased the ascorbic acid concentration. Marques et al. (2006) lyophilized fresh papaya and obtained 3.6 mg ascorbic acid g^{-1} dry solid, lower than found in our study (5.8 mg g^{-1}). This difference can be explained by cropping differences, such as soil, climate and maturity (LEE; KADER, 2000). The use of vacuum and low temperature minimizes the loss of vitamin C during the freeze-drying process (MARQUES et al., 2006). In conventional drying processes, the loss of ascorbic acid is especially affected by high temperatures (MARFIL et al., 2008).

Soluble solids ranged from 89.00 to 93.00° Brix, presenting significant difference ($p < 0.05$) between samples with and without maltodextrin. Powders showed high levels of sugars, from 74.02 to 92.29% for total sugars. All these parameters have significantly differed ($p < 0.05$), except the non-reducing sugars concentration of formulations 1 and 2. The highest sugar concentrations (non-reducing, reducing and total) were showed by formulation 1. The reduction of sugars concentration in the formulations 2 and 3 was due to maltodextrin addition (5 e 15% w w^{-1}) and increase of moisture content.

The freeze-dried papaya powder had moisture values from 1.65 to 2.63%. A significant difference ($p < 0.05$) was detected between samples with and without maltodextrin. The addition of maltodextrin resulted in increased moisture in papaya powders. This suggests that maltodextrin impedes the water output from papaya pulp during drying. Among the samples with maltodextrin (5 and 15%) there was no significant difference ($p > 0.05$). Tonon et al. (2009) spray-dried açai juice with maltodextrin addition (10, 20 and 30%) and found no significant difference ($p > 0.05$) for moisture content.

The fitting parameters of GAB, BET, Henderson and Oswin mathematical models are shown in Table 2. Analyzing the relative mean errors (E) and the model correlation coefficients (R^2), it can be verified that GAB model was the best model for all formulations. The BET model showed good results for formulation 1 and 2, whereas Henderson and Oswin models showed good results for formulation 3 and 2, respectively.

Table 1. Physicochemical results for freeze-dried papaya pulp powders.

Parameters	Formulations*		
	1	2	3
pH	4.61^{c**} ± 0.01	4.71^b ± 0.01	4.75^a ± 0.01
Total titratable acidity (% citric acid)	1.38^a ± 0.07	0.91^b ± 0.07	0.55^c ± 0.00
Ascorbic acid (mg 100g^{-1} dry basis)	580.1^a ± 0.00	418.2^b ± 0.00	241.4^c ± 0.00
Total soluble solids at 20°C (°Brix)	89.00^b ± 0.00	93.00^a ± 1.00	92.67^a ± 1.15
Non-reducing sugars (%)	56.15^a ± 0.15	55.88^a ± 0.14	47.65^b ± 0.20
Reducing sugars (%)	36.13^a ± 0.03	32.58^b ± 0.07	26.38^c ± 0.07
Total sugars (%)	92.29^a ± 0.16	88.46^b ± 0.16	74.02^c ± 0.16
Moisture (%)	1.65^b ± 0.11	2.63^a ± 0.10	2.53^a ± 0.10

*Formulations: 1) freeze-dried papaya pulp powder; 2) freeze-dried papaya pulp powder with 5% maltodextrin (w w-1); 3) freeze-dried papaya pulp powder with 15% maltodextrin (w w-1). **a,b,c – Mean values followed by the same letter in row are not statistically different ($p > 0.05$) by Tukey's test.

Table 2. Adjustment results of the adsorption isotherms at 25°C from the formulations of freeze-dried papaya pulp powders.

Models	Formulation*	PARAMETERS			R^2	E(%)
		X_m (g g^{-1})	C	K		
GAB	1	0.1106	0.8408	0.9998	0.9760	3.108
	2	0.1220	0.8606	0.9885	0.9999	2.219
	3	0.1470	0.5900	0.8977	0.9916	7.421
		X_m (g g^{-1})	C	n		
BET	1	0.1533	0.4847	171.9	0.9767	17.04
	2	0.1275	0.7103	27.05	0.9999	2.318
	3	0.1496	0.3572	11.41	0.9924	9.892
		a	b			
Henderson	1	0.4453	2.139		0.9708	29.65
	2	0.5349	2.406		0.9973	11.84
	3	0.6452	3.389		0.9920	8.206
		a	b			
Oswin	1	0.09972	1.167		0.9768	17.27
	2	0.1109	0.9925		0.9999	3.261
	3	0.09075	0.8388		0.9887	11.23

*Formulations: 1) freeze-dried papaya pulp powder; 2) freeze-dried papaya pulp powder with 5% maltodextrin (w w-1); 3) freeze-dried papaya pulp powder with 15% maltodextrin (w w^{-1}).

According to Rahman (2009), the BET model is applicable to water activity between 0.05 and 0.45. However, in this study, the BET model could be fairly adjusted for formulations with maltodextrin for the full range of water activity. For formulation 1, the BET model did not achieve satisfactory results showing a large relative mean error of 17%.

Relative mean errors (E) lower than 10% indicate good model fit to the experimental data (LOMAURO et al., 1985). In this work GAB and BET models presented mean errors below 10%. Only the BET adjustment for formulation 1 showed a higher value. The Oswin and Henderson models showed the greatest relative errors (Table 2) among the models. Jain et al. (2010), studying isotherms of osmotically dehydrated papaya cubes, have considered good adjustments those attained by Henderson and Oswin models, using only R^2 values (0.97 to 0.99) as a criterion. This work reveals R^2 values similar to those of Jain et al. (2010). On the other side, Alcântara et al. (2009), studying isotherm fitting for dried cashew peduncle, reported results similar to this study, obtaining the best fitting for GAB model followed by BET model.

The moisture content of the food monolayer (X_m) could be evaluated by GAB and BET models. This parameter is recognized as the food moisture content that provides greater stability and minimal loss of quality at constant temperature. Below this moisture content value, the rates of degradation reactions in food are minimal, except for unsaturated fat oxidation (GOULA et al., 2008). The values of X_m (Table 2) for each formulation obtained by these two models are very close, except for formulation 1 whose error was greater in the BET model. According to the GAB model, the lowest value of X_m was obtained by the powder without maltodextrin while the highest value was reached by formulation 3 (15% maltodextrin). It can be observed that as the maltodextrin concentration increases, the X_m values also increase.

According to Timmermann (2003), the constant K of GAB increases linearly as the interaction between adsorbate and adsorbent increases. Considering this, the strength of interaction between water vapor and freeze-dried papaya powder decreases with increasing maltodextrin concentration, and higher values of K are observed for the formulation 1 (Table 2). For Chirife et al. (1992), K values higher than one are physically inadequate because it means that the sorption will be unlimited. In this study all K values were lower than one.

In agreement with Blahovec (2004), for the Henderson model, 'a' should be greater than 0 and 'b' greater than 1, and for the Oswin model 'a' greater than 0 and 'b' between 0 and 1. Given these conditions, isotherms will not have inflection points, and therefore, Henderson and Oswin models will show mathematical and physical consistency (ALCÂNTARA et al., 2009). Compared to this study, all powder formulations presented consistent values for the parameters 'a' and 'b', except the parameter 'b' from formulation 1 in the adjustment of model Oswin, whose value was greater than 1.

Figures 1 to 4 illustrate experimental values of equilibrium moisture (X_{eq}) of freeze-dried papaya powders as a function of water activity. In all isotherms, an increase in the equilibrium moisture (X_{eq}) along with increasing water activity (a_w) was observed.

The isotherms showed a behavior typical of type III, according to the classification of the International Union of Pure and Applied Chemistry (IUPAC, 1985). Góis and Cal-Vidal (1984), studied the equilibrium moisture of lyophilized papaya powder and granules at different temperatures, and also obtained this type of isotherm. The 'J' shaped isotherms are typical of foods rich in soluble components such as sugars (AL-MUHTASEB et al., 2004). The presence of maltodextrin in the powders increased the size of the isotherms flattest area as can be seen when compared to Figures 1 to 3.

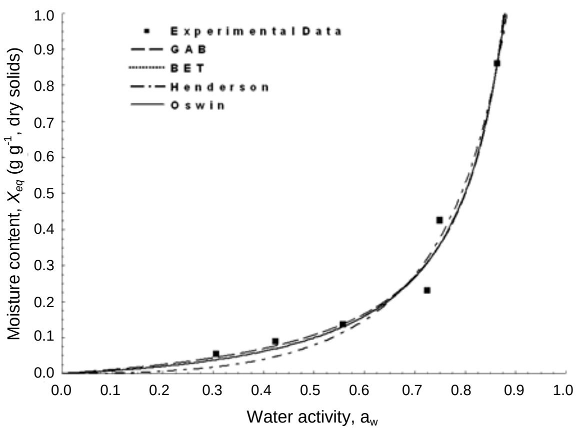

Figure 1. Adsorption isotherms at 25° C of freeze-dried papaya pulp powder.

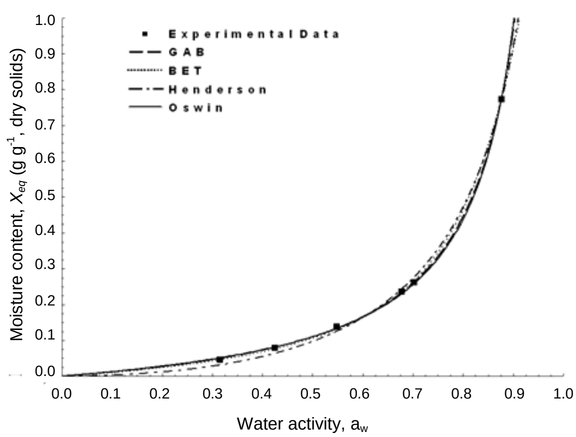

Figure 2. Adsorption isotherms at 25° C of freeze-dried papaya pulp powder with 5% maltodextrin.

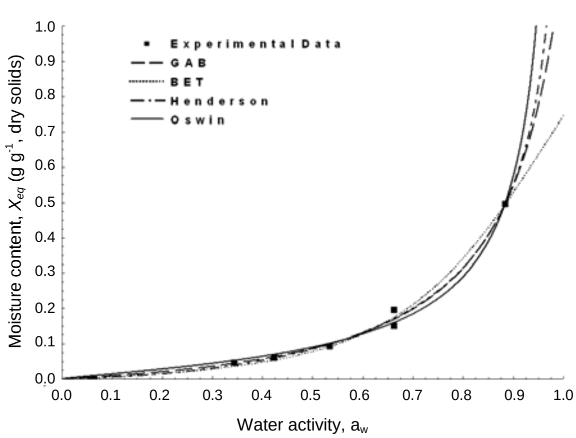

Figure 3. Adsorption isotherms at 25°C of freeze-dried papaya pulp powder with 15% maltodextrin.

Figure 4 shows isotherms for the three formulations adjusted by the GAB model. According to this representation, for the same amount of water activity (a_w), there were lower values of equilibrium moisture (X_{eq}) for the 15% maltodextrin papaya powder compared with other formulations. This behavior is more evident for values of water activity above 0.4. In comparison with formulation 1, the formulation 2 had lower values of X_{eq} for the same a_w especially those above 0.7. Mosquera et al. (2010), examined the stability of the freeze-dried borojo (*Borojoa patinoi)* powder, and obtained for the same a_w, lower X_{eq} in samples with maltodextrin (DE 4.0-7.0 16 or DE 16,5-19.5) when compared to samples without the addition of adjuvant. The addition of maltodextrin improved the stability, lowered the hygroscopicity and increased vitreous transition temperature of the powder.

Góis and Cal-Vidal (1984) reported the exponential growth of X_{eq} with small variations in a_w, particularly for values above 0.7, which indicates that freeze-dried papaya is a highly hygroscopic product. Similar behavior was observed in this study, where formulations 1 and 2 confirmed the high hygroscopicity of the powders for a_w above 0.7. For formulation 3, this tendency was clear for a_w above 0.8.

Analyzing the Figure 4, the increasing maltodextrin concentration shifted the isotherms to the right, resulting in increased powder stability. With a_w values close to 0.3, powders presented hygroscopic behavior very similar and independent from maltodextrin presence.

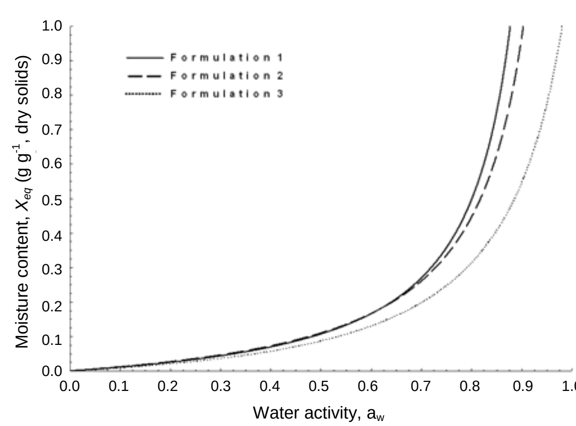

Figure 4. Adsorption isotherms at 25°C for freeze-dried papaya pulp powder (Formulation 1), freeze-dried papaya pulp powder with 5% maltodextrin (Formulation 2) and freeze-dried papaya pulp powder with 15% maltodextrin (Formulation 3).

Conclusion

The physicochemical characterization showed that freeze-dried papaya powders are good sources of ascorbic acid and sugars. The addition of maltodextrin to papaya pulp formulations influenced the physical-chemical composition of its powders, reducing the concentration of total titratable acidity, ascorbic acid and sugars.

Experimental data were best fitted to GAB model for all formulations studied, with errors below 10% and high correlation coefficients. Based on the isotherm analysis and model parameters, it is concluded that the addition of maltodextrin increases the water content at the monolayer, but decreases the hygroscopic characteristics of powders. The addition of maltodextrin to the freeze-dried papaya pulp improves the powder stability and decreases the powder hygroscopicity.

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