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Effect of Acidity Reduction and Anticaking Use on the Hygroscopic Behavior of Tamarind Pulp Powder

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The high acid content in tamarind pulp can inhibit consumer sensory acceptance of its products and cause problems during lyophilization, resulting in a powder with high hygroscopicity, and which is prone to caking due to its hydrophilic nature. Tamarind pulp, in turn, has great nutritional potential, and stands out in relation to other pulps because of its functional attributes that are under investigation due to its low glycemic index. This study sought to characterize tamarind pulp and the effects of acidity reduction on hygroscopic behavior. Tamarind pulp powder was evaluated by analyzing moisture content, hygroscopicity, degree of caking, and adsorption isotherms, by fitting experimental data to the GAB, BET, Henderson, and Oswin mathematical models. The results demonstrated that all tamarind powder treatments resulted in low moisture content and hygroscopicity values, but a high degree of caking. The GAB model represented the best fit to the adsorption isotherms, with coefficients of determination (R^2) between 0.97 and 0.99 and average errors (E) < 5.67 %, presenting a type III behavior, characteristic of foods rich in sugar, and it is recommended to store this product in environments with a relative humidity below 60 %.

1. Introduction

The most expressive sensory characteristics of tamarind pulp include acidity and bittersweet flavors, dark brown color, and fibrous texture (Souza, 2015). The high acidity of tamarind juice has been identified as a limiting factor for consumption. According to Maia (2018), in a sensory analysis of tamarind juice, there was an increase in the acceptance of the juice after acidity reduction (pH 3.5) compared to a control (pH 2.5). In its nutritional composition, the tamarind pulp contains fiber, phenolic compounds, and organic acids that confer beneficial health properties, such as antioxidant, anti-inflammatory, and antibacterial activities (Kuru, 2014). In addition, Passos (2017) evaluated tamarind consumption and its effects related with decrease of glycemic index in diabetic patients.

The high acidity and viscosity of tamarind pulp can cause hygroscopic problems during the lyophilization process to obtain pulp powder. According to Bhusari et al., (2014), some of these problems are due to low molecular weight acids and sugars in the pulp. Thus, to minimize the effects related to the high acid content as a limiting factor for good drying and for sensory acceptance, it is necessary to investigate the effects of acidity reduction in the lyophilization process of tamarind pulp, and the hygroscopic behavior of tamarind pulp powder.

The addition of an anticaking agent after lyophilization was also evaluated because of the satisfactory results found in reducing tamarind pulp powder caking in a study by Passos (2017), using calcium carbonate as an anticaking agent in the tamarind powder obtained by lyophilization. In this study, use of calcium carbonate concentrations of 2.5 %, 5.0 %, and 7.5 % improved the fluidity of the powder and reducing caking. Therefore, the lowest concentration (2.5 %) was recommended in order to avoid possible changes in the composition and functional effects of the powder.In powder product studies, it is essential to know their hygroscopic behavior; therefore, moisture adsorption isotherms are used to understand and predict product stability (Cano-Higuita et al., 2015).

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Given the above considerations, this study aimed to evaluate the effects of acidity reduction, and the use of anticaking agents on the hygroscopic behavior of tamarind pulp powder obtained by freeze-drying, through determination of adsorption isotherms and physicochemical characterization.

2. Material and methods

2.1 Materials

Tamarind fruits (Tamarindus indica L.) were obtained from a production area of Tabuleiro de Russas - Ceará and processed at the Embrapa Tropical Agroindustry facility. Dehusked fruits (pulp + seed) were hydrated with potable water, 1: 1 (m: v) and left to stand for 4 h with occasional stirring. The pulp was then separated from the seeds in a Bonina 0.25 DF (Bonina, Itabuna, Brazil) machine equipped with a 2.5 mm sieve. The total solids content of the pulp obtained were adjusted to 20 % with the addition of water.

2.2 Methods

Two tamarind pulp treatments were prepared: control “C” (pH 2.5), and pulp with acidity partially neutralized “N” (up to pH 3.5), which was adjusted using a 15 % potassium hydroxide solution. Treatments were frozen at -18 °C, and lyophilization was carried out in a Liobrás freeze-dryer (model Liotop LP 510, Piracicaba, Brazil), at a pressure < 3000 µm Hg, with a temperature program starting at -20ºC and programmed to reach up to 30 ºC in 50 h. The freeze-dried tamarind was ground into powder using a multiprocessor (Robot Coupe, model R502, Ridgeland, MS, USA), and for treatments with an anticaking agent, 2.5 % CaCO3 was added. The treatments were packed in barrier multilayer nylon-aluminum-polyethylene sachets and stored at room temperature (~27 °C) until use in analyses

2.3 Physicochemical characterization

Tamarind pulp powder from all treatments was carried out in triplicate using the following analyses: water activity (a_w) measured at 25 °C using a digital hygrometer (Aqualab® 4TE, Decagon, Pullman, WA, USA), and moisture content defined by infrared using a digital humidity scale (ID50, Marte, São Paulo, Brasil).

2.4 Color

Parameters L* (luminosity), a* (green intensity), and b* (intensity of yellow) were performed using a colorimeter (CR-410, Konica Minolta, Japan).

2.5 Hygroscopic characterization

The hygroscopic characterization of tamarind pulp powder before and after acidity reduction, and after addition of an anticaking agent, was performed according to the method given by Goula and Adamopoulos (2008). Hygroscopicity was expressed in g of water adsorbed per 100 g of dry solids.The degree of caking was also determined by a method described in Goula and Adamopoulos (2008). Solubility was also verified with hygroscopicity and solubility expressed as percentages. The results of hygroscopicity measurements and degree of caking were compared with the values presented by Pisecky, Westergaard and Refstrup (2012). The analyses were performed in 3 repetitions, in a randomized block design, with analysis of variance and Tukey's test among means ($p < 0.05$).The flow index (ffc) and bulk density of the tamarind pulp powder were determined using the powder flow tester (PFT; Brookfield, Middleboro, Massachusetts, USA). The type of flow index, proposed by Jenike (1964), was used: no flow ($ffc<1$), very cohesive ($1<ffc<2$), cohesive ($2<ffc<4$), easy flow ($4<ffc<10$), and free flow ($ffc>10$).

2.6 Sorption isotherms

Adsorption isotherms were performed with the use of a static gravimetric method. Tamarind powder samples (0.2 g) in triplicate were placed in an environment with relative humidity equilibrated by different saturated saline solutions, such as potassium acetate ($a_w = 0.21$), potassium carbonate ($a_w = 0.44$), sodium bromide ($a_w = 0.58$), tin (II) chloride ($a_w = 0.76$), potassium chloride ($a_w = 0.84$) and barium chloride ($a_w = 0.90$), prepared according to Greenspan (1977).This process was accompanied by weighing the samples every 24 h for approximately 18 days until they reached constant weight and with variation lower than 0.1 % (0.001 g g^{-1} solid), thus determining the equilibrium mass. Subsequently, the water activity (a_w) was measured using a digital hygrometer (Aqualab® 3TE, Decagon, Pullman, WA, USA) at 25 °C and 40 °C. The samples were weighed and fully dehydrated (105 ºC oven, 16 to 24 h) to determine the dry mass. The equilibrium moisture (Xe) was calculated as the difference between the sample mass at equilibrium and its initial dry mass. The data obtained were used to evaluate which mathematical model (GAB, BET, Henderson and Oswin, respectively shown in Equations 1-4) represents the adsorption isotherms.

$$Xe = \frac{X_m.C.K.a_w}{(1 - K.a_w).(1 - K.a_w + C.K.a_w)}$$ GAB model (1)

$$Xe = \frac{Xm.C.a_w}{(1 - a_w)}\left[\frac{1 - (n+1).(a_w)^n + n.(a_w)^{n+1}}{1 - (1 - C).a_w - C.(a_w)^{n+1}}\right]$$ BET model (2)

$$X_0 = \left[\frac{-ln(1 - a_w)}{b}\right]^{\frac{1}{a}}$$ Henderson model (3)

$$X_0 = a.\left[\frac{a_w}{1 - a_w}\right]^b$$ Oswin model(4)

The modeling was adjusted using Statistica 7.0 software (Statsoft Inc., Tulsa, OK), and model quality levels were assessed using the correlation coefficient (R^2) and the relative average error (E).

3. Results and discussion

The results of tamarind powder characterization are shown in Table 1, which presents the moisture content, water activity, color, flow index, and density of the tamarind pulp powder determined after freeze-drying.
Moisture content and water activity in all treatments were similar, with values < 3 % for moisture and up to 0.2 for aw. Compared with the results obtained by Bhusari et al., (2014), for spray-dried tamarind powder, the moisture levels ranging from 3.65 to 7.11 %. In addition, Muzaffar and Kumar (2016) obtained 2.62 % moisture value, similar to those found in the present study (2.63 % and 2.80 %).

Table 1: Physicochemical and flowability parameters of tamarind pulp powder (dry basis) obtained after freeze-drying and with or without the addition of calcium carbonate ($CaCO_3$) as an anticaking agent.

Parameter	Tamarind pulp powder			
	C (pH 2.5)	N (pH 3.5)	C + $CaCO_3$	N + $CaCO_3$
Moisture (%)	2.63 ± 0.13 ^{a}	2.68 ± 0.07 ^{a}	2.80 ± 0.78 ^{a}	2.80 ± 0.03 ^{a}
Water activity (a_w)	0.19 ± 0.02 ^{a}	0.16 ± 0.01 ^{ab}	0.15 ± 0.02 ^{b}	0.15 ± 0.01 ^{b}
L*	61.28 ± 2.20^a	65.93 ± 0.29 ^{b}	67.92 ± 0.84 ^{bc}	70.79 ± 0.01 ^{c}
a*	7.63 ± 0.62 ^{a}	5.97 ± 0.10 ^{b}	6.26 ± 0.25 ^{b}	4.90 ± 0.01 ^{c}
b*	29.48 ± 0.12 ^{a}	26.50 ± 0.02 ^{b}	24.56 ± 0.08 ^{c}	22.39 ± 0.03 ^{d}
Flow index	1.01 (very cohesive)	2.24 (cohesive)	4.20 (easy flow)	4.25 (easy flow)
Bulk density (kg/m^3)	621.9	585.5	663.5	619.3

Results as mean of three repetitions ± standard deviation. Different letters following the mean, in the lines, indicate significant difference in Tukey´s test ($p < 0.05$).

The color of the powdered tamarind pulp was slightly lighter in the case of treatments with added anticaking agent, with parameter L* between 67.92 and 70.79. Cavalcante et al., (2018) stated that a high value of L* is a positive aspect of drying conditions, because this indicates that there was no browning of the powder. In relation to the present study involving tamarind powder, this observation is only valid for treatments without $CaCO_3$ addition, since the additive has a white color and can influence L* values.
The addition of the anticaking agent contributed to the flowability of the tamarind powder. The flow index resulting from the treatments with $CaCO_3$ (C + $CaCO_3$ and N + $CaCO_3$) exhibited easy flow. The neutralized pulp powder presented a cohesive flow behavior (2.24 ffc), and the control treatment resulted in a "very cohesive" powder.
Powders that have a very cohesive flow exhibit difficult flowability and are highly susceptible to agglomeration. Therefore, flow problems and flow interruptions may imply a decrease in the efficiency of the processes in which they are involved, such as storage in silos, or even flow in packages when opened, at first use.
The lower bulk density value of Neutralized tamarind powder, when compared to Control treatment, indicates greater porosity. In addition, the use of $CaCO_3$ increased bulk density. This can be related to the size and porosity of the powder particles, as particles with higher porosity have lower bulk density (Cavalcante et al., 2018). The solubility, hygroscopicity, and caking degree data is presented in Table 2.

The solubility of a powder can be associated with moisture content, as this decreases as powder solubility increases (Muzaffar, Kumar, 2016). Thus, if the moisture content of the tamarind pulp powder is low, its solubility may be considered high.

Table 2: Hygroscopic parameters of tamarind pulp powder (dry base) obtained by freeze-drying with and without the addition of $CaCO_3$.

Parameter	Tamarind pulp powder			
	C (pH 2.5)	N (pH 3.5)	C + $CaCO_3$	N + $CaCO_3$
Solubility (%)	67.89 ± 0.41 ^{a}	72.97 ± 1.20 ^{b}	66.83 ± 1.77 ^{a}	71.86 ± 0.86 ^{b}
Hygroscopicity (g.100 g^{-1})	7.43 ± 0.26 ^{a}	5.85 ± 1.17 ^{a}	7.65 ± 0.69 ^{a}	6.03 ± 1.38 ^{a}
Caking degree (%)	30.32 ± 0.41 ^{a}	22.60 ± 0.85 ^{b}	29.16 ± 0.59 ^{a}	22.95 ± 0.68 ^{b}

Results as mean of three repetitions ± standard deviation. Different letters following the mean, in the lines, indicate significant difference in Tukey´s test ($p < 0.05$).

Cavalcante et al., (2018) considered the powder obtained from the soursop pulp to exhibit high solubility (62.46 ± 0.4). The powder of tamarind pulp obtained in the present study resulted in even higher solubility values (Table 2), indicating high solubility for all treatments studied.

According to Pisecky, Westergaard and Refstrup (2012), tamarind pulp powder presents a “non-hygroscopic” behavior (< 10 %) for all treatments. The hygroscopicity values obtained for all treatments (Table 2) were lower than those obtained by Bhusari et al., (2016), with values from 16.61 to 28.96 g.100 g^{-1} For tamarind powder obtained by spray-drying process.

Concerning the degree of caking, with values between 22.60 and 30.32 %, being classified as “caking formation” by Pisecky, Westergaard and Refstrup (2012). The control treatments (values of 30.32 % and 29.60 % for C and C+ $CaCO_3$, respectively) were higher ($p<0.05$) when compared to the treatments with acidity reduction (Neutralized, with 22.60 % and 22.95 %). In terms of particle agglomeration, reduction in tamarind pulp acidity was more beneficial than addition of an anti-caking agent to the pulp powder. In addition, the use of barrier packaging may be effective in preventing increases in the degree of caking, as this was efficient in controlling moisture levels.

Table 3: Mathematical modeling parameters to represent the adsorption isotherms of tamarind pulp powder, developed from control (C) and neutralized (N) pulp treatments, with and without the use of an anti-caking agent ($CaCO_3$), and obtained using a freeze-dryer.

Models	Parameters	Control		Control + $CaCO_3$		Neutralized		Neutralized + $CaCO_3$	
		25 °C	40 °C	25 °C	40 °C	25 °C	40 °C	25 °C	40 °C
GAB	Xm	0.156	0.184	0.139	0.173	0.095	0.094	0.088	0.088
	C	0.982	0.876	0.646	0.646	1.406	1.755	1.261	1.563
	K	0.946	0.934	0.970	0.946	0.972	0.990	0.990	1.005
	R^2	0.999	0.998	0.999	0.999	0.998	0.997	0.999	0.998
	E (%)	5.67	4.95	7.90	2.91	5.62	3.58	1.63	4.62
BET	Xm	0.036	0.134	0.196	0.126	0.538	0.088	0.280	0.092
	C	1890.6	1.260	5673.7	0.910	0.192	2.00	0.324	1.426
	N	0.727	20.459	0.003	22.932	1.666	39.414	1.487	169.16
	R^2	0.996	0.999	0.998	0.999	0.999	0.997	0.999	0.998
	E (%)	13.05	5.44	12.90	3.05	3.09	4.24	2.03	4.84
Henderson	A	0.641	0.650	0.545	0.588	0.637	0.643	0.587	0.599
	B	2.535	2.426	2.530	2.470	3.042	2.894	2.923	2.789
	R^2	0.998	0.998	0.997	0.998	0.996	0.994	0.997	0.995
	E (%)	5.01	7.64	6.33	9.30	7.81	11.59	10.60	12.23
Oswin	A	0.145	0.156	0.106	0.1262	0.107	0.116	0.096	0.106
	B	0.857	0.863	0.990	0.946	0.860	0.876	0.928	0.937
	R^2	0.999	0.998	0.998	0.998	0.998	0.997	0.999	0.998
	E (%)	8.25	7.25	9.34	6.84	5.95	3.28	1.55	4.97

X_m represents the moisture content in the molecular monolayer (g of water per g of dry solids); R^2 is the correlation coefficient; E (%) is the relative average error; C indicates the molecular layer sorption constant; N is the number of molecular layers; A, B are the adjustment parameters. predicted by the GAB model.

The results of the hygroscopic behavior obtained by adsorption isotherms (Table 3) indicated that the GAB model is suitable for adjusting the experimental data of the tamarind pulp powder obtained by freeze-drying. To define the mathematical model fitting of the adsorption isotherms, the correlation coefficient (R^2) represents the relationship between the observed responses and the values predicted by the adjusted model. The relative average error, E (%) is defined as the relative difference between experimental and predicted values (%) (Pedro et al., 2010). According to Labuza et al., (1985), E (%) value below 10 % indicates a reasonable adjustment, and up to 5 % is considered for isotherms representation.Thus, for greater data accuracy, the best model selected (GAB) considered the highest indices of the correlation coefficient (R^2) and the lowest relative average errors, E (%) showed in the Table 3. All parameters adjusted by the models applied in the adsorption isotherms of tamarind pulp powder, including the values of the correlation coefficient (R^2) and relative average errors E (%), are shown in Table 3.From observation of the isotherm data presented in Table 3, the R^2 values were greater than 0.91 in all mathematical models and the relative average error (E) exceeded 10 % for BET and Henderson models, while GAB and Oswin models presented the lowest values (average error lower than 10 %).Therefore, considering the E values, the GAB model is best suited to describe the adsorption isotherms of the tamarind pulp powder at temperatures of 25 °C and 40 °C for all treatments evaluated (Table 3). Muzaffar and Kumar (2016) also indicated the GAB model for the study of the adsorption isotherm of tamarind pulp obtained by spray drying.

The monolayer equilibrium moisture value (Xm) indicates the amount of water that is strongly adsorbed at specific locations on the food surface and is directly associated with product stability (Muzaffar and Kumar, 2016). The Xm values exhibited an increasing trend in the control tamarind pulp powder with and without the addition of anti-caking agent at temperatures of 25 °C and 40 °C. Meanwhile, the neutralized powder (with and without anti-caking additive), presented lower values of moisture in the monolayer, with a tendency to remain stable at both temperatures (25 °C and 40 °C). This behavior can be observed from the moisture monolayer data (Xm) presented in Table 3.

Figure 1 shows the adsorption curves of tamarind pulp powder obtained by the GAB model for each treatment at temperatures of 25 °C and 40 °C. It shows that for a 0.6 value of water activity (a_w), the control powder presents an equilibrium moisture of 0.2 g g^{-1}. In the neutralized powder, at the same a_w (0.6), the moisture was < 0.2 g g^{-1} at both temperatures evaluated (25 °C and 40 °C). This behavior may suggest that the pulp acidity reduction results in more stable powder during storage.

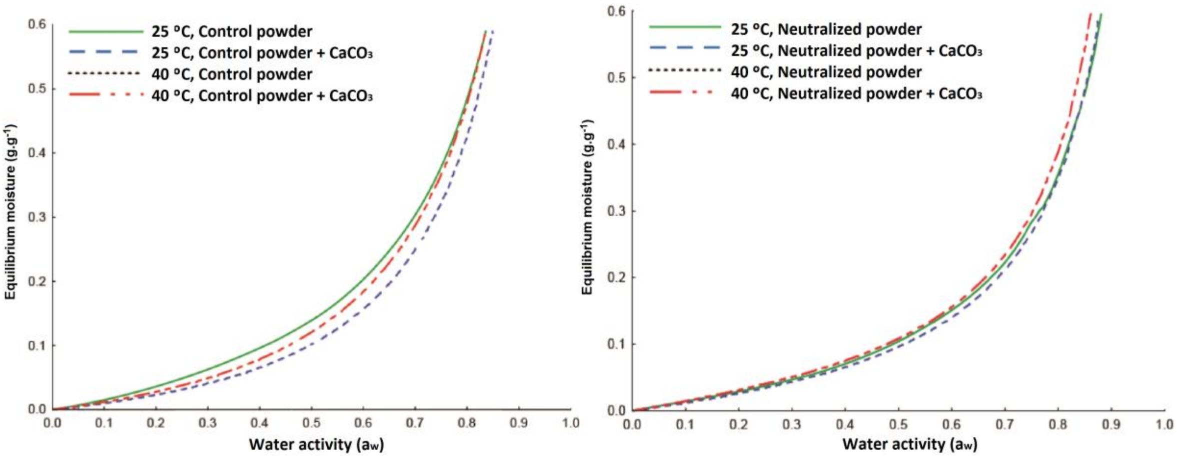

Figure 1: Adsorption isotherm curves of tamarind pulp powder at temperatures of 25 °C and 40 °C (lines) predicted by the GAB model.

In all treatments, the observed curves represent the typical behavior of foods rich in sugar, with "J" curves. This behavior is related to solute-solvent interactions associated with sugar dissolution (Cano-Higuita et al., 2015). This behavior is even more evident from a_w 0.2 for the control samples, in which a small increase in water activity results in a large rise in moisture in the powder. It is recommended to carefully consider high barrier packages when the storing of tamarind pulp powder in environments with relative humidity above 20 %.The reduction in acidity of the tamarind pulp improved lyophilization, presenting powders with more uniform curves (Figure 1), which is similar behavior to that of equilibrium moisture values, even with temperature increases. This indicated a positive influence of tamarind pulp acidity reduction before lyophilization and the addition of the anti-caking agent to the powder before packaging.Cavalcante et al., (2018) studying a spray-dried soursop powder, obtained isotherms similar to those of neutralized tamarind pulp powder and indicated a value of up to 60 % of

relative humidity during storage; above this value, there may be impacts on material stability. Thus, the same for tamarind pulp powder N (pH3.5), with or without the addition of $CaCO_3$ (2.5 %), can be recommended.
Acidity reduction and addition of $CaCO_3$ were useful in terms of enabling gradual and slow behavior of equilibrium moisture (Figure 1), softening the effects caused by the temperature (40 °C). The package was efficient as a barrier to the humidity of the air, maintaining the hygroscopic behavior and flowability, while the calcium carbonate helped to decrease the agglomeration of particles and maintaining the flow indices.

4. Conclusions

The freeze-drying of tamarind pulp, evaluated with the use of acidity reduction and use of anticaking agent, presented satisfactory results for hygroscopicity, solubility and flow index. However, all exhibited “caking formation” and a beneficial effect with an anticaking agent in the formulations of tamarind pulp powder to improve flowability. The GAB model better described the adsorption isotherms. Tamarind powder with acidity reduction (pH 3.5) was more resistant to the influence of air humidity than tamarind powder control (pH 2.5). It is recommended to store tamarind pulp powder in environments with a relative humidity of < 20 %. For tamarind pulp powder with reduced acidity (pH 3.5), the relative humidity during storage can be up to 60 %, with a reduced stability above this value.

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