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Desorption isotherms and isosteric heat of baru almond flour

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ABSTRACT: Baru almond (*Dipteryx alata* Vogel) is a nutritional and tasty product; the flour of this product is used in culinary recipes. In the processing of the almond, the chemical composition and structure of the product changes, influencing the hygroscopicity of the material. Thus, the objective was to determine the behavior of the water activity of baru almond flour at different temperatures, adjusting the best mathematical model to estimate the isotherms, as well as to determine the isosteric heat of desorption. The almonds were dried for moisture contents in the range of 5.4 to 3.3 ± 0.03% dry basis (d.b.), then the almonds were processed in an industrial blender to obtain the flours. To determine the desorption isotherms, the static-indirect method was used, in which the water activity was obtained at temperatures of 10, 20, 30, and 40 ºC, using the Hygropalm Model Aw1 equipment. The hygroscopic equilibrium moisture content of baru almond flour was directly proportional to water activity and decreased with increasing temperature, to the same water activity value. The Chung-Pfost model was the one that best represented the product's isotherms. The integral isosteric heat of desorption of baru almond flour ranged from 2590.69 to 2519.52 for the moisture content range of 3.24 to 5.43% d.b.

Key words: Chung-Pfost; *Dipteryx alata* Vogel; moisture content

Isotermas de dessorção e calor isostérico da farinha das amêndoas de baru

RESUMO: A amêndoa de baru (*Dipteryx alata* Vogel) é um produto nutricional e saboroso, sendo a farinha deste produto utilizada em receitas culinárias. No processamento da amêndoa altera-se a composição química e estrutura do produto, influenciando na higroscopicidade do material. Desta forma, objetivou-se determinar o comportamento da atividade de água da farinha da amêndoa de baru em diferentes temperaturas, ajustando o melhor modelo matemático para estimar as isotermas, bem como determinar o calor isostérico de dessorção. As amêndoas foram secas para os teores de água na faixa de 5,4 a 3,3 ± 0,03% base seca (b.s.), em seguida as amêndoas foram trituradas em liquidificador industrial para obtenção das farinhas. Para determinar as isotermas de dessorção utilizou-se o método estático-indireto, em que a atividade de água foi obtida nas temperaturas de 10, 20, 30 e 40 ºC, por meio do equipamento Hygropalm Model Aw1. O teor de água de equilíbrio higroscópico da farinha da amêndoa do baru foi diretamente proporcional a atividade de água e decresceu com o aumento da temperatura, para um mesmo valor de atividade de água. O modelo de Chung-Pfost foi o que melhor representou as isotermas do produto. O calor isostérico integral de dessorção da farinha de amêndoas do baru variou de 2590,69 a 2519,52 para a faixa de teor de água de 3,24 a 5,43% b.s.

Palavras-chave: Chung-Pfost; *Dipteryx alata* Vogel; teor de água

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Introduction

Within the richness of native cerrado species, the baru (*Dipteryx alata* Vogel) stands out as a fruit with great commercial attributes due to its sweet pulp and edible almonds, with high nutritional content (Cruz et al., 2011). The baru almond is a seed widely consumed by the local community; the fruit can be fully utilized for the production of cereal bars, breads, cookies, liqueurs, oil extraction (Reis et al., 2019).

It is estimated that the nutritional content of almonds *in natura* is 22.96 g $100g^{-1}$ of protein, 31.73 g $100g^{-1}$ of total lipids, 37.13 g $100g^{-1}$ of carbohydrates; the microminerals in greatest quantity are: 1,810 mg $100g^{-1}$ of potassium, 380 mg $100g^{-1}$ of sulfur, 330 mg $100g^{-1}$ of magnesium, and 240 mg $100g^{-1}$ of calcium, in addition, this product is very rich in bioactive compounds (Borges et al., 2014; Campidelli et al., 2019).

The baru almonds can be eaten raw or used in recipes, and the flour from this almond is used in cooking. Although the valorization of this product has expanded in recent years there is still little information regarding the post-harvest processes about the baru fruits (Oliveira et al., 2016), especially the by-products, such as the almond meal.

When a product is processed, in the case of almonds, dehydration and grinding alter the chemical composition and structure of the product; these factors are closely related to the hygroscopicity of the product, because they affect the sorption zones in the food (Park et al., 2001; Pumacahua-Ramos et al., 2017). Every plant product is hygroscopic, the moisture content of this material when in equilibrium with the psychrometric conditions of the air that surrounds it is considered as equilibrium moisture content (Corrêa et al., 2014).

Because of this, it is extremely important to know the relationship of the equilibrium moisture content with the relative humidity of the environment, for a given temperature, because this interaction is described by sorption isotherms, which illustrate the adsorption and/or desorption of the product. From the determination of these curves, one obtains parameters to establish processing and packaging technologies (Araújo et al., 2005; Corrêa et al., 2014).

The determination of isotherm curves is currently being increasingly employed for different plant products, for example: for sorghum saccharin (Ullmann et al., 2016), kidney bean (Jian & Jaias, 2018), grain sorghum (Fonseca et al., 2020), coffee beans (Santos et al., 2020), flax seed (Valente et al., 2020), and paddy rice (Zeymer et al., 2019). These curves are specific to the product, that is, any change in the composition of the product promotes a change in hygroscopic behavior (Brooker et al., 1992; Park et al., 2001).

The minimum amount of heat required to remove an amount of water is estimated by the isosteric heat of desorption (Lima et al., 2008). This thermodynamic property is determined from the estimated water activity data of the product by the mathematical model used to predict the sorption isotherms, through the Clausius-Clapeyron equation (Iglesias & Chirife, 1976).

In view of the importance of isotherm curves for the adequate processing and packaging of the baru kernel flours, the objective of this work was to determine the behavior of the water activity of this product at temperatures of 10, 20, 30, and 40°C, fitting the best mathematical model to estimate the isotherms, as well as to determine the isosteric heat of desorption.

Materials and Methods

Processing of almonds

The present research was developed in the Post-harvest Laboratory of Vegetable Products of the Goiano Federal Institute of Education, Science and Technology – Rio Verde Campus, located in the municipality of Rio Verde, GO, Brazil. Baru fruits were collected manually in the municipality of Porteirão, GO, Brazil.

The fruits were separated according to their integrity, discarding those that presented deterioration. They were then sanitized in sodium hypochlorite solution (500 mg L^{-1}) for 15 minutes, after which the residual sanitizer was removed under running water. After the sanitization step, the kernels were extracted from the interior of the woody endocarp using an equipment called the baru coconut-breaking machine, produced exclusively by the company Metalmix.

To conduct the experiment we worked with ranges of moisture content of the kernel flour from 5.4 to 3.3 ± 0.03% dry basis (db), these moisture contents were obtained by drying the kernel in an oven with forced ventilation, maintained at a temperature of 45 °C. The reduction in moisture content throughout the drying process was monitored by the gravimetric method, with weighing sequences until the desired moisture content was reached.

After being dried, the almonds were processed in an industrial blender to produce the almond meal. The moisture content of the kernel flour was determined by the gravimetric method in an oven at 105 ± 3 °C until constant mass, in three repetitions of 20 g.

Desorption isotherms

The sorption isotherms of the baru kernel (*Dipteryx alata* Vogel) were determined using the indirect static method, and the water activity (a_w) was determined using the Hygropalm Model Aw1 equipment. For each moisture content, three samples of approximately 20 g were used, which were individually placed in the container of the equipment, and this was conditioned in B.O.D. regulated at 10, 20, 30, and 40°C.

The mathematical models (Eq.s 1 to 6) frequently used to represent the hygroscopicity of agricultural products were fitted to the experimental data, and the equations are presented in Table 1.

For the adjustment of the mathematical models, a non-linear regression analysis was performed, using the Gauss Newton method. To verify the degree of fit of each model, the magnitude of the coefficient of determination (R^2), the values

Table 1. Mathematical models used to predict the desorption isotherms of baru (*Dipterix alata* Vogel) kernel flour.

Model designation	Model	
$Xe = a - b \cdot \ln[-(T + c) \cdot \ln(a_w)]$	Chung-Pfost	(1)
$Xe = \exp[a - (b \cdot T) + (c \cdot a_w)]$	Copace	(2)
$Xe = [\exp(a - b \cdot T)/-\ln(a_w)]^{\frac{1}{c}}$	Halsey Modified	(3)
$Xe = \exp\{a - (b \cdot T) + [c \cdot \exp(a_w)]\}$	Sigma Copace	(4)
$Xe = (a + b \cdot T)/[a_w/(1 - a_w)]^{\frac{1}{c}}$	Oswin Modified	(5)
$Xe = (a \cdot b \cdot a_w) \cdot \frac{\left(\frac{c}{T}\right)}{\left(1 - b \cdot a_w + \left(\frac{c}{T}\right) \cdot b \cdot a_w\right) \cdot (1 - b \cdot a_w)}$	GAB Modified	(6)

Where: Xe – Equilibrium moisture content, % d.b.; a_w – Water activity, decimal; T – Temperature, °C; a, b e c - Model coefficients.

of the average relative error (P), average estimated error (SE), chi-square (χ^2) and the behavior of the distribution of residuals were considered. The average relative error, average estimated error, and chi-square test were calculated for each mathematical model using Equations 7, 8, and 9, respectively:

$$P = \frac{100}{n} \sum \frac{\left|Y - \hat{Y}\right|}{Y} \quad (7)$$

$$SE = \sqrt{\frac{\sum \left(Y - \hat{Y}\right)^2}{GLR}} \quad (8)$$

$$\chi^2 = \sum \frac{\left(Y - \hat{Y}\right)^2}{GLR} \quad (9)$$

where:

Y - experimental value;

Ŷ - estimated value by the model;

n - number of experimental observations;

GLR - model degree of freedom (number of observations minus number of model parameters).

To select a single model to best describe the hygroscopicity of baru kernel flour, the models that obtained good fits according to the previous parameters were submitted to analysis of the AIC (Akaike Information Criterion) and BIC (Bayesian Schwarz Information Criterion). The information criteria were obtained by Equations 10 and 11, respectively:

$$AIC = -2 \log like + 2p \quad (10)$$

$$BIC = -2 \log like + p \cdot \ln(N - r) \quad (11)$$

where:

p - number of model parameters;

N - total number of observations;

R - classification of matrix X (incidence fixed effect matrix);

loglike - log-likelihood function value considering parameter estimates.

Isosteric heat of desorption

The values of the net isosteric heat of sorption (or differential enthalpy), for each equilibrium moisture content, were obtained using the Clausius-Clapeyron equation (Iglesias & Chirife, 1976) as shown in Equation 12.

$$\frac{\partial \ln(a_w)}{\partial T} = \frac{\Delta h_{st}}{R T_a^2} \quad (12)$$

where:

T_a - absolute temperature, K;

Δh_{st} - net isosteric heat of sorption, kJ kg^{-1};

R - universal gas constant, 8.314 kJ kmol^{-1} K^{-1}, being for water vapor 0.4619 kJ kg^{-1} K^{-1}.

Integrating Equation 12 and assuming that the net isosteric heat of sorption is independent of temperature, one obtains the net isosteric heat of sorption, for each equilibrium moisture content, as shown in Equation 13.

$$\ln(a_w) = -\left(\frac{\Delta h_{st}}{R}\right)\frac{1}{T_a} + C \quad (13)$$

where:

C - model coefficient.

The values of water activity, temperature and equilibrium moisture content were obtained from the desorption isotherms of the baru kernel flour, using the model with the best fit to the experimental data. The integral isosteric heat of sorption was obtained by adding to the net isosteric heat of sorption values, the value of the latent heat of vaporization of the free water according to Equation 14.

$$Q_{st} = \Delta h_{st} + L = a \exp\left(-b \cdot Xe^*\right) + L \quad (14)$$

where:

Q_{st} - isosteric integral heat of sorption, kJ kg^{-1};

a, b, and c - model coefficients;

L - latent heat of vaporization of free water, kJ kg^{-1}.

The latent heat of vaporization of free water (L), in kJ kg^{-1}, necessary to calculate Q_{st}, was obtained using the average temperature (T) in the range under study, in °C, through Equation 15.

$$L = 2502.2 - 2.39T \quad (15)$$

Results and Discussion

Table 2 presents the average values of the equilibrium moisture content of the baru kernel flour as a function of temperature and water activity. We observe that the moisture content decreases with increasing temperature, this desorption occurs due to the predominance of lipids in the composition of the almond meal (Campidelli et al., 2019). Foods rich in lipids have a lower affinity for water (Brooker et al., 1992), that is, the increase in temperature promotes the elevation of the water vaporization energy inside the product, which culminates in the evaporation of free water from the flour.

It can be seen in Table 2, that for the same moisture content, as the temperature increases, the higher the water activity values are. This behavior was observed by Fonseca et al. (2020) in a survey of experimental data on water activity by the static-indirect method for grain sorghum grains.

Table 3 shows the values of coefficients of determination (R^2), average estimated error (SE), and average relative error (P), chi-square (χ^2), AIC and BIC for the models fitted to the experimental data.

The fits of the different models showed high values of the coefficient of determination, greater than 94%. The fit that obtained the best index for this parameter was the Chung-Pfost model, with a determination coefficient of 96.65%. The value of the average relative error (P) was less than 10%, the maximum limit defined by Mohapatra & Rao (2005), for all models tested when fitting the isotherms, with the Chung-Pfost model showing the best results, 3.406%. As for the values obtained for the average estimated error (SE), it can be seen that the Chung-Pfost model presented the lowest magnitude, being 0.169 (decimal).

Table 2. Average values of the equilibrium moisture content (% db) of the flour from the baru kernel (*Dipteryx alata* Vogel), obtained by the desorption process, as a function of temperature (°C) and water activity (decimal).

Water activity (decimal)	Temperature (°C) 10	20	30	40
0.2917	3.33			
0.3100	3.85			
0.3130		3.33		
0.3160			3.24	
0.3205				3.28
0.3260		3.85		
0.3450			3.76	
0.3485				3.76
0.4340	4.83			
0.4350		4.83		
0.4383			4.79	
0.4427				4.71
0.5213	5.43			
0.5260		5.43		
0.5340			5.43	
0.5370				5.43

Regarding the Chi-square (χ^2) test, the analyzed models are in the 95% confidence interval (χ^2 tab = 22.362). Comparing the values, the Chung-Pfost model obtained the lowest value (0.0029), considering that lower values for this parameter indicate less deviation of the estimated data from the experimental conditions (Eq. 9), one can compare the Chi-square values of the model fits that presented the calculated χ^2 within the confidence interval, as performed by Oliveira et al. (2018).

Table 3. Parameters of the models fitted to the experimental data of hygroscopicity of flour from baru (*Dipteryx alata* Vogel) kernels with their respective values of average estimated error (SE, decimal), chi-square (χ^2, decimal), coefficients of determination (R^2, %), average relative error (P, %), AIC and BIC (decimal).

Models	Parameters	SE	χ^2	R^2	P	AIC	BIC
Chung-Pfost	a = 23.5871** b = 3.3484** c = 322.1728**	0.169	0.029	96.65	3.406	-6.7887	-3.6983
Copace	a = 0.6673** b = 0.0019^{ns} c = 2.0589**	0.204	0.042	95.13	4.093	-0.8187	2.27165
Halsey Modified	a = 1.9307** b = 0.0026^{ns} c = 1.3552**	0.211	0.044	94.79	4.222	0.2528	3.3432
Sigma Modified	a = -0.5136** b = 0.0019^{ns} c = 1.3383**	0.223	0.050	94.19	4.471	2.0128	5.1032
Oswin Modified	a = 5.4628** b = -0.0101^{ns} c = 2.0196**	0.192	0.037	95.66	3.815	-0.4346	1.8832
GAB Modified	a = 2.6775** b = 1.0098** c = 537.2552*	0.222	0.049	94.22	4.431	-1.8213	1.2691

** Significant at 1% by t test; * Significant at 5% by t test; ^{ns}Not significant by t test.

The models showed low values for AIC and BIC, with the Chung-Pfost model showing the lowest values for these parameters, and this model was chosen to represent the hygroscopicity of the baru kernel. Ferreira Junior et al. (2018) working with hygroscopicity of Jatobá (*Hymenea stignocarpa* Mart.) seeds satisfactorily used AIC and BIC to select the best model, which in the respective study was the Modified Oswin model.

Therefore, all the models tested fitted the hygroscopic equilibrium moisture content of the baru kernel flour satisfactorily. The Chung-Pfost model was selected to represent the hygroscopicity phenomenon of the baru kernel flour because it had the highest determination coefficient and the lowest relative and average estimated error values of the chi-square, AIC and BIC tests.

The Chung-Pfost model has been recommended to estimate isotherms various plant products such as, kidney beans (Jian & Jaias, 2018), paddy rice (Zeymer et al., 2019), and flaxseed (Corrêa et al., 2020). Isotherm curves are a response to the interaction between moisture content and water activity, and several factors can influence the behavior of a product curve, such as chemical composition and product structure (Park et al., 2001).

Figure 1 shows the experimental values of equilibrium moisture content and desorption isotherms estimated by the Chung-Pfost model for the baru (*Dipteryx alata* Vogel) kernel flour, under different temperature and water activity conditions.

Note that in Figure 1 a good fit of the data estimated by the Chung-Pfost model to the experimental data of the equilibrium moisture content of the baru kernel flour, Ullmann et al. (2016) also obtained a good fit of this model to the data of the hygroscopicity of saccharine sorghum seeds. Observe that, as the temperature increases for the same water activity value, the higher are the equilibrium moisture contents.

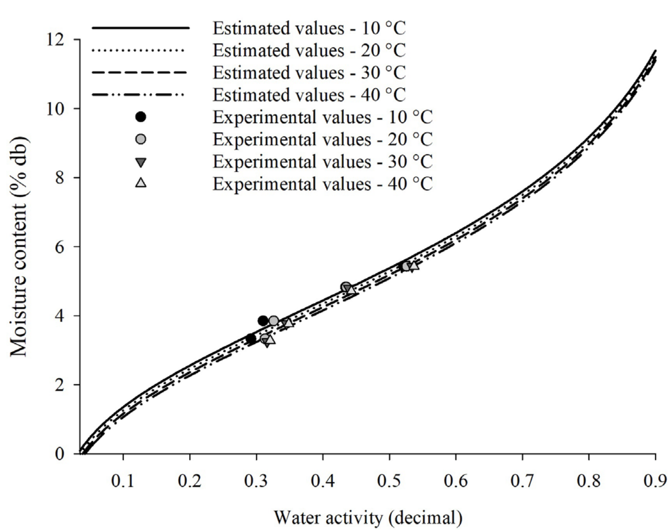

Figure 1. Experimental values of equilibrium moisture content and desorption isotherms estimated by the Chung-Pfost model for baru (*Dipteryx alata* Vogel) kernel flour under different temperature and water activity conditions.

Corroborating with Cavalcante et al. (2018) who observed the same behavior for the desorption isotherms of dried cajá pulp in foam bed.

Oliveira et al. (2005) define that the safe limit of water activity for storage of vegetable products is 0.7, since values higher than this initiate microbiological growth. Considering this a_w limit, the moisture content values for the baru kernel flours should be lower than 7.60, 7.50, 7.40, and 7.31% db, for temperatures of 10, 20, 30, and 40 ºC, limits estimated by the Chung-Pfost model (Figure 1) through Equation 16.

$$Xe = 23.5871^{**} - 3.3484^{**} \cdot \ln\left[-\left(T + 322.1728^{**}\right)\ln\left(a_w\right)\right] \quad (16)$$

** Significant at 1% by t test.

It is observed in Figure 2 that the isosteric integral heat of desorption decreases with increasing moisture content, a behavior observed by other authors such as Choque-Quispe et al. (2018) for amaranth beans and flour, Santos et al. (2020) for Arabica coffee beans, and Valente et al. (2020) for flaxseed.

The integral isosteric heat of desorption of the baru kernel flour ranged from 2590.69 to 2519.52 for the moisture content range of 3.24 to 5.43% db (Figure 2). For quinoa starch the isosteric heat of adsorption was 3731.7 to 2867.1 for the moisture content range of 0.5 to 10% db (Pumacahua-Ramos et al., 2017). It can be seen that the values of the isosteric integral heat of desorption of the baru kernel flour, are lower when compared to the quinoa starch due to the different characteristics presented by the products.

The predominant chemical composition of lipids in almond flour influences its interaction with water, which occurs differently when compared to quinoa starch, a product rich in carbohydrates. Substances rich in starch tend to be more hygroscopic, thus retaining a large amount of water, while those rich in lipids are hydrophobic and do not adsorb a large amount of water.

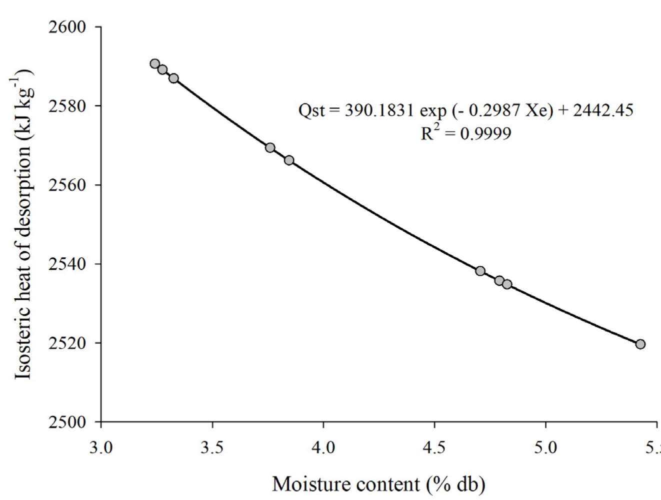

Figure 2. Experimental and estimated isosteric integral heat of desorption values as a function of equilibrium moisture content for baru (*Dipteryx alata* Vogel) kernel.

Conclusions

The hygroscopic equilibrium moisture content of the baru kernel flour was directly proportional to the water activity and decreased with increasing temperature, for the same water activity value.

The Chung-Pfost model best represented the hygroscopicity of the baru kernel flour.

The integral isosteric heat of desorption of the baru kernel flour ranged from 2590.69 to 2519.52 for the moisture content range of 3.24 to 5.43% db.

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Compliance with Ethical Standards

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