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FITTINGS OF ADSORPTION ISOTHERM MODELS AND THERMODYNAMIC PROPERTIES OF URUNDAY SEEDS

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KEYWORDS

water activity, isokinetic, sorption isotherms, *Myracrodruon urundeuva*, relative humidity.

ABSTRACT

This study aimed to obtain and evaluate thermodynamic properties and hygroscopic equilibrium isotherms of urunday seeds as a function of air relative humidity and temperature, as well as identify the best-fitted mathematical models for adsorption phenomenon. A static-gravimetric method was used to obtain hygroscopic equilibrium moisture content. Seeds were placed in airtight containers with different saturated saline solutions for relative humidity control. These were then kept in BOD incubator chambers at 10, 20, 30, and 40 ºC. Hygroscopic equilibrium moisture content decreased as temperature increased, for the same water activity. The Sigma-Copace model best-fitted seed adsorption data for equilibrium water activity intervals between 0.1129 and 0.8232 (dry basis). Both vaporization latent-heat and differential sorption entropy increased with decreasing hygroscopic equilibrium moisture content. Adsorption process in urunday seeds was controlled by enthalpy.

INTRODUCTION

Urunday (*Myracrodruon urundeuva* Allem.) is a tree species native to Brazil, naturally distributed in its northeast, southeast, and midwest regions (Carmello-Guerreiro & Paoli, 1999). It is a hardwood with characteristics such as hardness, elasticity, termite resistance, high density, long life, and excellent polishing, being valued in construction and furniture markets, as well as for fencing (Lorenzi, 2014). Besides, its bark can be used as tinting material (Carvalho, 2006), and its inner bark has anti-inflammatory and healing effects (Albuquerque et al., 2004).

Urunday has been exploited extractively and disorderly due to its properties and vast uses, with a strong impact on natural populations, compromising its preservation within natural habitats (Lima et al., 2017).

Conversely, a regular supply of quality seeds can favor urunday conservation, as germplasm conservation for future generations and success of forest plantations directly depend on the quality of seeds used for seedling production (Caldeira et al., 2012). In this sense, studies on seed storage conditions become essential to ensure viability and vigor for extended periods (Guedes et al., 2012).

Agricultural products in general, including seeds, undergo undesirable changes when stored, most often associated with their moisture content. Thus, each product behavior should be known under different temperature and relative humidity conditions to minimize oxidation, respiration, and growth of microorganisms (Labuza & Altunakar, 2008).

Seeds tend to remain in balance with temperature and relative humidity conditions due to their hygroscopic characteristics. Such traits allow analysis through curves, known as sorption isotherms. For instance, these curves can be used to determine the relationship between a product moisture content and its water activity at a given temperature (Corrêa et al., 2014; Bustos-Vanegas et al., 2018; Zeymer et al., 2019).

Moreover, sorption isotherms enable calculation of some thermodynamic properties such as isosteric heat of sorption, as well as differential entropy and enthalpy. Determining these thermodynamic properties in drying processes of agricultural products (grains and seeds) is essential for designing equipment, calculating required energy, studying adsorbed water properties, besides evaluating microstructures and physical phenomena

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occurring on the surface of such products (Oliveira et al., 2014; Campos et al., 2019).

Therefore, this study was carried out to characterize and evaluate thermodynamic properties, as well as determining and modeling hygroscopic equilibrium isotherms of urunday seeds, and identifying the mathematical models best fitting water adsorption phenomenon.

MATERIAL AND METHODS

Seeds of urunday (*Myracrodruon urundeuva*) were acquired from a commercial establishment in the city of Cáceres in Mato Grosso State (Brazil). They originated from northwestern São Paulo State (Brazil) and were processed by Arbocenter Comércio de Sementes in 100-g packages, with an initial moisture content of 0.04 g g^{-1} (db). A static-gravimetric method was used to determine hygroscopic equilibrium moisture content.

Samples of 3 ± 0.2 g seeds were placed into airtight containers, totaling three replications. These seeds were surrounded by an inert and permeable fabric (voile) for free passage of air.

Air temperature and relative humidity were monitored using a digital thermo-hygrometer, with a probe inserted into the airtight containers.

Different saturated saline solutions were used to control environmental conditions inside the airtight containers with seeds, which were submitted to controlled temperatures (10, 20, 30, and 40 °C) in BOD incubator chambers (Table 1).

TABLE 1. Relative air humidity (%) as a function of saturated solutions and temperatures used to determine hygroscopic equilibrium.

Saturated saline solution	Temperature (°C)			
	10	20	30	40
Lithium chloride	11.29	11.31	11.28	11.21
Potassium carbonate	43.14	43.16	43.17	43.17
Magnesium nitrate	57.36	54.38	51.40	48.42
Potassium iodide	72.11	69.90	67.89	66.09
Potassium chloride	86.77	85.11	83.62	82.32

Equilibrium moisture content was determined by weighing the samples every 24 hours. Hygroscopic equilibrium was reached when the mass remained unchanged after two consecutive weighings. Then, seed moisture content was determined in an oven at 105 °C for 24 h (Brasil, 2009).

Mathematical models used to describe hygroscopicity of agricultural products were fitted to equilibrium moisture content data for urunday seeds, whose expressions are shown in Table 2.

TABLE 2. Models used to verify the behavior of adsorption isotherms of urunday seeds.

Model	Equation	Reference	Eq.
Chung Pfost	$X_e=a-b[-(T+c)\ln(a_w)]$	Chung & Pfost et al. (1967)	(1)
Copace	$X_e=\exp[a-(bT)+(ca_w)]$	Corrêa et al. (1995)	(2)
Modified Henderson	$X_e=\{\ln(1-a_w)/[(-aT+b]\}^{1/c}$	Henderson (1952)	(3)
Oswin	$X_e=(a-bT)/[(1-a_w)/a_w]^{1/c}$	Oswin (1945)	(4)
Sabbab	$X_e=a\,(a_w^{b}/T^{c})$	ASAE (2003)	(5)
Sigma-Copace	$X_e=\exp\{a-(bT)+[c\exp(a_w)]\}$	Corrêa et al. (1995)	(6)

Where:

Xe is the product moisture content (db);

a_w is the water activity (decimal);

T is the ambient air temperature (°C);

T_{abs} is the absolute ambient air temperature (K), and

a, b, and c are the product nature parameters.

The mathematical models were fitted using non-linear regression analyses by the Gauss-Newton method, through STATISTICA 7.0® software.

Experimental data were compared with estimates from each model for representativeness. To this end, a chi-square test (χ^2) was applied, and coefficient of determination (R^2), relative mean error (P), and estimated mean error (SE) were estimated using eqs (10), (11) and 12, respectively.

$$\chi^2 = \sum\left(\frac{Y-\hat{Y}}{DF}\right)^2 \tag{10}$$

$$P = \frac{100}{n}\sum_{i=1}^{n}\left(\frac{|Y-\hat{Y}|}{Y}\right) \tag{11}$$

$$SE = \sqrt{\frac{\sum_{i=1}^{n}(Y-\hat{Y})^2}{DF}} \tag{12}$$

Where:

n is the number of experimental observations;

Y is the experimental value;

$\hat{Y}$ is the model estimate, and

DF is the degree of freedom of the model.

According to Mohapatra & Rao (2005), relative mean error is a parameter used to recommend or reject a model. Relative mean errors reflect observed value deviations relative to the model estimated curve (Kashaninejad et al., 2007). Estimated mean error, on the other hand, demonstrates a model's ability to describe a particular physical process accurately; the lower its value, the better the model-fit quality regarding experimental data (Siqueira et al., 2012).

Net isosteric sorption heat (or differential enthalpy) was calculated for each equilibrium moisture content using the Clausius-Clayperon expression (Iglesias & Chirife 1976):

$$\frac{\partial \ln(a_w)}{\partial T} = \frac{\Delta h_{st}}{R T_a^2} \tag{13}$$

Where:

T_a is the absolute temperature (K);

Δh_{st} is the net isosteric sorption heat (kJ kg^{-1}), and

R is the universal gas constant (8,314 kJ $kmol^{-1}$ K^{-1}) for water vapor of 0.4619 kJ kg^{-1} K^{-1}.

Integrating [eq. (13)] and assuming that the net isosteric sorption heat is independent of temperature, this value was obtained for each equilibrium moisture content according to [eq. (14)] (Wang & Brennan, 1991):

$$\ln(a_w) = -\left(\frac{\Delta h_{st}}{R}\right).\frac{1}{T_a} + C \tag{14}$$

Where:

C is the model coefficient.

Water activity, temperature, and equilibrium moisture content were obtained from adsorption isotherms of urunday seeds. And integral isosteric sorption heat was obtained by summing net isosteric sorption heat and free-water vaporization latent-heat, as in [eq. (15)]:

$$Q_{st} = \Delta h_{st} + L \tag{15}$$

Where:

Q_{st} is the integral isosteric sorption heat (kJ kg^{-1}), and

L is the vaporization latent-heat of free water (kJ kg^{-1}).

The free-water vaporization latent-heat (L, kJ kg^{-1}), which is required for calculation of Q_{st}, was obtained by the mean temperature (T, °C) of the range under study, according to [eq. (16)]:

$$L = 2502.2 - 2.39T \tag{16}$$

Differential sorption entropy was calculated from the Gibbs-Helmholtz equation (Rizvi, 1995), as in [eq. (17)]:

$$\Delta S = \frac{\Delta h_{st} - \Delta G}{T_a} \tag{17}$$

Where:

ΔS is the differential sorption entropy (kJ kg^{-1} K^{-1}), and

ΔG is the Gibbs free energy (kJ kg^{-1}).

Gibbs free energy was calculated using [eq. (18)]:

$$\Delta G = R\,.\,T\,.\ln(a_w) \tag{18}$$

The effects of changes in water sorption on free energy are usually accompanied by changes in enthalpy and entropy values. Thus, replacing [eq. (18)] in (17) and rearranging, we have [eq. (19)]:

$$\ln = \frac{\Delta h_{st}}{R\,.T_a} - \frac{\Delta S}{R} \tag{19}$$

The calculated values of differential enthalpy (Δh_{st}) and entropy (ΔS) of sorption were correlated by [eq. (20)] (Beristain et al., 1996):

$$\Delta h_{st} = T_B\,(\Delta S) + \Delta G_B \tag{20}$$

Where:

T_B is the isokinetic temperature (K), and

ΔG_B is the Gibbs free energy at isokinetic temperature (kJ kg^{-1}).

The isokinetic temperature was compared with the harmonic mean temperature used to determine sorption isotherms to confirm the existence of compensation and determine the control of the process by enthalpy or entropy, according to [eq. (21)]:

$$T_{hm} = \frac{n}{\sum\left(\frac{1}{T}\right)} \tag{21}$$

Where:

T_{hm} is the harmonic mean temperature (K), and

n is the number of used temperatures.

According to Krug et al. (1976), linear chemical compensation or compensation theory only exists if the isokinetic temperature (T_B) is different from the harmonic mean temperature (T_{hm}). An approximate confidence interval of (1−α) 100% for isokinetic temperature was calculated by the following equation:

$$T_B = \hat{T} \pm t_{m-2,\alpha/2\sqrt{Var(T_B)}} \tag{22}$$

Where:

$$\hat{T}_B = \frac{\sum\left(\Delta h_{st} - \overline{\Delta h_{st}}\right)\left(\Delta S - \overline{\Delta S}\right)}{\sum\left(\Delta S - \overline{\Delta S}\right)^2} \tag{23}$$

and

$$Var(T_B) = \frac{\sum\left(\Delta h_{st} - \overline{\Delta G_B} - \hat{T}_B \Delta S\right)^2}{(m-2)\sum\left(\Delta S - \overline{\Delta S}\right)^2} \tag{24}$$

Where:

m is the number of enthalpy and entropy data pairs;

$\overline{\Delta h_{st}}$ is the mean enthalpy (kJ kg^{-1}), and

$\overline{\Delta S}$ is the mean entropy (kJ kg^{-1}).

In the verification of the enthalpy-entropy linear isokinetic, if $T_B > T_{hm}$, the process is controlled by enthalpy, otherwise ($T_B < T_{hm}$) the process is controlled by entropy; if T_{hm} is in the isokinetic temperature range, it is considered an experimental error (Corrêa et al., 2012).

RESULTS AND DISCUSSION

Table 3 shows that only the Sigma-Copace model exhibited a reduced relative mean error, below 10%. Therefore, the deviation can be considered acceptable. Also, this model was the one that stood out the most for having the smallest SEs compared to the others.

TABLE 3. Coefficients of models fitted to hygroscopic equilibrium moisture contents of urunday seeds, with their respective coefficients of determination (R^2, %), estimated error (SE, decimal), and relative mean error (P, %), as well as chi-square test (χ^2).

MODEL	COEFFICIENT			R^2	P	SE	X^2
	A	B	C				
CHUNG PFOST	0.2989	0.04910	65.2245	94.29	15.09	0.0164	0.00027
SABBAB	0.2462	0.95855	0.0859	87.27	22.76	0.0241	0.00058
COPACE	−3.3046	0.00376	1.9479	96.12	11.55	0.0136	0.00018
OSWIN	0.0005	−0.000003	0.0170	38.78	48.22	0.0455	0.00207
MODIFIED HENDERSON	0.1122	746.072	0.1122	85.58	24.68	0.0255	0.00065
SIGMA-COPACE	−4.1475	0.00340	1.0761	97.88	8.53	0.0100	0.00010

The chi-square test showed that all models were within 95% confidence interval, but when comparing magnitudes, the Sigma-Copace model had the lowest value. Low chi-square values show the better fit of a model (Siqueira et al., 2012).

The coefficients of determination of the models Sabbab, Oswin, and Modified Henderson were below 88%, and the other models had CVs above 94%. Despite being an additional analysis, in this case, the highest CV of the Sigma-Copace model reinforces its good fit to the data.

Among all tested models, Sigma-Copace stood out and met statistical parameters and hence selected for prediction of the hygroscopic balance of urunday seeds. Similar and satisfactory results were also obtained for this model in crambe fruits (Costa et al., 2015), Barbados nut seeds (Chaves et al., 2015), and Bahia rosewood and angico seeds (Mesquita Basílio et al., 2015).

The isotherms in Figure 1 show the good fit of the Sigma-Copace model to urunday seed hygroscopic balance. We noticed that as temperature increased moisture content decreased at a constant water activity. Besides, as water activity increased at a constant temperature, equilibrium moisture content tended to increase as well. Most agricultural products follow this same behavior. Once the Sigma-Copace model is fundamentally exponential, there is no isotherm inflection to predict moisture contents when water activity tends to zero, which limits this model to a restricted water activity range of urunday seeds.

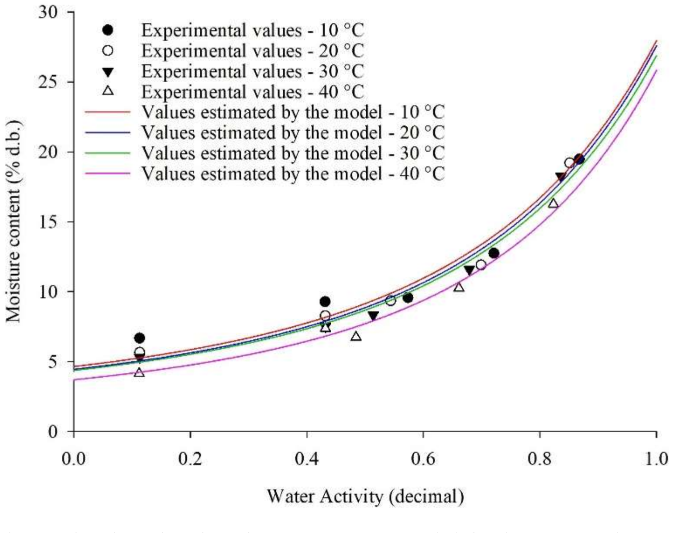

FIGURE 1. Experimental and estimated values by the Sigma-Copace model for hygroscopic equilibrium of urunday seeds under different temperature and water activity conditions

Figure 2 shows the experimental and estimated values of integral isosteric heat of adsorption (A), differential sorption entropy (B), and linear enthalpy-entropy relationship (C) as a function of the equilibrium moisture content of urunday seeds.

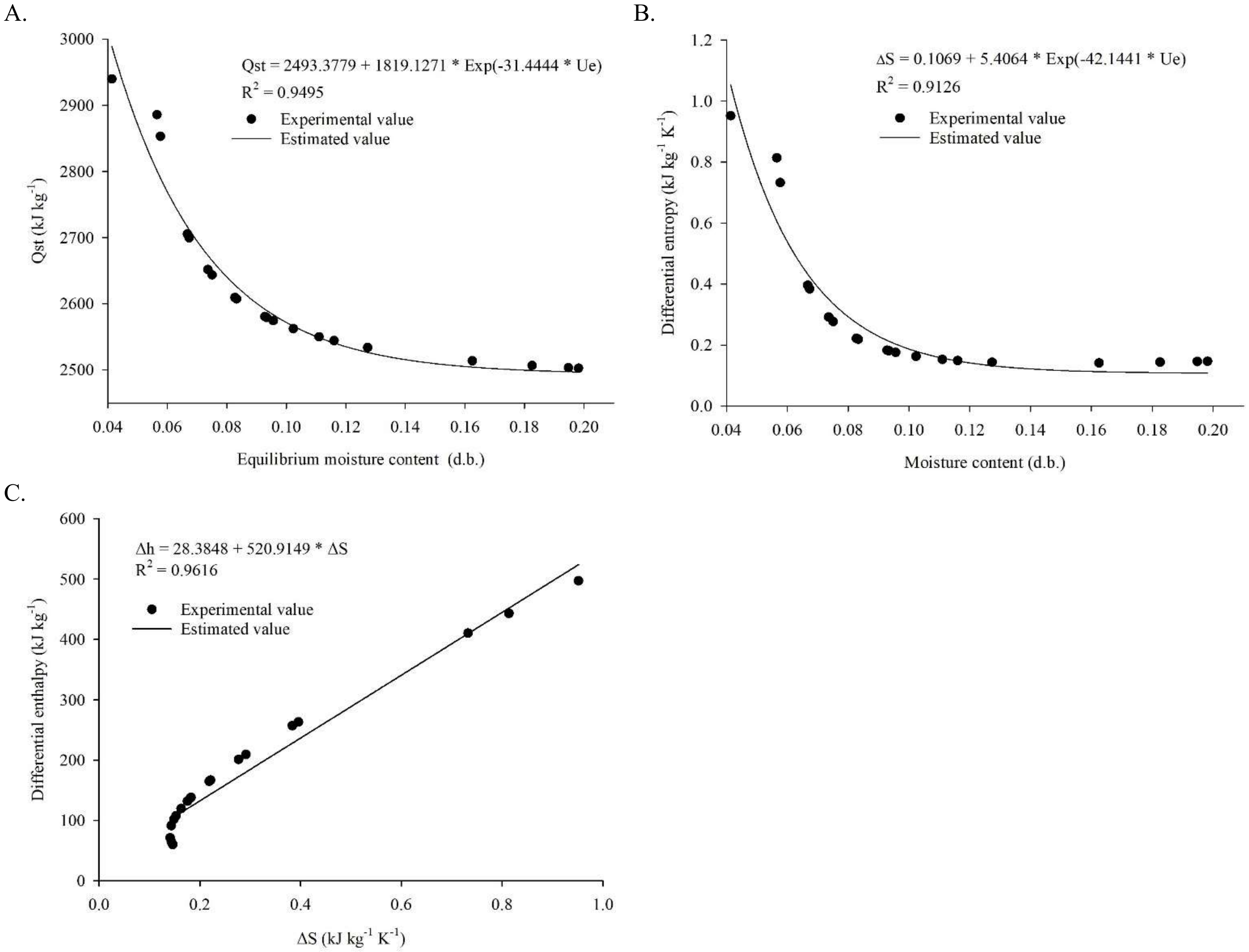

FIGURE 2. Experimental and estimated values of integral isosteric sorption heat (A), differential sorption entropy (B), and linear enthalpy-entropy relationship (C) for sorption process as a function of equilibrium moisture content

Figure 2A shows that the lower the moisture content, the higher the energy to remove water from the product, which is represented by integral isosteric sorption heat (Q_{st}). Other researchers have already observed such behavior for several other agricultural products, such as *Coffea canephora* fruits (Corrêa et al., 2014), baru fruits (Resende et al., 2017), pequi diaspores (Sousa et al., 2016), and butternut pumpkin seeds (Teixeira et al., 2018). It occurs because a higher amount of energy is needed compared to that required for wetter products, so that water could be removed from seeds with low moisture content (Brooker et al., 1992).

Differential entropy and enthalpy were strongly related to seed moisture content (Figure 2B) because there is a marked decrease in differential entropy as moisture content increases, with a trend to stabilize at high moisture contents. Goneli et al. (2010) described this behavior with okra seeds, showing that a high-water activity led to fewer sites available for bonds between molecules, resulting in decreased mobility of water molecules.

The isokinetic theory for water adsorption of urunday seeds can be considered valid based on the good fit of the linear regression for the relationship between differential enthalpy and differential entropy (Figure 2C).

The validity of the enthalpy-entropy isokinetic theory is tested by comparing the isokinetic temperature (T_B) with the harmonic mean temperature (T_{hm}) of the studied range, used to determine the desorption process in urunday seeds.

The isokinetic temperature for water desorption was 520.91 K, ranging from 563.58 to 478.25 K. The calculated harmonic mean was 297.73 K, which was different from the described isokinetic temperatures, confirming enthalpy-entropy compensation and showing that enthalpy controls the process. These results are in line with those of other studies by several researchers, who used the isokinetic theory for different agricultural products (Oliveira et al., 2014; Sousa et al., 2015; Silva et al., 2015; Resende et al., 2017; Corrêa et al., 2017).

CONCLUSIONS

The hygroscopic equilibrium moisture content of urunday seeds increased with increasing water activity but decreased with increasing temperature at a constant water activity.

The Sigma-Copace model can be used to describe adsorption isotherms of urunday seeds.

Both vaporization latent-heat and differential sorption entropy increased with decreasing hygroscopic equilibrium moisture contents.

Enthalpy controls the adsorption process in urunday seeds.

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