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Thermodynamic approach on water vapor sorption of *Curcuma longa* L. flour

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ABSTRACT

In this study, water vapor sorption isotherms of *Curcuma longa* were determined over ranges of storage (5–40 °C) and drying (40–80 °C) temperatures. From the sorption isotherms, thermodynamic parameters (net isosteric heat of sorption, differential enthalpy and entropy, and Gibbs free energy) were estimated, and the enthalpy-entropy compensation theory was evaluated. Water vapor sorption isotherms of turmeric flour were determined using the gravimetric-statistical method. The net isosteric heat of sorption, enthalpy, entropy, and Gibbs free energy were determined via the Clausius-Clapeyron equation. The GAB model showed the best fit to describe the isotherms of turmeric flour ($R^2_{adj} \geq 0.998$; RMSE ≤ 0.0065; $X^2 \leq 4.22 \bullet 10^{-06}$). The equilibrium moisture content (X_{eq}) of turmeric flour decreased with increasing temperature. The isotherms for all temperatures exhibited type II behavior. The isosteric heat of sorption, differential enthalpy, and entropy decreased with increasing temperature. It was possible to validate the enthalpy-entropy compensation theory, and the process was driven by enthalpy, characterizing it as non-spontaneous.

1. Introduction

Turmeric (*Curcuma longa* Linn.) belongs to the Zingiberaceae family and is a perennial plant that thrives in tropical and humid climates, originating in India and Southeast Asia (Kocaadam & Şanlier, 2017). Its rhizome has a yellow-orange color and is widely used as a spice in Latin American countries, and in folk medicine, especially Ayurveda and Chinese medicine, particularly in the treatment of minor ailments, such as abdominal pain, and more complex diseases, such as cardiovascular and respiratory pain (Zhang & Kitts, 2021). In addition, turmeric is widely used in the food industry as a natural dye to replace synthetic dye, being applied in canned products, mustard, cheese, and in the preparation of curry (Lima et al., 2022).

Due to its application as a dye, turmeric is stored and marketed as powder. The preparation of the rhizome to obtain the powder is done by drying and contributes to increasing the shelf life of the product, as it reduces moisture content and water activity (a_w), which diminishes the possibility of development of microorganisms and minimizes the chances of physical, chemical and sensory reactions that would affect the characteristics of the rhizome (Lima et al., 2022). Thus, turmeric is traditionally dehydrated for commercialization.

Studies on the preservation of dehydrated products can be conducted by obtaining water sorption isotherms that allow predicting chemical reactions that affect the chemical, nutritional and microbiological characteristics of the food (Collazos-Escobar et al., 2022; Domínguez-Chávez et al., 2023; Staniszewska et al., 2021). Thus, isotherms can be an important tool to understand the storage and durability of the product during its commercialization (Betiol et al., 2020). Water sorption isotherms serve as a crucial tool for ingredient selection aimed at adjusting a_w, enhancing stability, and estimating moisture uptake or loss in the material when packaged in different packages (Labuza & Altunakar, 2020).

From additional calculations, based on thermodynamic parameters, such as enthalpy and entropy, and from the sorption isotherm pattern, it is possible to predict the microstructure of the material and suggest theoretical interpretations for the matrix-water interactions (Borges-Machado et al., 2024; Rosa et al., 2021). Gibbs free energy can also be studied and calculated, providing information on the spontaneity of the

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sorption process as well as the absorbent affinity of water (Rizvi, 2014).

The novelty of this study lies in determining the water vapor sorption isotherms of turmeric flour under a wide range of storage and drying temperatures, combined with the determination of thermodynamic parameters and the evaluation of the enthalpy-entropy compensation theory. We hypothesize that the water vapor sorption of turmeric flour can be affected by temperature and that thermodynamic characterization will provide important information to control the stability and storage behavior of turmeric flour, which is essential for its commercialization and application in the food industry.

Thus, the aim of this study was to determine the water vapor sorption isotherms of *Curcuma longa* L. flour under different storage (5–40 °C) and drying (40–80 °C) temperatures, in addition to determining the main thermodynamic parameters (net isosteric heat of sorption, enthalpy and differential entropy and Gibbs free energy) to evaluate the enthalpy-entropy compensation theory and deepen the understanding of turmeric flour's stability and storage properties.

2. Material and methods

2.1. Raw material

Rhizomes of turmeric (*Curcuma longa* L.) were obtained in the municipality of Rio Verde, GO, Brazil, with geographic location 17° 51′10.55″ S and 50°50′ 20″ W, altitude of 663 m. Turmeric was randomly collected using a hoe and by manual uprooting and then transported to Instituto Federal Goiano – Campus of Rio Verde, GO, Brazil. The rhizomes were selected, cleaned under running water and sanitized (0.001 mol L^{-1} sodium hypochlorite solution for 10 min), peeled, sliced into uniform pieces (59.46 ± 0.30 mm long, 15.62 ± 0.30 mm wide and 2.63 ± 0.01 mm thick) and stored under refrigeration (4 ± 2 °C).

For dehydration, the rhizomes were dried in an air circulation oven at temperature of 65 °C until reaching constant mass, as described by Lima et al. (2022). After drying, the rhizomes were crushed in a cyclone-type rotor mill (TECNAL® brand TE-651/2 model, with an 18-mesh stainless steel sieve) to obtain the flour, which was stored at room temperature until the analysis was performed.

2.2. Methods

2. 2. 1. Nutritional composition

Moisture content was determined by the gravimetric method in an oven (105 ± 1 °C) according to the AACC method (44-15 A). Ash, protein and lipid contents were determined by the methods AOAC n°. 923.03, AOAC n°. 960.52 with nitrogen conversion factor of 6.25, and AOAC n°. 920.39 (AOAC, 2019), respectively. Carbohydrates were calculated by difference. All analyses were performed in triplicate.

2. 2. 2. Determination of sorption isotherms

The turmeric flour was placed in a 10 mL beaker, approximately 1 g, inside desiccators containing salt solutions and maintained at storage temperatures (5, 10, 20, 30 and 40 °C) in a BOD chamber (MA415, Marconi, Piracicaba, Brazil) and drying temperatures (40, 50, 60, 70 and 80 °C) in an oven (MA030, Marconi, Piracicaba, Brazil). As turmeric is a widely used product around the world, the storage temperature range was selected to represent various conditions that turmeric powder can be subjected to.

The relative humidity (RH = 100•a_w) of the samples varied from 7.43 to 98.48% and this variation was obtained using saturated saline solutions. The solutions used were prepared with potassium nitrate, ammonium sulfate, magnesium chloride, magnesium nitrate, potassium chloride, sodium bromide, lithium iodide, sodium iodide, lithium iodide, lithium chloride, potassium carbonate, sodium chloride, potassium sulfate, potassium iodide from Sigma-Aldrich (St. Louis, MO, USA). The isotherms were determined as described by Spiess and Wolf (1983), with weighing performed on analytical balance (AUW220D, Shimadzu, Japan), in triplicate, until the samples reached a constant mass and did not show difference in mass, on a dry basis, of 0.001 g/g of dry solids.

To prevent microbial deterioration, a beaker containing toluene was placed inside desiccators with a relative humidity exceeding 60% ($a_w >$ 0.60). The experiments persisted for six weeks, during which data collection was conducted. The salt solutions utilized to establish atmospheres with varying relative humidity levels (refer to Table 1) were prepared following the methodologies outlined by Labuza (1963), Martins et al. (2023), and Borges-Machado et al. (2024a).

2. 2. 3. Modelling of sorption isotherms

The equilibrium moisture content (X_{eq}) values were graphically depicted as a function of water activity (a_w) utilizing the Origin 2023 software (OriginLab Corporation, Northampton, MA). Theoretical sorption isotherm models (GAB), semi-empirical models (Halsey and Henderson) and empirical models (Peleg and Oswin) were used for linear regression analysis, as shown in Table 2. The purpose of comparing these models is to provide a simplified mathematical representation of water sorption behavior, facilitating prediction and analysis under various conditions. These models are widely used due to their simplicity and ability to offer good fits for different materials and experimental conditions (Al-Muhtaseb et al., 2002; Sanches et al., 2023; Nayab et al., 2023; Borges-Machado et al., 2024a).

C, k, k_1, k_2, n1, n2, M, N, h_1, h_2, H1 and H2 are constants of the models, X_m is moisture content of the monolayer (kg of water • kg dry $matter^{-1}$), X_{eq} is equilibrium moisture content, and a_w is water activity.

The assessment of the quality of nonlinear fit of water vapor sorption models to experimental data was conducted based on several criteria, including the adjusted coefficient of determination (R^2_{adj}), residual analysis of reduced chi-square (χ^2), and root mean square error (RMSE). Additionally, Akaike (AIC) and Bayesian (BIC) information criteria were computed using Equations (6) and (7) to determine the most suitable model for turmeric flour water sorption. This comparison involved evaluating the fit results of the five models analyzed, as outlined by Akaike (1974) and Stone (1979). AIC and BIC incorporate a fit quality term and a penalty to prevent overfitting, thereby offering a standardized approach for balancing sensitivity and specificity (Wolfinger, 1993).

$$AIC = -2\log() + 2p \tag{6}$$

$$BIC = -2\log() + p\ln(n) \tag{7}$$

Where n is the total value of observations carried out, p is the total of estimated parameters, and $\mathscr{L}\left(\hat{\theta}\right)$ is the value of the likelihood function relative to the model parameters.

2. 2. 4. Ideal a_w

A third-degree polynomial equation was applied to model the sorption isotherm from the experimental sorption points, which were plotted on the same graph. This calculation process involved performing a polynomial decomposition of the equilibrium moisture content (X_{eq}) based on water activity (a_w). The analysis aimed to determine the inflection point of the second derivative of X_{eq}, which indicates the optimal a_w for storage (Lahnine et al., 2016).

2. 2. 5. Thermodynamic properties

With the aid of the Clausius-Clapeyron equation (Equation (8)), considering different values of X_{eq}, it was possible to calculate the net isosteric heat of sorption (qst) (Rizvi, 2014).

$$\left.\frac{\partial(\ln aw)}{\partial\left(\frac{1}{T}\right)}\right|_{Xeq} = -\frac{Q_{st} - \lambda}{R} = -\frac{q_{st}}{R} \tag{8}$$

Table 1
Water activity of saturated salt solutions at different temperatures (Labuza, 1963).

Salt	Water activity								
	Storage temperatures				Storage/Drying	Drying temperatures			
	5 °C	10 °C	20 °C	30 °C	40 °C	50 °C	60 °C	70 °C	80 °C
LiBr	0.0743	0.0714	0.0661	0.0616	0.0580	0.0553	0.0533	0.0523	0.0520
LiCl	0.1126	0.1129	0.1131	0.1128	0.1121	0.1110	0.1095	0.1075	0.1051
LiI	0.2168	0.2061	0.1856	0.1657	0.1455	0.1238	0.0998	0.0723	–
$MgCl_2$	0.3366	0.3347	0.3307	0.3244	0.3160	0.3054	0.2926	0.2777	0.2605
NaI	0.4242	0.4183	0.3965	0.3615	0.3288	0.2921	0.2595	0.2357	0.2252
K_2CO_3	0.4313	0.4314	0.4315	0.4317	–	–	–	–	–
$Mg(NO_3)_2$	0.5886	0.5736	0.5438	0.5140	0.4842	0.4544	–	–	–
NaBr	0.6351	0.6215	0.5914	0.5603	0.5317	0.5093	0.4966	0.4970	0.5033
KI	0.7330	0.7211	0.6990	0.6789	0.6609	0.6449	0.6311	0.6193	0.6097
NaCl	0.7565	0.7567	0.7547	0.7509	0.7468	0.7443	0.7450	0.7506	0.7629
$(NH_4)_2SO_4$	0.8242	0.8206	0.8134	0.8063	0.7991	0.7920	–	–	–
KCl	0.8767	0.8677	0.8511	0.8362	0.8232	0.8120	0.8025	0.7949	0.7890
KNO_3	0.9627	0.9596	0.9462	0.9231	0.8903	0.8478	–	–	–
K_2SO_4	0.9848	0.9818	0.9759	0.9700	0.9641	0.9582	–	–	–

Table 2
– Theoretical (GAB), semi-empirical (Halsey and Henderson), and empirical (Peleg and Oswin) sorption models were employed in the analysis of sorption isotherms for turmeric flour.

Model name	Equation	Reference	
GAB	$Xeq = \frac{X_m \bullet C \bullet k \bullet a_w}{(1-k\bullet a_w)(1+(C-1)k \bullet a_w)}$	Lomauro et al. (1985)	(1)
Henderson	$Xeq = \left(-\frac{1}{H1} \bullet \ln(1-a_w)\right)^{\frac{1}{H2}}$	Bouquet et al. (1978)	(2)
Halsey	$Xeq = (-h_1 \bullet \ln(a_w))^{-\frac{1}{h_2}}$	Halsey (1948)	(3)
Peleg	$Xeq = k_1 \bullet a_w^{n1} + k_2 \bullet a_w^{n2}$	Peleg (1993)	(4)
Oswin	$Xeq = M \bullet \left(\frac{a_w}{1-a_w}\right)^N$	Oswin (1946)	(5)

Where Q_{st} is the integral heat of sorption, λ is the latent heat of vaporization of water, and R is the universal gas constant (8.314 J mol^{-1} K^{-1}).

Differential enthalpy (ΔH) and differential entropy (ΔS) were calculated using Equation (9) (derived from the Gibbs-Helmholtz equation), according to Rizvi (2014).

$$\ln a_w = \left(\frac{\Delta H}{R}\right) \bullet \left(\frac{1}{T}\right) - \frac{\Delta S}{R} \quad (9)$$

Enthalpy of sorption (ΔH) and entropy (ΔS) were linearly correlated, according to Equation (10) of the enthalpy-entropy compensation theory (Polachini et al., 2016).

$$\Delta H = T_B \bullet (\Delta S) + \Delta G_B \quad (10)$$

Where T_B is the isokinetic temperature, i.e., the temperature at which all sorption phenomena occur at the same rate; ΔG_B is the Gibbs free energy at isokinetic temperature. Gibbs free energy can provide information on whether water vapor sorption is occurring by a spontaneous process ($-\Delta G_B$) or not ($+\Delta G_B$).

In order to validate the theory proposed in this study, a comparison of the isokinetic temperature with the mean harmonic temperature (T_{hm}) was performed according to Equation (11) and Krug et al. (1976).

$$T_{hm} = \frac{n}{\sum_{i=1}^{n}\left(\frac{1}{T}\right)} \quad (11)$$

A confidence interval of (1 – α) x 100% was calculated from Equations (12)–(14).

$$T_B = T_B \pm t_{m-2,\alpha/2}\sqrt{Var(T_B)} \quad (12)$$

Where:

$$T_B = \frac{\sum(\Delta H - \overline{\Delta H}) \bullet (\Delta S - \overline{\Delta S})}{\sum(\Delta S - \overline{\Delta S})^2} \quad (13)$$

$$Var(T_B) = \frac{\sum\left(\Delta H - \overline{\Delta G_B} - T_B \bullet \Delta S^2\right)}{(m-2) \bullet \sum(\Delta S - \overline{\Delta S})^2} \quad (14)$$

Where m is the number of data pairs (ΔH, ΔS), $\overline{\Delta H}$ (kJ mol^{-1}) is the mean enthalpy and $\overline{\Delta S}$ (kJ kg^{-1} K^{-1}) is the mean entropy.

2. 2. 6. Statistical analysis

Nutritional characterization and parameters of the sorption isotherm model were computed based on three replicates and reported as mean values with standard deviation. The quality of fit was assessed utilizing the adjusted coefficient of determination (R^2_{adj}) and reduced chi-square (X^2), acquired through the use of OriginPro 8.0 software (OriginLab Corporation, Northampton, MA).

3. Results and Discussion

3.1. Nutritional characterization

Turmeric flour had moisture content of 10.98 ± 0.28 g 100 g^{-1}. For the nutritional composition, the flour showed, in wet basis, 5.65 ± 0.73 g of ashes, 8.80 ± 0.24 g of proteins, 9.37 ± 1.09 g of lipids, and 61.44 ± 1.80 g, 100 g^{-1} of carbohydrates in 100 g of turmeric flour. Other authors have reported that turmeric had 6–13% moisture, 60–70% carbohydrates, 6–8% proteins and 5–10% lipids which are similar to those determined in the present study (Kotha & Luthria, 2019; Nelson et al., 2017; Prasad et al., 2014).

3.2. Sorption isotherms

The sorption isotherms of *Curcuma longa* were obtained in a_w ranges of 0.05–0.98 for storage temperatures (Fig. 1 - A) and 0.05 to 0.96 for drying temperatures (Fig. 1 - B). The X_{eq} values (Supplementary Material - Table S1) ranged from 0.024 ± 0.001 to 0.701 ± 0.003 g of water • g^{-1} of dry matter for the temperature range studied.

When analyzing the isotherms at constant temperature, it is noted that there was an increase in the X_{eq} of the turmeric flour due to the increase in a_w. On the other hand, it is possible to observe that, at the same water activity, the X_{eq} of turmeric flour decreased with increasing temperature, which indicates a decrease in its hygroscopicity. This behavior is probably related to the increase in the kinetic energy and state of excitement of the water molecules (Al-Muhtaseb et al., 2002)

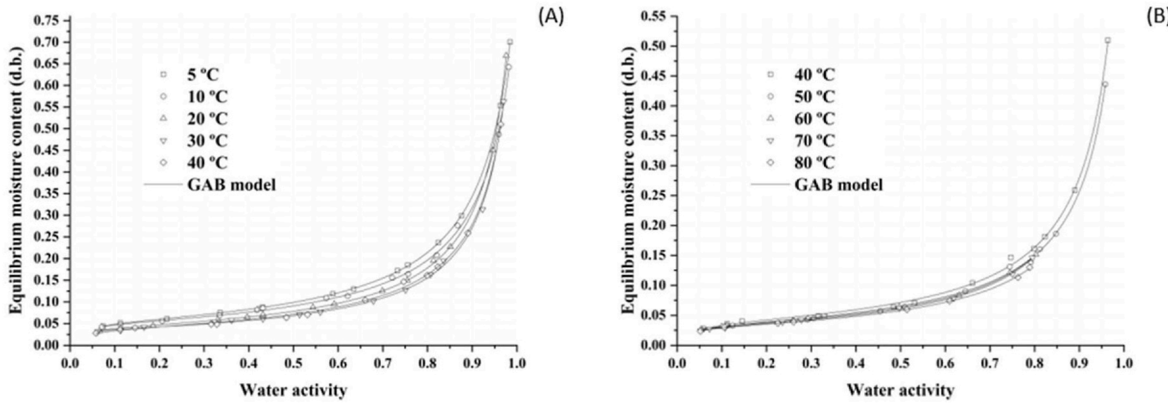

Fig. 1. Sorption isotherms of turmeric flour at storage (A) and drying (B) temperatures.

present in the turmeric flour, due to the increase in temperature, which probably reduced the attractive forces between the water molecules and binding sites to the solid matrix. This facilitated its separation from the binding sites, thus promoting a reduction in the moisture content of the turmeric flour.

However, regardless of temperature, the sorption curves of turmeric flour showed abrupt sorption of water in the range of $a_w \geq 0.7$. This is probably related to its chemical composition, especially the presence of carbohydrates (61.44%) and proteins (8.80%) in turmeric flour, which are hydrophilic structures capable of adsorbing water. Therefore, foods with these characteristics should be kept at a_w below 0.7, which would be possible at X_{eq} below 0.15 kg of water/kg of dry matter for storage temperatures and X_{eq} below 0.125 kg water/kg of dry matter for drying temperatures (Fig. 1 – A and B).

The sorption isotherms of turmeric flour exhibited type III characteristics, according to the BET classification (Brunauer et al., 1940). This behavior is typical of foods rich in soluble components, due to the high solubility of these components in water. At high a_w, dissolution of soluble components occurs, resulting in a significant increase in X_{eq}. Other foods also showed the same characteristics, such as maize by-products

Table 3
The constants and fitting parameters derived from the sorption equations for turmeric flour.

Model	Parameters	Temperature (°C)								
		5	10	20	30	40	50	60	70	80
GAB	X_m	0.054	0.049	0.042	0.036	0.038	0.035	0.033	0.031	0.030
	C	40.502	63.917	46.416	94.032	33.720	35.139	53.587	50.441	57.029
	K	0.937	0.940	0.961	0.965	0.959	0.960	0.977	0.986	0.968
	R^2_{adj}	0.999	0.998	0.999	0.998	0.998	0.998	0.999	0.999	0.998
	RMSE	0.0015	0.0065	0.0030	0.0056	0.0053	0.0041	0.0010	0.0012	0.0014
	X^2 x 10^6	2.101	42.280	8.957	31.447	27.527	16.920	1.075	1.436	1.837
	AIC	−173.953	−131.929	−153.655	−136.073	−126.915	−133.242	−109.335	−106.735	−88.084
	BIC	−175.842	−133.817	−155.544	−137.961	−129.655	−135.982	−118.547	−115.946	−101.100
Peleg	k_1	0.174	0.156	0.170	0.140	0.125	0.098	0.067	0.069	0.060
	n1	0.682	0.633	0.900	0.801	0.697	0.562	0.331	0.370	0.314
	k_2	0.608	0.570	0.696	0.641	0.572	0.506	0.263	0.283	0.212
	n2	10.852	10.739	14.423	14.083	10.948	9.424	4.934	5.460	4.552
	R^2_{adj}	0.996	0.993	0.993	0.989	0.991	0.994	0.999	0.998	0.997
	RMSE	0.013	0.015	0.016	0.015	0.013	0.008	0.002	0.002	0.002
	X^2 x 10^6	160.046	211.403	241.423	229.845	160.143	69.058	2.676	4.380	4.699
	AIC	−109.571	−105.675	−103.816	−104.504	−99.822	−110.756	−90.769	−86.336	−63.690
	BIC	−113.876	−109.980	−108.121	−108.809	−105.568	−116.503	−109.783	−105.350	−93.293
Oswin	A	0.116	0.104	0.085	0.072	0.074	0.067	0.071	0.070	0.065
	B	0.443	0.465	0.561	0.591	0.585	0.595	0.497	0.500	0.484
	R^2_{adj}	0.984	0.992	0.996	0.996	0.996	0.995	0.971	0.971	0.973
	RMSE	0.025	0.016	0.011	0.009	0.009	0.008	0.008	0.007	0.007
	X^2 x 10^6	610.930	267.216	126.308	89.019	77.551	65.156	56.492	54.832	42.667
	AIC	−97.366	−108.942	−119.433	−124.331	−116.545	−118.808	−79.495	−79.763	−70.798
	BIC	−97.848	−109.425	−119.916	−124.814	−117.516	−119.780	−83.703	−83.971	−76.560
Halsey	A	0.008	0.008	0.013	0.013	0.013	0.012	0.015	0.015	0.012
	B	2.071	1.968	1.633	1.544	1.547	1.509	1.418	1.392	1.456
	R^2_{adj}	0.975	0.986	0.994	0.998	0.996	0.998	0.996	0.995	0.996
	RMSE	0.031	0.021	0.014	0.007	0.009	0.006	0.003	0.003	0.003
	X^2 x 10^6	972.923	452.761	185.303	47.620	74.296	30.875	8.139	8.574	6.987
	AIC	−90.851	−101.560	−114.067	−133.090	−117.102	−128.518	−96.932	−96.463	−85.274
	BIC	−91.334	−102.043	−114.550	−133.573	−118.074	−129.489	−101.140	−100.671	−91.035
Henderson	A	5.707	5.941	5.047	5.362	5.501	5.896	28.429	29.724	36.189
	B	0.871	0.853	0.706	0.688	0.712	0.717	1.441	1.444	1.479
	R^2_{adj}	0.985	0.982	0.981	0.970	0.973	0.965	0.932	0.933	0.937
	RMSE	0.024	0.024	0.025	0.025	0.022	0.021	0.012	0.011	0.010
	X^2 x 10^6	576.493	574.818	621.585	644.918	486.253	428.884	133.987	126.959	99.940
	AIC	−98.178	−98.219	−97.123	−96.608	−92.679	−94.311	−71.722	−72.207	−63.989
	BIC	−98.661	−98.701	−97.606	−97.090	−93.651	−95.283	−75.930	−76.415	−69.751

(Marques et al., 2020), freeze-dried açaí (*Euterpe oleracea*) (Matuda et al., 2024), and dried cranberry powders (Staniszewska et al., 2021).

The models that had the best values of the coefficients of determination (R^2_{adj}), close to one, were GAB and Peleg (Table 3), in addition to showing low calculated values of X^2 and RMSE at all temperatures studied. AIC and BIC data were also used to identify the best model. From a phenomenological point of view, AIC and BIC avoid overfitting, where an overly complex model fits the experimental data perfectly but fails in its simplicity. Lower values of AIC and BIC indicate the model that best balances the accuracy of the fit to the experimental data and the simplicity of the model. When comparing the values of AIC and BIC of these models, GAB showed the lowest magnitudes, so it was selected to describe the sorption isotherms of turmeric flour. Henderson was the model that was least suitable for the parameters, due to the low values of coefficients of determination (R^2_{adj}). Delgado et al. (2016) also applied GAB model to the study of turmeric flour isotherm due to the better mathematical fit compared to other mathematical models. Furthermore, the GAB model constants assist in the sorption process due to their physical representation of the sorption process (Labuza & Altunakar, 2020).

The moisture content of the monolayer (X_m) described by the GAB model contributes to the description of information about the volume of water adhered to the polar sites of the food matrix, which can guarantee the stability of the product during storage (Kurozawa et al., 2015). The X_m values (Table 3) for turmeric flour were higher for lower temperatures (5, 10, 20 °C) compared to higher temperatures (30, 40, 50, 60, 70, 80 °C). According to Yogendrarajah et al. (2015) increasing the storage or drying temperature decreases the sorption surface area due to the filling of water binding points in the food monolayer.

When analyzing the behavior of X_m values with increasing temperature, it was observed that these values varied from 0.036 to 0.030 between 30 and 80 °C, indicating an extremely small difference, despite the wide temperature range. This suggests that, above 30 °C, the impact of temperature on the amount of water in the monolayer is less significant. This finding is relevant for the design of turmeric drying processes, as, together with drying kinetics and energy consumption, it allows optimizing the process by avoiding excessive energy consumption without a substantial reduction in the moisture content of the monolayer.

Storing food at a_w above the X_m value allows the occurrence of chemical and microbiological reactions and storing food at a_w below the X_m value allows an increase in the rates of lipid oxidation reactions, which can compromise the sensory and nutritional characteristics of the food (Labuza & Altunakar, 2020). Thus, turmeric flour can be stored at temperatures of 5–40 °C, at relative humidity of 36–54% (RH = $100.a_w$).

The C and K parameters of the GAB model are also important for the stability in the storage of the product; C indicates the strength of the interaction of water with the binding sites present on the surface of the food, and K represents an estimate of the interactions between the molecules in the multilayers of the product and the food matrix (Muzaffar & Kumar, 2016; Velázquez-Gutiérrez et al., 2015). The sorption of turmeric flour showed higher C values at temperatures of 10, 30 and 80 °C, confirming that at these temperatures the water molecules were more firmly bound to the binding sites on the surface of the sorbent, requiring more energy to be removed (Tavares & Noreña, 2021).

Gichau et al. (2020) on sorption isotherms of amaranth-sorghum grains and Bennaceur et al. (2015) on sorption isotherms of henna leaves described that the higher C value at high temperatures, such as 80 °C for turmeric flour in the present study, may be a result of the surface hardness of the material. This phenomenon arises when the rate of surface evaporation surpasses the rate of moisture diffusion within the product, thereby inducing changes in its physical structure and leading to the obstruction of pores. According to Blahovec and Yanniotis (2008), K values should be kept at $0 < K \leq 1$, as determined in this study, and C values should be greater than zero; for $C \geq 2$, the isotherm has a sigmoidal shape with an inflection point, according to the type II isotherm of Brunauer et al. (1940).

3.3. Optimal storage conditions

The second derivative of the third-degree polynomial equation indicated that the optimal a_w to preserve turmeric flour is 0.39 (Fig. 2).

In general, biochemical and microbiological reactions in food systems can be inhibited and product spoilage can be prevented when a_w is less than 0.6. It is important to highlight that the a_w of 0.39, which corresponds to a X_{eq} of 0.07 kg of water/kg of dry matter, is in accordance with the range of X_m values reported by the GAB model. This suggests that controlling moisture and a_w is crucial for the stability and preservation of turmeric flour. By maintaining a_w at 0.39, it is possible to minimize degradative and microbiological reactions, thus ensuring the quality and safety of the product over time.

3.4. Thermodynamic properties

The isosteric heat of sorption of turmeric flour is an important indicator of the interaction between water and the food matrix. Isosteric heat was determined for X_{eq} content ranging from 0.07 to 0.63 g of water/g of dry matter (Fig. 3) and decreased with the increase of X_{eq} content, with a sharp drop between moisture contents of 0.10 and 0.20 g of water/g of dry matter. This variation occurred because, at a lower X_{eq} content, water molecules present in turmeric flour are more strongly bound to the food matrix, which is mainly composed of proteins (8.80 ± 0.24 g, 100 g^{-1}, w. b.), and carbohydrates (61.44 ± 1.80 g, 100 g^{-1}, w. b.), requiring a greater amount of energy to be removed.

On the other hand, at high levels of X_{eq} in turmeric flour, the scarcity of hydrophilic sites ensues, leading to a reduction in active site coverage and the formation of multilayers (Ojike et al., 2022). This phenomenon necessitates less energy for the removal of water. The same trend was reported by Delgado et al. (2016) for turmeric drying and by Mallek-Ayadi et al. (2020) for melon seeds. The net isosteric heat of sorption and the X_{eq} content of turmeric flour were empirically and exponentially related, and the model showed 98.9% accuracy.

Like isosteric heat, the differential enthalpy (ΔH) of sorption is responsible for measuring the binding force of water, being of paramount importance for the design of efficient equipment in dehydration processes, in addition to contributing to the qualitative understanding of the state of water on the surface of food (Tagnamas et al., 2021). Rosa et al. (2021) described, for the drying of papaya seeds, that ΔH signs are important for understanding the binding process of the water molecule, being negative when it responds to the binding force and positive when

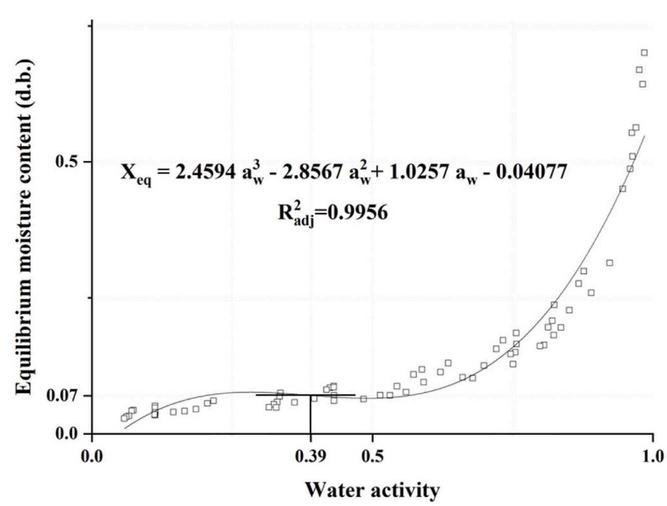

Fig. 2. – Determination of the optimal water activity for preserving turmeric flour.

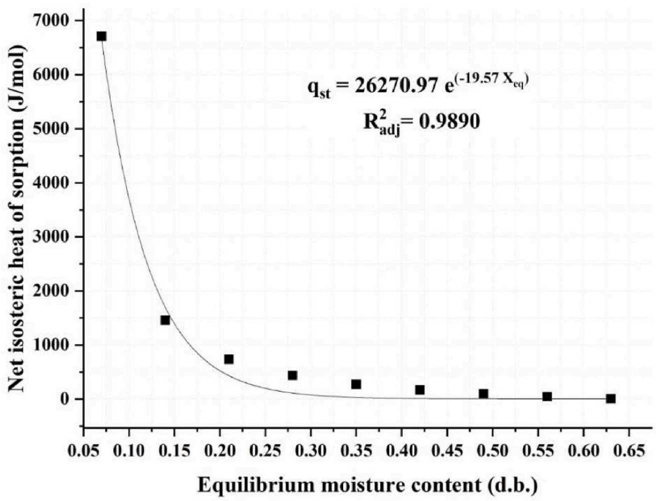

Fig. 3. – Net isosteric heat of sorption for turmeric flour as a function of equilibrium moisture content.

it responds to the repulsion force of these molecules. Turmeric flour showed an increase in ΔH as X_{eq} decreased (Fig. 4), demonstrating that highly accessible sites were occupied and greater energy was required for water removal (Borges-Machado et al., 2024a,b).

In the X_{eq} range studied (0.07–0.63g of water/g of dry matter), the ΔH values (in modulus) increased from −6.81 to −6712.89 J mol^{-1} as the equilibrium moisture content of the turmeric flour decreased. However, note also that there was a sharp increase in ΔH value (from −1458.94 to −6712.89 J mol^{-1}) in X_{eq} between 0.14 and 0.07 g of water/g of dry matter. These results suggest that, prior to this increase, there were residual water molecules within the food matrix and less energy was required to remove them. During and after the increase, it was observed that the increase in energy required for water removal may indicate that the molecules were more strongly adhered to the active hydrophilic polar groups on the surface. This behavior has also been observed for other vegetables, such as blueberry powder (Tao et al., 2018), apples (Mbarek & Mihoubi, 2019), and tiger nuts (Zhang et al., 2022).

The proportion of order or disorder of a system during water vapor sorption can be described by entropy due to its association with the spatial arrangements at the water-absorbent interface and, therefore, its relationship with the binding or repulsive forces of the entire system (Khiari et al., 2020; McMinn et al., 2005). In this investigation, entropy was computed as the slope of the line $\ln(a_w)$ versus 1/T derived from Equation (9), revealing a notable correlation with moisture content (Fig. 4). The highest ΔS value was recorded at 15.24 J mol^{-1} K^{-1} for the lowest X_{eq} value, while the lowest ΔS value stood at −0.17 J mol^{-1} K^{-1} for the highest X_{eq} value, indicating that a decrease in X_{eq} corresponded to an increase in the system's entropy. The lower differential entropy values are expected due to their relation to the number of sorption sites, with these values corresponding to the water forming the monolayer of the material matrix (Velázquez-Gutiérrez et al., 2015). This phenomenon echoes findings reported by Borges-Machado et al. (2024b) regarding ora-pro-nóbis (*Pereskia aculeata* Miller) leaves. It is important to highlight that the equilibrium moisture levels at which the maximum values of isosteric heat of sorption, enthalpy, and entropy for turmeric flour were obtained are very close to the monolayer moisture (X_m) values predicted by the GAB model. This result is consistent because X_m shows the amount of water strongly adsorbed at specific locations. Compared to other moisture contents, the energy required to break these bonds is the highest.

Physical and chemical phenomena that can be used to describe the molecular interaction of water and to understand the organization or order of the system (related to enthalpy) and the disorganization of these molecules (related to entropy) are described by the compensation theory and, In this way, it allows the evaluation of the reduction in the freedom of water molecules in foods (Velázquez-Gutiérrez et al., 2015). Two conditions must be met to ensure compensation: firstly, a linear correlation between enthalpy and entropy (Sanches et al., 2023); and secondly, the isokinetic temperature (TE) must be statistically distinct from the harmonic mean temperature (T_{hm}) (Krug et al., 1976).

The linear relationship ($R^2 = 0.9983$) of the compensation theory was confirmed in the present study (Fig. 5) between q_{st} and entropy. Confirmation was also possible from the statistical difference between T_B and T_{hm}, with values of 430.91 ± 23.75 K and 311.72 K, respectively. T_{hm} was lower than T_B, meaning that the process is driven by enthalpy, just as reported by Rosa et al. (2021) for papaya seeds. Water sorption processes controlled by enthalpy suggest that the microstructure of the material is stable and, therefore, does not change during the phenomenon (Gabas et al., 2000). Thus, water vapor adsorption mechanisms are determined by energetic interactions associated with the chemical composition of the product (Silva et al., 2014).

The Gibbs free energy value in the present study was 200.11 J mol^{-1}, demonstrating that the process is non-spontaneous (positive value of Gibbs free energy). One of the factors that contributed to the lack of spontaneity is the fact that horticultural products have more hydrophobic proteins that impair the water sorption process (Walstra, 2013).

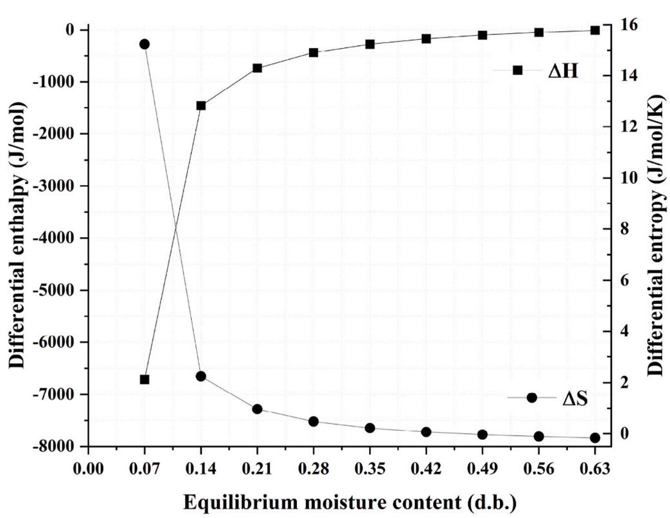

Fig. 4. – Change in enthalpy and entropy of turmeric flour as a function of equilibrium moisture content.

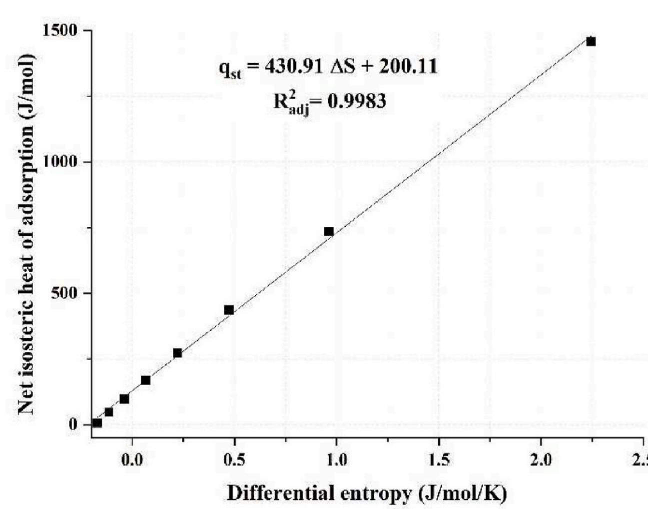

Fig. 5. – Linear relationship between net isosteric heat of sorption and entropy.

Similar results were reported by Rosa et al. (2021) for papaya seeds and by Rodrigues et al. (2020) for "Cumari-do-Pará" pepper seeds. However, it should not be generalized that the adsorption process for foods containing hydrophobic components in their composition will mean that the process is not spontaneous, as the temperature must be taken into account together with the structure of the adsorbent.

4. Conclusion

At all temperatures studied, the pattern of the sorption isotherms was type III. GAB was the mathematical model that best described the sorption isotherms at all temperatures analyzed ($R^2_{adj} \geq 0.998$; RMSE ≤ 0.0065; $X^2 \leq 4.22 \bullet 10^{-6}$). The moisture content of turmeric flour increased with the increase in relative humidity and the reduction in temperature. According to X_m, turmeric flour should be stored at temperatures of 5–40 °C and relative humidity of 38–54% to ensure microbiological safety. in addition, the optimal water activity is 0.39 in the same range of temperature. The net isosteric heat of sorption decreased with the increase in equilibrium moisture content, and entropy decreased at $X_{eq} > 0.07$. The enthalpy-entropy compensation theory was confirmed, sorption was driven by enthalpy ($T_B > T_{hm}$), suggesting that the water vapor adsorption mechanisms of turmeric flour are determined by energetic interactions associated with the chemical composition of the product. The process was considered non-spontaneous ($\Delta G > 0$), due to the presence of hydrophobic components in its composition. This research expands understanding of the water sorption characteristics of turmeric flour, which may contribute to a longer shelf life for food applications.

CRediT authorship contribution statement

Maria Siqueira-de-Lima: Writing – review & editing, Writing – original draft, Methodology, Investigation, Funding acquisition, Formal analysis, Conceptualization. **Caroline Cagnin:** Writing – review & editing, Writing – original draft. **André Luiz Borges-Machado:** Writing – review & editing, Writing – original draft, Methodology, Investigation, Funding acquisition, Formal analysis, Data curation, Conceptualization. **Marcio Augusto Ribeiro-Sanches:** Writing – review & editing, Writing – original draft, Methodology, Formal analysis, Data curation. **Javier Telis-Romero:** Writing – review & editing, Writing – original draft, Supervision, Resources, Project administration, Methodology, Funding acquisition, Conceptualization. **Osvaldo Resende:** Writing – review & editing, Writing – original draft, Supervision, Resources, Project administration, Methodology, Conceptualization.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Data availability

Data will be made available on request.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.lwt.2024.116592.

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