Thermal and thermodynamic characterization of a dye powder from liquid turmeric extracts by spray drying

Caracterización térmica y termodinámica de un colorante en polvo a partir de extractos líquidos de cúrcuma obtenidos por secado por aspersión

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ABSTRACT

Key words:

Sorption isotherms
Turmeric powder
Glass transition
temperature
TGA analysis

This study aimed to evaluate the thermodynamic properties of sorption isotherms and glass transition temperature (T_g) and the thermal properties of a dye powder obtained from turmeric extracts using spray drying. The sorption isotherms were evaluated at 15, 25 and 35 °C using the dynamic gravimetric method, wherein the isotherm data of the experiment were fit to GAB and BET models. Likewise, the T_g was measured using differential scanning calorimetry (DSC). Thermogravimetric analysis (TGA) was used to determine the mass loss, and the thermal properties (heat capacity, diffusivity and thermal conductivity) were determined using transient flow method. The results demonstrated that the GAB model best fit the adsorption data. The DSC analysis presented a glass transition temperature of 65.35 °C and a loss of volatiles at 178.07 °C. The TGA analysis indicated a considerable mass loss starting at 193 °C, resulting in degradation of the product. The thermal properties demonstrated a heat capacity of 2.45 J/g °C, a thermal conductivity of 0.164 ± 0.001 W/mK and a thermal diffusivity of 8.7×10^{-8} ± 0.000 m²/s.

RESUMEN

Palabras claves:

 Isotermas de sorción
Polvo de curcumina
Temperatura de
transición vítrea
Análisis TGA

El objetivo del trabajo fue evaluar las propiedades termodinámicas por isotermas de sorción, temperatura de transición vítrea (DSC), y propiedades térmicas del colorante en polvo obtenido a partir de extracto de cúrcuma usando secado por aspersión. Se evaluaron las isotermas de sorción a 15, 25 y 35 °C mediante el método gravimétrico dinámico donde los datos experimentales de las isotermas se ajustaron a un modelo de GAB y BET. Así mismo, la temperatura de transición vítrea mediante calorimetría diferencial de barrido (DSC), análisis termo gravimétrico (TGA) para determinar pérdida de masa y propiedades térmicas (calor especifico, difusividad y conductividad térmica) fueron evaluadas por el método transitorio. Los resultados mostraron que el modelo que mejor se ajustó a los datos experimentales de las isotermas de adsorción fue el modelo de GAB. El análisis DSC mostró una temperatura de transición vítrea de 65,35 °C y una pérdida de volátiles a 178,07 °C. El análisis TGA indicó una pérdida de masa considerable a partir de 193 °C, mostrando degradación del producto. Las propiedades térmicas del producto en polvo mostraron un valor para el calor específico de $2,45 \times 10^{-3}$ J/kg °C, conductividad térmica de 0,164 ± 0,001 W/mK y difusividad térmica de $8,7 \times 10^{-8}$ ± 0,000 m²/s.

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Turmeric is a natural mixture of compounds, including polyphenols, with an orange-yellow color found in *Curcuma longa* rhizomes. It is used as a dye in the food and textile industries. It is a potential protective agent against illnesses such as cancer, HIV infection, neurological disorders and cardiovascular and skin diseases; furthermore, it possesses antioxidant, anti-carcinogenic and anti-inflammatory properties (Li *et al.*, 2013). Some applications of turmeric have been limited by its sensitivity to alkaline conditions, thermal treatments, light, metal ions, enzymes, oxygen and ascorbic acid, demonstrating its low bioactivity. Turmeric is yellow in an acidic medium (pH 2.5-7) and red in a basic medium (pH > 7) (Paramera *et al.*, 2011).

Food systems can present physical, chemical and biological changes, which can cause phase transitions due to resulting from temperature and pressure effects. Water is a relevant factor because it affects the physical properties of products; therefore, a relationship can be established between food preservation and the food's water content through water activity, which is equal to the relative humidity in equilibrium with the product at the same temperature (Figueira *et al.*, 2004). The sorption isotherms relate the equilibrium water content and the equilibrium relative humidity at a fixed temperature. In accordance with the fixed relative humidity, the product can adsorb or lose moisture with the surroundings, thus the phenomena of adsorption or desorption, respectively, can be present (Chen and Chen, 2014).

In the case of powders obtained by spray drying, one important transition corresponds to first and second order phase changes, where the glass transition temperature is an important factor. A product obtained by spray drying can be considered a non-crystalline material; it can exist in the glassy state or in the super-cooled liquid state (LeBail *et al.*, 2003).

The glass transition temperature (T_g) corresponds to a change in the plasticity of the material over a range of temperatures where the material is more brittle and glassy below the T_g and more flexible above this temperature (LeBail *et al.*, 2003). The importance of the T_g can be seen in its relationship with the stability and shelf-life of a powdered product. Knowing the T_g is essential to ensure that the system is kept under that temperature

in suitable conditions, thereby avoiding negative effects on the physical stability of the product and decreasing deterioration of the product while increasing its shelf-life. It is necessary to know the moisture sorption behavior of the material during storage because the presence of water decreases the T_g to a temperature that can be close to or below the storage temperature (Hernandez *et al.*, 2011; Carter *et al.*, 2012).

Little reported information exists on the thermodynamic characterization of turmeric powder, although some studies have evaluated the glass transition temperature and have performed thermogravimetric analyses (TGA) on spray-dried products (Laczkowski and Sousdaleff, 2013). Knowing these properties, characteristics and behaviors is important because they provide information on the mechanics of adsorption and the interaction between the food components and the food moisture, which are of vital importance to the storage, stability and/or shelf-life of the product (Ayala, 2011). Based on these considerations, this study aimed to evaluate the thermal and thermodynamic properties of the dye powder from liquid turmeric extracts using spray-drying technology.

MATERIALS AND METHODS
Production of the drying suspension
The drying suspension was prepared using a concentrated extract of turmeric (TECNAS, S.A.), maltodextrin (DE 10) (TECNAS, S.A.) and water. The concentrated turmeric extract was obtained in a glycerin mixture under rotoevaporation at 50 °C and 90 rpm and then stored in the dark under refrigeration at 4 °C. The curcuminoid concentration was measured in the extract using high performance liquid chromatography (HPLC).

Spray drying process
The suspension was mixed with maltodextrin (DE 10) and water and homogenized (Ultra-Turrax IKA-T25) at 7400 rpm for 2 minutes. A VIBRASEC completely automatic spray dryer was used with an evaporative capacity of 1.5 L h^{-1}. Atomizer rotational speed, air drying temperature (inlet and outlet), air flow rate, feed flow into the dryer and feed temperature were all monitored and regulated. The drying conditions were previously optimized statistically to the following: inlet air temperature (149 °C), outlet air temperature (75 °C) and atomizer rotational speed of 28000 rpm.

Sorption isotherms

To define the sorption isotherms, the gravimetric method was used. Powder samples of 2 g were placed in triplicate in an Aqualab VSA1020 Decagon, with relative humidity varying from 10 to 90% at temperatures of 15, 25 and 35 °C. The experimental data were fitted to Brunauer, Emmett and Teller (BET) and Guggenhein, Anderson and de Boer models (GAB).The following equations were used to represent the two models (Figueira *et al.*, 2004; Furmaniak *et al.*, 2007; Sinija and Mishra, 2008; Ceballos *et al.*, 2009; Perdomo *et al.*, 2009; Ayala, 2011; Djendoubi *et al.*, 2012):

$$BET\ Model\quad X_e = \frac{X_m C a_w}{(1 - a_w)(1 + (C - 1)a_w)} \qquad (1)$$

$$GAB\ Model\quad X_e = \frac{X_m C K a_w}{(1 - K a_w)(1 + (C - 1)K a_w)} \qquad (2)$$

where X_e corresponds to the equilibrium water content, X_m is the monolayer water content, C corresponds to the constants of the BET and GAB models, K is a constant for the GAB model and a_w is the water activity. The best fit was defined by the highest value of R^2 and the lowest values of X^2 and E_{RMS} (Demir *et al.*, 2004; Goyal *et al.*, 2006; Ayala, 2011).

The sorption models were calculated using the following equations:

$$X^2 = \frac{\sum_{i=1}^{N}(M_{R,exp,i} - M_{R,pre,i})^2}{N - z} \qquad (3)$$

$$E_{RMS} = \left[\frac{1}{N} \sum_{i=1}^{N} = (M_{R,exp,i} - M_{R,pre,i})^2 \right]^{1/2} \qquad (4)$$

In these expressions, $M_{R,exp,i}$ and $M_{R,exp,i}$ are the calculated and experimental values for each sorption model, N corresponds to the number of experimental data and z is the number of constants for each model.

Equation (5) was used to find the thermodynamic properties (Clausius-Clapeyron equation) for the liquid vapor equilibrium at the experimental temperature and pressure (Yan *et al.*, 2008; Bell and Labuza, 2000). With

this equation, it was possible to calculate the change in enthalpy for the different moisture levels associated with the sorption processes. The water vapor partial pressure was determined by the following equations:

$$nPw = -\frac{\Delta H}{R}\frac{1}{T} + Cte \qquad (5)$$

$$P_w = P_w^0 a_w \qquad (6)$$

Where P_w corresponds to the water vapor partial pressure in equilibrium with the material at a determined temperature, P_w^0 corresponds to the water vapor pressure at a defined temperature for each product, and a_w corresponds to the water activity.

The Gibbs free energy (ΔG) and the differential entropy (ΔS) were also calculated:

$$\Delta H = \Delta H_V + Q_S \qquad (7)$$

$$\Delta G = RTln\left(\frac{P_w}{P_w^0}\right) = RT\ ln(a_w) \qquad (8)$$

$$\Delta S = \frac{\Delta H - \Delta G}{T} \qquad (9)$$

where ΔH corresponds to the differential adsorption heat (J/mol), ΔH_V is the latent heat of water (J/mol), Q_s corresponds to the net sorption heat (J/mol), R is the universal gas constant (8.314 J/mol K), and T is the absolute temperature (K).

Differential scanning calorimetry (DSC) analysis

The glass transition temperature (T_g) was determined using a differential scanning calorimeter from Q2000-TA Instruments calibrated with indium. DSC was performed in a nitrogen atmosphere (50 mL/min flow speed) with a temperature range of 20 to 300 °C at a rate of 10 °C/min. Approximately 8 mg of each sample were hermetically sealed in an aluminum capsule, and the reference was an empty aluminum capsule. A thermogram was obtained for each sample, and the T_g was taken as the starting point of the change in the heat flow curve (Cheng *et al.*, 2013; Enrione *et al.*, 2010; Li *et al.*, 2013; Sablani *et al.*, 2007).

Thermogravimetric analysis (TGA)
The possible mass loss was quantified using a Linseis STA PT1600 thermobalance. Approximately 10 mg of powder was placed in a ceramic capsule in a nitrogen atmosphere (50 mL/min), and the sample was heated from ambient temperature to 300 °C at a heating rate of 10 °C/min (Laczkowski and Sousdaleff, 2013).

Thermal properties
The thermal conductivity (k) and the thermal diffusivity (α) were obtained using a KD2-Pro (Decagon Devices, Inc., USA) equipped with an SH-1 sensor. The heat capacity value was calculated using the classical definition (Onita and Ivan, 2005; Bergeron *et al*., 2012; Perusello *et al*., 2013), where the density (ρ) of the powdered product was quantified by pycnometry:

$$Cp = \frac{k}{\rho\alpha} \qquad (10)$$

RESULTS AND DISCUSSION
Sorption isotherms
Figure 1 shows the experimental sorption isotherms fitted to the BET and GAB models. The experimental data are shown as equilibrium water content (X_e) as a function of water activity (a_w), which is a characteristic relationship in systems that contain water-soluble compounds such as sugars (maltodextrin). This type of behavior has also been observed by Ceballos (2009), who studied sorption isotherms in dehydrated soursop powder, by Cano *et al*. (2013) in mango powders obtained by spray drying and by Gabas *et al*. (2007) in pineapple pulp powder.
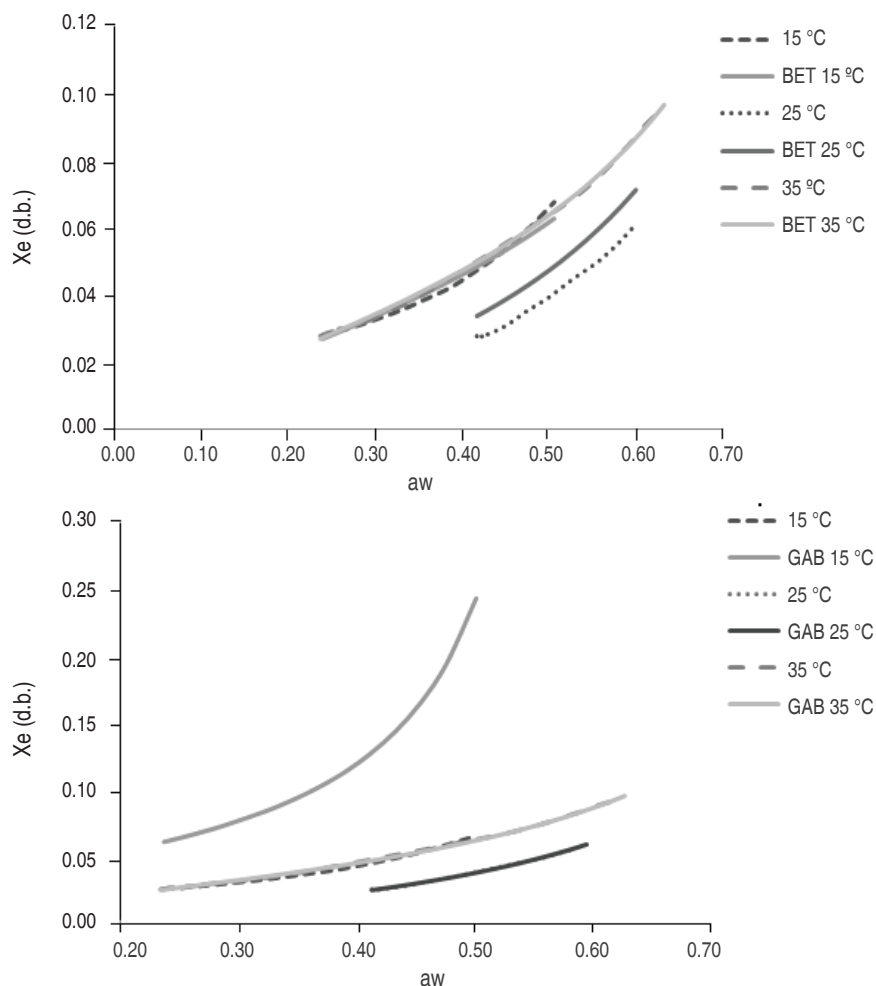

Figure 1. Sorption isotherms for the turmeric powder in the BET (a) and GAB (b) models.

In Figure 1, X_e of the product increased with a_w, a behavior that, according to Ayala (2011), is common in many food types. The results demonstrated that for a specific a_w value, the increase in temperature contributed to the decrease of X_e of the product, which was related to the hygroscopicity of the powder, which decreased at higher temperatures (Sinija and Mishra, 2008; Ayala, 2011; Cano et al., 2013).

Vega et al. (2006), Goula et al. (2008), Ayala (2011) and Martínez et al. (2014) suggested that physical and chemical changes caused by increasing temperatures could decrease the active sites in the product compounds where water interacts with the solid surface of the food. Furthermore, it is important to emphasize that the high excitation state of water molecules at high temperatures could result in decreased attraction between water and the food, which would explain why the moisture content in the powder is lower at higher temperatures (Figure 1).

Table 1 contains the BET and GAB model parameters. The GAB model fit the experiment data better than the BET model with higher R^2 values and lower X^2 and E_{RMS} values.

Table 1. Constants and results for the BET and GAB models at different temperatures.

Model	Constants	Turmeric powder		
		15 °C	25 °C	35 °C
BET	X_m (g/100 g powder)	0.040	0.048	0.042
	C	3.30	0.95	3.05
	R^2	0.91	0.74	0.99
	X^2	0.00	6.23×10^{-6}	0.00
	$E_{(RMS)}$	0.0016	0.0070	0.0005
GAB	X_m	0.0425	0.0403	0.037
	C	14.25	0.81	3.63
	K	1.64	1.04	1.05
	R^2	0.99	0.99	0.98
	X^2	0.002	0.000	0.000
	$E_{(RMS)}$	0.0400	0.0004	0.0005

The GAB model provided information on the monolayer water content (X_m), showing that water was strongly absorbed at the active sites on the surface of the food, which relates to suitable storage conditions for low-moisture products (Cano et al., 2013). For the turmeric powder, the X_m value varied between 3.70 and 4.25 g/100 g powder, similar to the findings of Cano et al., (2013) for spray-dried mango powder and Al-Muhtaseb et al. (2004a) for starch powder. The value of the Guggenheim constant (C) indicated the relationship between the sorption heat of the monolayer molecules and temperature. (Here, this value varied indiscriminately with temperature.) The value of the constant K related the sorption heat in the multilayer molecules to the temperature increase from 15 to 25 ºC, which was very similar between 25 and 35 °C; this behavior was also reported by Sharma et al. (2009). Figure 2 shows the sorption thermodynamics of the dried product, where Figure 2(a) contains the net sorption heat value (Q_s) that corresponded to the sorption isosteric heat, which described the interaction between the water and product components. There is an increase from 72,480 to 105,630 J/mol for water content between 1 and 2 g/100 g turmeric powder dry basis (d.b.). A decrease from 94,930 to 69,520 J/mol for water content between 3 and 7 g/100 g turmeric powder (d.b.) was observed. The maximum value was achieved with a moisture value close to 2 g/100 g turmeric powder (d.b.). The increase of the sorption isosteric heat in low water contents could be explained by an elevated solid-vapor interaction in the active sites of the product surface, while the sorption heat decreased with an increase in the moisture; the water occupied fewer active sites, decreasing the interaction of the existing kinetic energy between the water molecules and the powder product. A similar behavior was observed by Ayala (2011) and Martínez et al. (2014).

Figure 2(b) shows the Gibbs free energy (ΔG) of the turmeric powder. The change in the ΔG indicated that water adsorption onto the powder product is spontaneous because the obtained values were negative in the evaluated water activity range, demonstrating that the reactions that occurred in the microstructure of the product were stable in the range of temperatures evaluated. Furthermore, these values indicate that the powder product possesses a high hygroscopicity because the ΔG value was more negative

for lower water contents. For moisture values over 7 g/100 g turmeric powder, the ΔG value was approximately zero, indicating that this process had a tendency to reach equilibrium. A similar behavior was observed by Cano *et al.* (2013) and Martínez *et al.* (2014).

Figure 2(c) shows the differential entropy (ΔS), demonstrating a strong dependency on the moisture content. Changes in entropy are related to the number
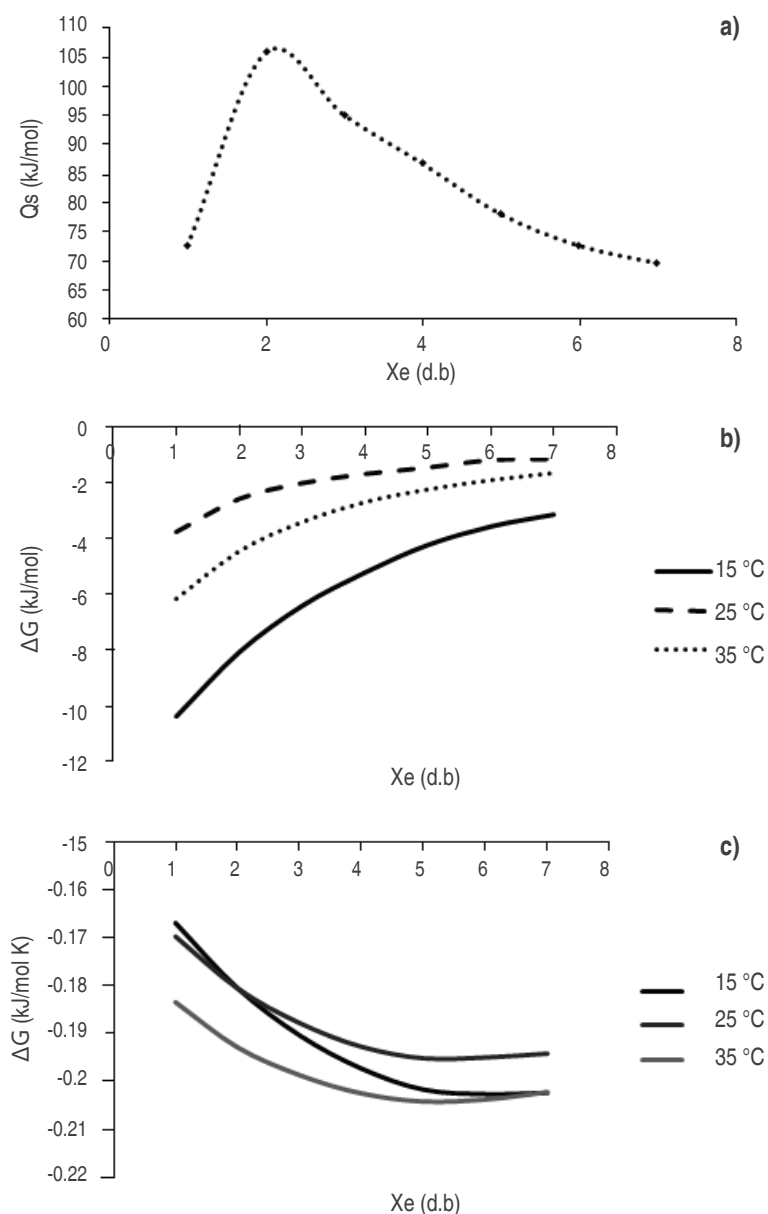

Figure 2. Thermodynamic sorption study: (a) sorption heat (b) Gibbs free energy and (c) change in entropy.

of available adsorption sites at a specific energy level proportional to the change in entropy, which is related to the degree of disorder and random movement of water molecules; lower entropy is an indicator of better stability (Goneli *et al.*, 2013). This type of behavior was observed by Goula *et al.* (2008) and Al-Muhtaseb *et al.* (2004b). The negative ΔS values indicated the water adsorption process by the compound molecules of turmeric powder. The highest change in entropy was obtained at a temperature of 35 °C, where the kinetic energy of the molecules is higher. Furthermore, for temperatures between 15 and 25 °C, smaller changes were observed due to the lower kinetic energy resulting from the difference between the initial and final entropy. A similar behavior was observed by Jamali *et al.* (2006).

Differential scanning calorimetric analysis

Figure 3 shows the thermogram obtained from differential scanning calorimetry (DSC). An endothermic peak was observed at 178.07 °C, which corresponded to a desolvation
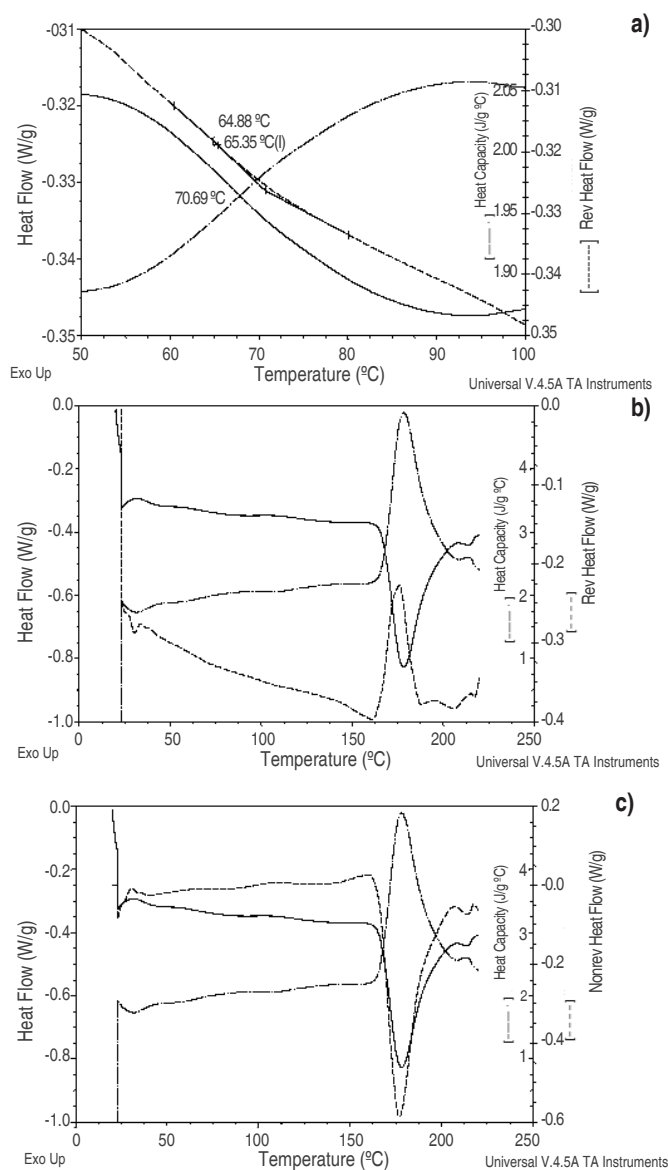

Figure 3. Thermogram of the turmeric powder obtained by DSC. (a) Glass transition temperature. (b) Total heat flow, heat capacity and reversible heat flow. (c) Total heat flow, heat capacity and non-reversible heat flow.

or loss of volatiles, which is probably why the total heat flow peak and the reversible heat flow peak did not coincide and were opposites. Likewise, fusion points were not observed, indicating the role played by maltodextrin as a protective agent, similar to the observations of Laczkowski and Sousdaleff (2013), who obtained an endothermic peak at these temperatures for turmeric encapsulated with maltodextrin. Parize *et al.* (2012) demonstrated endothermic peaks at similar temperatures for chitosan with turmeric obtained by spray drying. Paramera *et al.* (2011) found slightly higher values in endothermic peaks for turmeric encapsulated in yeast cells because, for turmeric molecules integrated within yeast cells, the transitional phases are increased. Pawar *et al.* (2012) found values of 182 °C for amorphous turmeric; however, this was related to a melting event because only turmeric was used.

According to the results of DSC, the glass transition temperature (T_g) was 65.35 °C. Pawar *et al.* (2012) observed a T_g value of 69.4 °C for amorphous turmeric, and Parize *et al.* (2012) found that the presence of chitosan increased the T_g of turmeric to 91.30 °C, demonstrating that the turmeric colorant is encapsulated between the polymeric chains of this compound. Wang *et al.* (2010) observed T_g values of 105.95-110.10 °C in turmeric contained in hyperbranched polymers, which

increased the T_g. The T_g value of 65.35 °C shows that there was a phase change in the product above this temperature, so the storage temperature must be lower than this value.

Thermogravimetric analysis (TGA). Figure 4 shows the curve for mass loss as a function of temperature. The initial temperature of the mass loss was approximately 193 °C, resulting in degradation or decomposition of the turmeric powder; below this temperature, the decrease in the thermogram corresponded to moisture loss. Laczkowski and Sousdaleff (2013) demonstrated that the mass loss in turmeric encapsulated with maltodextrin at 228 °C was due to the interaction of the colorant with the maltodextrin protecting the compound because loose turmeric had a mass loss value starting at 201 °C. Furthermore, the drying process was performed by lyophilization; therefore, encapsulated turmeric is more stable, although it is necessary to emphasize that the profile demonstrated by the authors is very similar to those of the present study. Parize *et al.* (2012) reported a degradation temperature of 321 °C for turmeric as a result of the presence of chitosan and the interactions between this polymer and turmeric that contribute to the thermal stability; although in that study, the degradation temperature was higher, the mass loss profile is very similar.
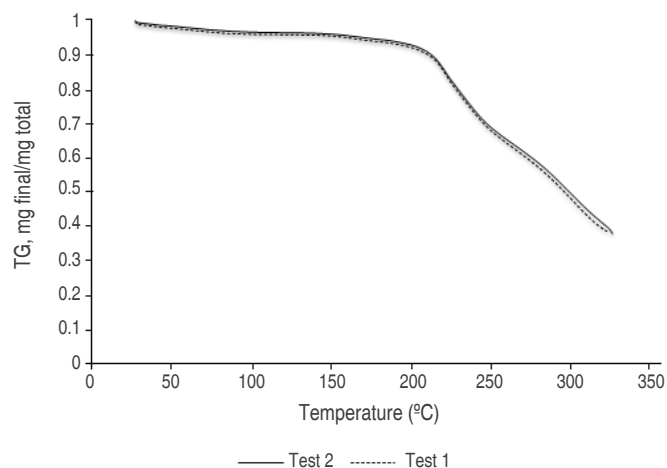

Figure 4. Thermogravimetric analysis (TGA) of turmeric powder.

Thermal properties

The thermal conductivity was 0.16 ± 0.00 W/m °C, a low value related to the moisture content of the product (Perusello *et al.*, 2013). The thermal diffusivity value of

the product was 8.7×10^{-8} ± 0.00 m^2/s; similar values were obtained by Perusello *et al.* (2013) and Tiwari *et al.* (2011). The heat capacity value (C_p) corresponded to 2.45 J/g °C, whereas the value obtained in the DSC analysis was 1.95

J/g °C. This unexpectedly low value is probably because during the measurements with the KD2-Pro device, factors such as ambient temperature and the location of the sample sensor affected the results. The C_p value is a thermodynamic measurement of the temperature that both induces and is related to structural changes, and it is an important parameter for the characterization of the amorphous state (Pawar *et al.* 2012). In a study conducted by these authors on amorphous turmeric, C_p values of 530 J/mol K (1.44 J/g ºC) were found; the difference with the present study could be because, in their study, the authors did not use coadjuvant agents such as maltodextrin, whose heat capacity is 1.50 J/g ºC, because there was an increase in the C_p value of the powder turmeric (Frias *et al.*, 2001).

CONCLUSIONS

The GAB model for the turmeric powder obtained by spray drying describes the sorption isotherms and the adsorption process well, showing the better fit modeling the experimental data. The final product is stable under storage conditions at temperatures below 65.35 °C, in the range of temperatures where the turmeric powder is in the amorphous state. Additionally, it is thermally stable, although for higher temperatures (above 200 °C), the degradation and decomposition of curcumin is high due to loss of volatiles. The values obtained for thermal properties (heat capacity, conductivity and thermal diffusivity) are low, related to the low moisture content present in the turmeric powder and as well as the properties of a mixture of maltodextrin and curcumin.

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