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Moisture sorption behaviour and thermodynamic properties of adsorbed water of Jerusalem artichoke (*Helianthus tuberosus* L.) powder

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Abstract

Moisture sorption isotherms of Jerusalem artichoke (*Helianthus tuberosus* L.) powders were determined at 20, 30, and 40°C by the static gravimetric method over water activity (a_w) in the range of 0.11 and 0.85. The sorption isotherms exhibited type III behaviour according to Brunner-Emmett-Teller (BET) classification. Seven sorption isotherm models were fitted to the experimental data, and the Guggenheim-Anderson-de Boer (GAB) model was selected to evaluate the thermodynamic properties to determine the characteristics of adsorbed water and energy requirements associated with the sorption process. The monolayer moisture contents of the sorption process were 8.08, 7.09, and 5.06 g/100 g dried solids at 20, 30 and 40°C, respectively. Both the net isosteric heat of sorption and differential entropy increased with the increase of moisture content, below the monolayer moisture content above which both registered a sharp decline. The maximum isosteric heat of sorption and differential entropy for these powders were 17.91 kJ/mol and 47.15 J/(mol K), respectively. The application of enthalpy-entropy compensation theory showed that the moisture sorption mechanism was controlled by enthalpy, and it was a non-spontaneous process. The values of specific surface areas available for sorption at 20, 30, and 40°C were 286.50, 251.43 and 179.48 m^2/g, respectively. These results can be used to design efficient dehydration and storage conditions for Jerusalem artichoke powders, and to predict their storage stability.

Introduction

Jerusalem artichoke (JA; *Helianthus tuberosus* L.) is a species of sunflower. It is considered to be originated from central North America, and now widely cultivated in China and other Asian countries due to its fast breeding and strong adaptability (Khuenpet *et al.,* 2017). The tubers of Jerusalem artichoke provide vitamins and minerals, such as calcium, iron, selenium, and potassium. Tubers of JA are also rich in inulin, a prebiotic compound with similar properties of dietary fibre (Afoakwah *et al.,* 2015). Thus, JA is drawing increasing interest as it can be used as functional component in many food formulations. For example, Guo *et al.* (2018) reported that addition of 4% of oven-dried JA powders could be utilised as a nutritious prebiotic ingredient and fat-replacer in low-fat yoghurt. Afoakwah *et al.* (2015) found that the application of JA powders enhanced the anti-microbial properties and oxidative stability, and increased water-holding capacity of emulsion-type sausage. However, since JA powders contain an amount of carbohydrate (e.g. fructo-oligosaccharides, reducing sugar, soluble dietary fibre) and protein, some undesirable physical changes related with water adsorption, such as agglomeration, caking, or loss of solubility may take place if they are stored under suboptimal conditions. Hence, knowledge for water sorption characteristics is very important to ensure the stability and acceptability of JA powders during storage and marketing.

The measurement and modelling of moisture sorption behaviour of JA powders are of great practical significance to predict their storage stability and also selecting suitable packaging material (Carvalho Lago and Noreña, 2015). The moisture sorption isotherm is a helpful method to illustrate the relationship between equilibrium moisture content (EMC) of agricultural products and environmental relative humidity (ERH) at a chosen temperature. Many empirical or semi-empirical models have been provided for describing moisture sorption properties of agricultural products. For example, Brunner-Emmett-Teller (BET) model is the most commonly used two-parameter model; however, its applicability could be limited to water activity (a_w)

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in the range 0.05 - 0.45 (Yogendrarajah *et al.,* 2015). The Guggenheim-Anderson-de Boer (GAB), which is a three-parameter model, is also extensively used to represent agricultural material's water sorption behaviour because of its versatility. Also, the GAB parameters have special physical meanings, for example, K and C are constants related to the adsorption heat of the multilayer and monolayer, respectively (Bhandari and Adhikari, 2008; Yogendrarajah *et al.,* 2015). X_M represents the amount of water needed to surround the matrix surface with just one layer of water molecules. X_M is a critical parameter because it denotes the water content at which the spoilage reaction rate is the lowest as a result of the strong binding of water by the hydrophilic sites on the agricultural products (Moreira *et al.,* 2008). In addition, other empirical models, i.e. Smith model and Halsey model, have also been applied to fit the experimental moisture sorption data for different agricultural products. However, none of these empirical models are as versatile as the GAB and BET models.

The other application of moisture sorption isotherm is to evaluate some typical thermodynamic properties of agricultural products, which include differential heat of sorption, spreading pressure, enthalpy-entropy compensation, and so on. These data can provide greater insights into the water characteristics and energy requirements about the sorption behaviour (Carvalho Lago and Noreña, 2015; Cervenka *et al.,* 2015). The net isosteric heat of sorption is considered as the difference of the total sorption heat and vaporisation heat at the experimental temperature. This parameter indicates the binding strength of water vapor to sorption sites of agricultural products. The differential entropy, on the other hand, is proportional to the number of available sorption sites at a specific energy level (Moreira *et al.,* 2016). The enthalpy-entropy compensation theory is a valuable tool to provide the information of the mechanisms which control the water sorption in agricultural products (Silva *et al.,* 2014). According to Krug *et al.* (1976), the compensation theory could be applied for evaluating moisture sorption process only if the isokinetic temperature (T_β) is different from the harmonic mean temperature (T_{hm}). In addition, the information of product's structural features including special surface area and pore radius can also be obtained by analysing the water sorption data. These data and parameters are very reliable and useful for predicting the shelf life of agricultural products and the choice of packaging materials. These are the reasons why the thermodynamic analysis was extensively carried out in the last decade to study the moisture sorption mechanism of various agricultural products.

Although the moisture sorption behaviours of various agricultural products have been previously studied, the adsorption kinetics and thermodynamic properties of JA powders are not available in the literature. Hence, the aims of the present work were to obtain the moisture adsorption isotherms of JA powders at three temperatures (20, 30, and 40°C), to explore the applicability of several sorption models to represent the experimental sorption isotherm data, and finally, to analyse the thermodynamic mechanism of adsorption behaviour.

Materials and methods

Sample preparation

Fresh Jerusalem artichoke tubers were purchased from a supermarket in Wuhu, China. To ensure the uniformity of the physicochemical properties, the JA tubers with uniform size, shape and stage of ripening were selected and then stored at 4°C until used. These tubers were washed with tap water, peeled and cut into cylinders with thickness of 4 mm and diameter of 3 cm, and then dried in a hot air dryer (LDO-9123A, Shanghai Longyue Instrument and Equipment Co. Ltd, Shanghai, China) at 70 ± 1°C until no significant changes in their mass were observed. Finally, the dried samples were ground using laboratory scale grinder (HS-500A, Hengshun Laboratory Equipment Manufacturing Co. Ltd, Nanchang, China) and further dried in a desiccator containing phosphorus pentoxide (P_2O_5) for 1 - 4 w to obtain dried samples.

Physicochemical property analyses

The moisture, ash, and protein contents of the dried JA powders were analysed according to Ruiz-Cano *et al.* (2014). Reducing sugar content of JA powders was determined by the 3, 5-dinitrosalicylic acid (DNS) method (Guo *et al.,* 2018). The total carbohydrate analysis was performed using phenol-sulphuric acid method (Albalasmeh *et al.,* 2013).

The crystallinity of JA powders was determined using X-ray diffraction (MinFlex 600, Rigaku Co., Tokyo, Japan) with a Ni-filtered CuKα radiation ($\lambda = 1.54$ Å), generated by an anode device operating at 40 kV and 30 mA. The diffraction patterns were recorded with a fixed time of 0.4 s per 0.02° step in the $4° < 2\theta < 30°$ range. The particle size distribution of the JA powders was measured by a laser diffraction analyser (Mastersizer 3000, Malvern Instruments co., Ltd, Worcestershire, U.K.) with a dry powder dispersion attachment. The particle size distribution was recorded in terms of volume mean particle size of 10% (D_{10}), 50% (D_{50}), and 90% (D_{90}) using the Mastersizer 3000 software.

Moisture sorption isotherms

The sorption isotherms of JA powders were determined using static gravimetric method (Bhandari and Adhikari, 2008). Seven saturated salt solutions (LiCl, KNO_3, $MgCl_2{\cdot}6H_2O$, K_2CO_3, NaBr, NaCl, and KCl) were prepared to produce relative humidity values varying from 11 to 85%. Approximately 1.0 g of samples were weighed into sample cups and then stored in sealed glass desiccators containing saturated salt solution. The desiccators were placed in an incubator (SPX-250BZ, Boxun Co., Shanghai, China) maintained at the set temperatures (20, 30, and 40°C). Besides, a 5 mL beaker containing 0.2 g thymol was also put into the desiccators of higher a_w ($a_w > 0.6$) to inhibit microbial growth in JA powders during equilibrium process (Suhr and Nielsen, 2003). Samples were periodically weighed until their mass reached constant. After reaching equilibrium, the water content of samples was gravimetrically determined by method 934.06 of AOAC (1984). The experiments were carried out in triplicate in each tested temperature.

Modelling of moisture sorption data

In the present work, seven equations shown in Table 1 were fitted to the experimental sorption data of JA powders. The GAB equation is considered to be the most useful ones to predict the critical water content for good storage stability because of its versatility. Besides, other semi-empirical or empirical models, i.e. Caurie, Oswin, Halsey, Smith, Henderson, and Peleg have also been widely applied to describe water sorption characteristics of agro-food products. The parameters of these models were calculated by non-linear regression analysis using Origin-Pro 8.5 software. The goodness of fit of the models was assessed using root mean square error (RMSE) and adjusted determination coefficient (R^2).

$$RMSE = \left[\frac{1}{N}\sum_{i=1}^{N}\left(X_{\exp,i} - X_{pre,i}\right)^2\right]^{1/2} \quad \text{(Eq. 1)}$$

$$R^2 = 1 - \frac{\sum_{i=1}^{N}(X_{exp,i} - X_{pre,i})^2}{\sum_{i=1}^{N}(\overline{X_{\exp,i}} - X_{exp,i})^2} \quad \text{(Eq. 1)}$$

where, and = experimental and predicted values, respectively, and N and n = number of observation and constants in each model, respectively. The model with lowest RMSE and highest R^2 was selected. The parameters C, K, and X_M in GAB equation are functions of temperature. The effect of temperature on GAB parameters C, K, and X_M can be calculated by Arrhenius-type Eqs. (3), (4), and (5), respectively (Moreira *et al.,* 2010; Hidar *et al.,* 2018).

$$K = K_o\, exp\left(\frac{H_L - H_N}{RT}\right) \quad \text{(Eq. 3)}$$

$$C = C_o\, exp\left(\frac{H_M - H_N}{RT}\right) \quad \text{(Eq. 4)}$$

$$X_M = X_{M_o}\, exp\left(\frac{\Delta H}{RT}\right) \quad \text{(Eq. 5)}$$

where, ΔH = Arrhenius energy factor (kJ/mol). H_M and H_N = heat of sorption for the monolayer and multilayer water, respectively, and H_L = condensation heat of water vapor (kJ/mol). The values of H_M, H_N, ΔH, X_{M0}, C_0, and K_0 were determined by fitting Eq. (3) - (5) to the corresponding GAB parameters (X_M, C, and K).

Thermodynamic properties of adsorbed water

Net isosteric heat and differential entropy of sorption

The values of net isosteric heat (*qst*) or differential enthalpy (ΔH_d), at constant water content (X), can be determined using the Clausius-Clapeyron equation (Eq. 6) (Tsami, 1991).

$$q_{st} = \Delta H_d = -R\left(\frac{d\,ln\,a_w}{d(1/T)}\right)_X \quad \text{(Eq. 6)}$$

The free energy changes in moisture sorption process are generally accompanied by variations of both enthalpy and entropy. Hence, the Gibbs-Helmholtz equation could be used to calculate the differential entropy of sorption.

$$(ln\,a_w)_X = -\frac{\Delta H_d}{RT} + \frac{\Delta S_d}{R} \quad \text{(Eq. 6)}$$

where, q_{st} = net isosteric heat of water adsorbed in JA powders (J/mol), ΔH_d and ΔS_d = differential enthalpy (J/mol) and differential entropy (J/(mol K)) of water adsorbed in JA powders, respectively, R = universal gas constant (8.314 J/(mol K)), a_w = the water activity, and T = temperature (K).

To calculate ΔH_d and ΔS_d, ln(a_w) was plotted against 1/T at a fixed water content. Values of ΔH_d and ΔS_d were calculated from the slope ($-\Delta H_d/R$) and intercept ($\Delta S_d/R$) of regression line, respectively.

Application of enthalpy-entropy compensation theory

Enthalpy-entropy compensation theory can be

Table 1. Parameters of fitted models for the moisture sorption isotherm of Jerusalem artichoke powder at 20, 30, and 40°C.

Model	Mathematical expression	Temperature (°C)	Goodness of fit parameters		Model coefficients			
			RMSE (× 10^{-2})	R^2	A	B	C	D
GAB	$X = \frac{X_M C K a_w}{(1 - K a_w)(1 - K a_w + C K a_w)}$	20	1.386	0.965	0.0808 ± 0.014, X_M	87.052 ± 4.935, C	0.819 ± 0.062, K	
		30	0.541	0.995	0.0709 ± 0.005, X_M	16.023 ± 5.777, C	0.873 ± 0.022, K	
		40	2.056	0.924	0.0506 ± 0.011, X_M	14.743 ± 1.898, C	0.971 ± 0.057, K	
Caurie	$X = exp(A + B a_w)$	20	1.452	0.961	-3.067 ± 0.132	1.987 ± 0.184		
		30	0.669	0.992	-3.227 ± 0.066	2.199 ± 0.093		
		40	2.445	0.892	-3.393 ± 0.273	2.356 ± 0.382		
Oswin	$X = A\left(\frac{a_w}{1 - a_w}\right)^B$	20	1.128	0.977	0.126 ± 0.005	0.415 ± 0.029		
		30	0.571	0.994	0.120 ± 0.003	0.463 ± 0.017		
		40	2.122	0.918	0.109 ± 0.009	0.513 ± 0.072		
Halsey	$X = \left(\frac{-A}{ln\, a_w}\right)^{1/B}$	20	1.324	0.968	0.014 ± 0.004	1.843 ± 0.147		
		30	0.639	0.993	0.0196 ± 0.002	1.643 ± 0.063		
		40	1.881	0.936	0.0251 ± 0.008	1.460 ± 0.172		
Smith	$X = A - B\,ln(1 - a_w)$	20	1.268	0.971	0.0461 ± 0.008	0.112 ± 0.008		
		30	0.520	0.995	0.0364 ± 0.003	0.120 ± 0.003		
		40	2.284	0.906	0.029 ± 0.014	0.118 ± 0.015		
Henderson	$X = \left(\frac{-ln(1 - a_w)}{A}\right)^{1/B}$	20	1.423	0.963	17.202 ± 4.484	1.589 ± 0.149		
		30	1.132	0.977	13.352 ± 2.497	1.428 ± 0.107		
		40	2.574	0.880	11.970 ± 5.128	1.319 ± 0.237		
Peleg	$X = k_1 a_w^{n_1} + k_2 a_w^{n_2}$	20	0.95	0.984	1287.71 ± 6.879, k_1	61.27 ± 3.395, n_1	0.227 ± 0.022, k_2	0.751 ± 0.102, n_2
		30	0.584	0.994	0.141 ± 0.028, k_1	0.473 ± 0.127, n_1	0.273 ± 0.035, k_2	4.360 ± 1.206, n_2
		40	0.627	0.993	0.177 ± 0.015, k_1	0.678 ± 0.085, n_1	36.95 ± 1.331, k_2	28.88 ± 1.918, n_2

X is the equilibrium moisture content (g water/g dry solids); a_w is water activity; X_M is the monolayer moisture content (g water/g dry solids); A, B, C, K, k_1, k_2, n_1, n_2 are parameters of the selected model. GAB is Guggenheim-Anderson-De Boer model.

used to assess physicochemical phenomena which prevail in the water sorption processes. This theory puts forward a linear relationship between q_{st} and ΔS_d as given by Eq. (8).

$$q_{st} = T_\beta \Delta S_d + \Delta G_\beta \quad \text{(Eq. 6)}$$

where, T_β = isokinetic temperature (K), and ΔG_β = Gibbs free energy (J/mol) at T_β. Values of T_β and ΔG_β were calculated from a linear regression analysis. To validate the compensation theory, Krug *et al.* (1976) put forward a test method to compare between isokinetic temperature (T_β) and harmonic mean temperature (T_{hm}), as defined by Eq. (9).

$$T_{hm} = \frac{n}{\sum_{i=1}^{n}(\frac{1}{T_i})} \quad \text{(Eq. 6)}$$

where, n = number of isotherms involved. The compensation theory is valid if . If then the sorption process is considered to be enthalpy driven, while if the opposite condition is observed, then the process is entropy-driven (Hassini *et al.,* 2015).

Specific surface area of sorption

The sorbed surface area (S_o) of JA powders was calculated using Eq. (10) (Yogendrarajah *et al.,* 2015).

$$S_0 = X_M \frac{1}{M_W} N_o A_{H_2O} \quad \text{(Eq. 6)}$$

where, X_M = monolayer water content (g/g dried solids), M_W = molecular weight of water molecule (kg/mol), N_0 = Avogadro number (6×10^{23} molecules/mol), AH2O = area of a water molecule (1.06×10^{-19} m^2/molecule).

Statistical analysis

The experimental results obtained were expressed as means ± SD. A nonlinear regression analysis by the least square method was performed using the Origin-Pro 8.5 software to estimate the model parameters over the a_w range of 0 - 0.85. The parameters were analysed through the student's *t* test, and the level of significance was set at 0.05 in all cases. The root mean square error (RMSE) and adjusted determination coefficient (R^2) were applied for selecting the best model to describe the moisture adsorption data of JA powder.

Results and discussion

Chemical composition and structural properties of JA powders

Table 2. Chemical composition of Jerusalem artichoke powders produced by hot air drying.

Chemical composition	Values (on dry basis)
Moisture (g/100 g)	4.47 ± 0.32
Ash (g/100 g)	3.23 ± 0.12
Protein (g/100 g)	7.56 ± 0.31
Reducing sugar (g/100 g)	6.48 ± 0.47
Total carbohydrate (g/100 g)	83.29 ± 3.64

The chemical composition and structural properties of adsorbing materials determine their moisture sorption behaviour. The chemical composition of the JA powders is listed in Table 2. The total carbohydrate content (83.29 g/100 g dried solids) is close to the value (81.67 g/100 g) reported by Afoakwah *et al.* (2015) in the case of oven-dried JA powders. Similarly, the protein content in the present work (7.56 g/100 g dried solids) is within the range of the values previously reported in the literature for dried JA tubers (6.4 - 10.4 g/100 g dried solids). The X-ray diffractogram of JA powder (Figure 1a) in the $4^\circ < 2\theta < 30^\circ$ range showed a characteristic of broad peak implying that it was amorphous. The result of XRD indicated that crystalline fraction was negligible in these JA powders. Particle size distribution of JA powders (Figure 1b) showed bimodal particle size distributions (PSD) with volume mean particle sizes at 10% (D_{10}), 50% (D_{50}) and 90% (D_{90}) of 25.4, 69.1, and 456.7 μm, respectively. Similar bimodal PSDs have been observed in earlier work on commercial water chestnut flour (Ahmed *et al.,* 2016).

Moisture sorption behaviours

The moisture sorption isotherms of JA powder at tested temperatures (20, 30, and 40°C) are illustrated in Figure 2. The equilibrium moisture content (EMC) slowly increased at a_w values below 0.65, and then increased quite rapidly at higher a_w values. Based on BET classification, these sorption isotherms are typical type III isotherms (Brunauer *et al.,* 1940). The "J" shape of the isotherm is a corresponding characteristic of agricultural products with high sugar content which adsorb small quantity of water at low a_w values, but their water adsorbing capacity increases sharply at higher relative humidity levels (Mrad *et al.,* 2012). These isotherms are similar to powders of blueberry (Tao *et al.*, 2018) and prickly pear seeds (Hassini *et al.,* 2015).

As can be observed from Figure 2, the sorption isotherms of JA powders were significantly affected by temperature. At lower a_w values (<0.65), the EMC values decreased when temperature increased, which

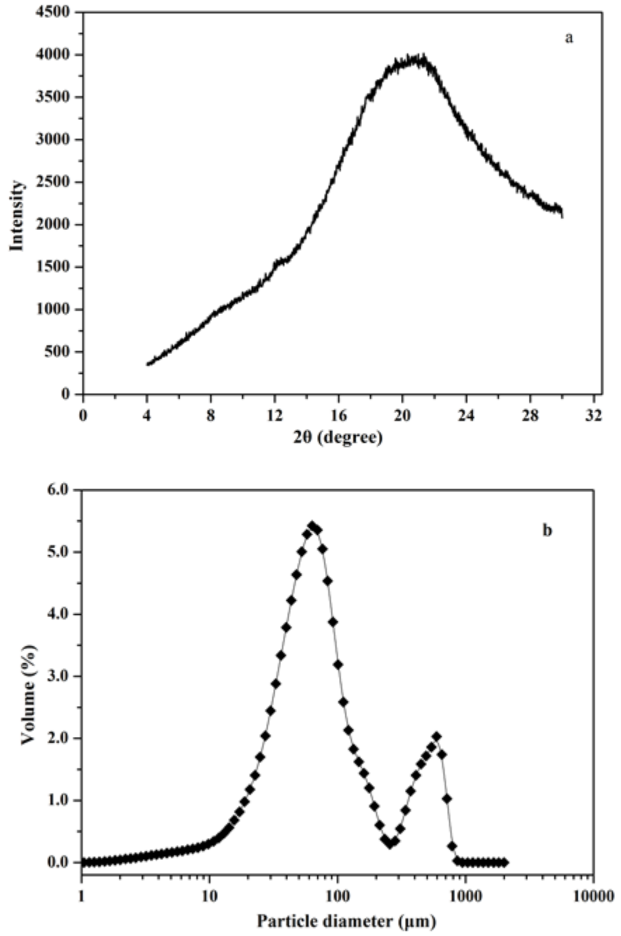

Figure 1. X-ray diffractogram (a), and particle size distribution (b) for Jerusalem artichoke powders produced by hot air drying.

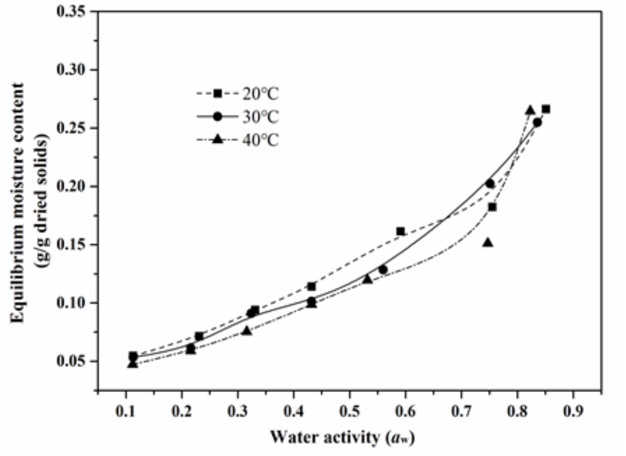

Figure 2. Moisture sorption isotherms for Jerusalem artichoke powder at 20, 30, and 40°C.

could be attributed to the reduction of active sites number, owing to physiochemical and structural changes produced by temperature increases (Ozturk and Takhar, 2018; Tao *et al.,* 2018). Moreover, the increase in energy level and temperature can cause a decrease in water stability and consequently separation of water molecules from binding sites (Noshad *et al.,* 2012). At a_w values higher than 0.65, there was a crossover point in moisture sorption isotherms at which the temperature effect was reversed, most probably due to the increasing solubility of sugars or the swelling behaviour of polymeric substances (e.g. polysaccharide and protein) at high relative humidity and temperatures (Moreira et al., 2016; Noshad et al., 2012). The swelling of polymeric substances can result in the development of free volumes within the structure which progressively increases the effective sorbing surfaces of the polymer. As shown in Table 2, JA powders produced in the present work contained 83.29 and 7.56% of total carbohydrates and protein contents (on dry basis), respectively. Similar tendency of crossover in sorption isotherms at high aw was also observed in other agricultural products such as brown seaweed (Moreira *et al.,* 2016; 2017) and quince (Noshad *et al.,* 2012). It is important to point out that the crossover point of sorption isotherms is highly dependent on the composition of products and the solubility of sugars. For example, the cross over a_w values for quince and brown seaweed (F. vesiculosus) were 0.75 and 0.50, respectively (Noshad *et al.,* 2012; Moreira *et al.,* 2017).

Mathematical modelling of water sorption

The parameters for each sorption model of JA powder are summarised in Table 1. It can be decided that the best model for fitting the experimental data was the Peleg model with the highest R^2 values (0.98 - 0.99), and the lowest RMSE at all tested temperatures. The GAB model also showed good capacity to fit with high R^2 values from 0.92 - 0.99, and remarkably low values of RMSE. It is commonly accepted that the GAB model is capable of fitting experimental isotherms data of many agricultural products (Chenlo *et al.,* 2011; Chranioti *et al.,* 2016; Torres and Seijo, 2016).

The validity of a moisture sorption model is not suitable to be established solely based on its ability to fit the experimental data; a theoretical basis also needs to be considered (Yogendrarajah *et al.,* 2015). When compared with other empirical models, the parameters of GAB model carry important physical meanings, and explains some important phenomena. For instance, the constant *K* is relevant to the sorption heat of multilayer. The values of parameter *K* in the present work ranged from 0.818 to 0.971, and close to 1.000, which indicated no distinction in chemical potential between water molecules adsorbed in multilayer and liquid water molecules (Carvalho Lago and Noreña, 2015; Edrisi Sormoli and Langrish, 2015). Parameter *C* denotes the difference in enthalpy between water molecules adsorbed in monolayer and multilayer (Cervenka *et al.,* 2015). The values of *C* in the present work were 87.052, 16.023, and 14.743 at 20, 30 and 40°C, respectively. These high values suggested that the monolayer water molecules were strongly bound to the active sorption sites of JA powders. Similarly, the parameter X_M represents the monolayer moisture content that covers or saturates the accessible hydrophilic sites of an agricultural product. Knowledge of X_M is essential as storage of agricultural products at relative humidity that equilibrates to X_M will result into longest shelf life because threats of deteriorative reaction are minimal under this condition (Moreira et al., 2008). As displayed in Table 1, the obtained X_M values of JA powders were 8.08, 7.09, and 5.06 g/100 g dried solids at 20, 30 and 40°C, respectively. The values of X_M decreased when the temperature increased, which is associated to the decrease in the active sites number of sorption as a result of physicochemical changes caused by the temperature (Torres and Seijo, 2016).

A detailed analysis of temperature dependence of GAB parameters was performed using Eq. (3) - (5). The $(H_M - H_N)$ value for JA powders was positive (101.65 kJ/mol), which indicated that there was great exothermic interaction of water vapour with primary sorption sites inside JA powders. Similar results were also claimed by Cervenka *et al.* (2015) in Yerba mate. On the other hand, negative value of $(H_L - H_N)$ (-6.62 kJ/mol) in JA powders implied that the heat of condensation of water is less than that of multilayer adsorption, which may be caused by the endothermic dissolution of tiny molecular solutes (Al-Muhtaseb *et al.,* 2004).

Thermodynamic properties of adsorbed water

Net isosteric heat of adsorbed water

The net isosteric heat of sorption (q_{st}) or differential enthalpy (ΔH_d) represents the amount of energy involved in the sorption process in addition to the vaporisation heat of pure water, which can be estimated by applying Eq. (7) (see Figure 3a). This parameter reflects the state of water adsorbed in the studied materials (Tao *et al.,* 2018). The variations in q_{st} as a function of EMC for JA powders are presented in Figure 3b. As shown, the values of qst sharply increased up to a small increase in EMC to attain a maximum value, and then continuously reduced with the rise in EMC until it attained a zero value. The maximum value of q_{st} (17.91 kJ/mol) was observed at EMC = 0.07 g/g dried solids, which approximately corresponded to the monolayer moisture content. The relatively high q_{st} values at initial stages of sorption are relative with monolayer formation, which represents the strength interaction between water molecules and highly active sorption sites located on the surface of JA powders or within these powers (Goneli *et al.,* 2013; Sahu *et al.,* 2018). The initial increase in q_{st} with the values of EMC may be relevant to the occupation of highly accessible sites in the JA powders. At low relative humidity, the high energy sorption sites in the matrix were gradually occupied by water molecules as water content increases, resulting into the increase of adsorption heat (Kaya and Kahyaoglu, 2007). On the other hand, the sharp decrease in q_{st} with the increase of adsorbed water content can be attributed to the reduction of active available sites, prompting the newly-arriving water vapour molecules to search other lower energy active sites, which ultimately leads to the formation of multilayer (Moreira *et al.,* 2016). Similar tendencies were reported for sorption behaviours of borojó fruit powder (Rodriguez-Bernal *et al.,* 2015) and chironji kernels (Sahu *et al.,* 2018).

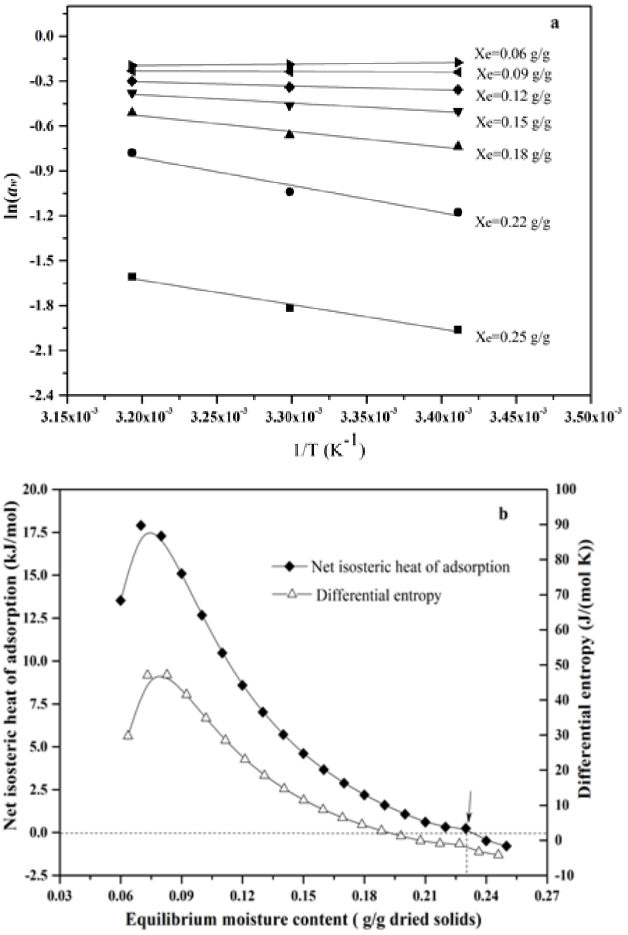

Figure 3. Curves of ln(a_w) *versus* (1/T) (a), and values of net isosteric heat and differential entropy versus water content (b) at temperatures ranging from 20 to 40°C.

The isosteric heat of sorption cannot be interpreted as a transition enthalpy; however, it can provide information about the energetic changes occurring in agricultural products at a specific hydration level (Viganó *et al.,* 2012). As shown in Figure 3b, there was an arrow in the differential enthalpy curve (the point corresponds to water content of 0.230 g/g dried solids), which indicated the transformation from a positive to a negative value in differential enthalpy. This phenomenon may be ascribed to the incipient solubilisation of soluble solids such as sugar. Similar behaviour has been reported by Rodriguez-Bernal et al. (2015), who found that the incipient solubilisation of soluble solids occurred at water content of 0.114 g/g dried solids in borojó pulp powder. The incipient solubilisation of soluble solids occurring at different water contents can be attributed to varying chemical composition of agricultural products which leads to different dissolution enthalpies. Flores-Andrade *et al.* (2018) pointed out that the dissolution of low molecular weight sugars is an endothermic process. Conversely, the dissolution of inorganic salt, such as calcium chloride, is an exothermic process in which the direction of heat as in its adsorption process.

Differential entropy of adsorbed water

The values of ΔS_d with water content for JA powder are illustrated in Figure 3b. It can be seen that ΔS_d values rapidly rose to the maximum value (47.15 J/(mol K)) in the initial stage of adsorption process; however, when the EMC values were > 0.08 g/g dried solids, it sharply declined with the increase of EMC. This is because the accessible active sites in the agricultural products at higher EMC are occupied by water molecules, resulting in the reduction of sorption capacity. The variation of ΔS_d with the increase of water content in JA powder differs from that in other agricultural products (Carvalho Lago and Noreña, 2015; Velazquez-Gutierrez *et al.,* 2015). Nevertheless, the trend observed in JA powder is in agreement with the ΔS_d observed inchironji kernels (Sahu *et al.,* 2018), and black peppercorns (Yogendrarajah *et al.,* 2015).

Enthalpy-entropy compensation

The value of harmonic mean temperature (T_{hm}) calculated by Eq. (9) was 302.9 K, which was significantly lower than the isokinetic temperature (T_β, 360.2 K), confirming the fact that this is the enthalpy driven process. These results represented that the compensation theory could be used to evaluate the sorption process of JA powders. According to Silva *et al.* (2014), the water sorption mechanism of JA powders in the experimental a_w range was governed by energy interactions associated with the chemical composition of samples, rather than the resistance of product microstructure to the water molecules. This result is similar with other authors who applied the isokinetic theory to gain insight on the sorption process of different food products, such as blueberry powders (Tao *et al.,* 2018), and dehydrated yacon bagasse (Carvalho Lago and Noreña, 2015). However, the water sorption process of yogurt product was controlled by entropy at low aw, and it is driven by enthalpy at intermediate and high relative humidity ranges (Azuara and Beristain, 2006). Silva *et al.* (2014) reported that there were two lines of compensation in the moisture adsorption for rosemary essential oil microparticles based on the enthalpy-entropy compensation approach. They found that the isokinetic temperature (T_{hm}) at low relative humidity range was 195.75 K, while it was 489.39 K at high a_w range.

The variations of ΔG indicates the affinity of adsorbing matrix to water molecules, and it is an important criterion determining whether the water sorption process is spontaneous ($\Delta G < 0$) or non-spontaneous ($\Delta G > 0$) (Carvalho Lago and Noreña, 2015). The ΔG value for JA powders was 613.11 J/mol, suggesting that these samples did not spontaneously adsorb water from the environment.

Specific surface area

Specific surface area available for the sorption in JA powders was calculated by Eq. (10) using X_M values calculated by GAB model, and the values for sorption process at 20, 30 and 40°C were 286.50, 251.43, and 179.48 m^2/g, respectively. The results indicated that the binding energies related with the mono- and multilayer water to the active sites in JA powders reduced with increasing temperature. This is possibly due to the reduction of ability of JA powders to adsorb water molecules, and reflects the temperature-induced physicochemical modifications. Tao *et al.* (2018) pointed out that the high surface area available for sorption of water was due to the existence of microporous structure in blueberry juice powders, which determined the rate and extent of hydration. Similar behaviours were also reported by Velazquez-Gutierrez *et al.* (2015) for chia seed mucilage powders. These authors found the specific surface area available for sorption of water molecules to freeze-dried chia mucilage to be 277.52, 186.44, and 141.69 m^2/ g at 25, 35, and 40°C, respectively.

Conclusion

This is the first report describing the water sorption properties of Jerusalem artichoke powders. JA powders were amorphous in nature and their mean particle sizes at D_{50} was 69.1 µm. The water sorption isotherms of JA powders in the temperature range of 20 - 40°C were type III based on BET classification. A crossover of water sorption isotherms was observed when the value of a_w close to 0.65. The GAB and Peleg were the optimal models to describe the moisture sorption behaviour of JA powders in the range of a_w and temperature studied. The thermodynamic properties of adsorbed water were determined by applying

GAB models. The monolayer values were 8.08, 7.09, and 5.06 g/100 g dry solids at 20, 30, and 40°C, respectively. Both the net isosteric heat and differential entropy increased with the increase in water content until the monolayer level, and sharply decreased above this point. Enthalpy-entropy compensation theory was successfully used and revealed that sorption mechanism was governed by enthalpy. Valuable monolayer values as well as the thermodynamic properties documented in the present work will help design the energy efficient process for producing JA powders and predicting storage stability.

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