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Research Article

Thermodynamic Study of Adsorption Properties of Rocoto Pepper (*Capsicum pubescens*) Obtained by Freeze-Drying

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Abstract: The aim of this study was to establish a study of the thermodynamic adsorption to rocoto pepper obtained by freeze-drying at temperatures of 15, 25 and 35°C, by the gravimetric method in the range of water activity between 0.131 and 0.847. Rocoto pepper (*Capsicum pubescens*) is a natural source of capsaicinoids which gives the level of pungency or hotness attractive gastronomy. Actually, dehydrated and powered food matrices are a good option to ensure stability and techno-functional properties for later uses and applications. A mathematical modeling of the respective isotherms was obtained using different models reported in the literature. The BET model (Brunauer-Emmett-Teller), GAB (Guggenheim-Anderson-de Boer), Herdenson, Smith, Oswin, Peleg and Caurie, based on the moisture content in equilibrium reached freezer-dried samples pepper. The statistical results indicated that the model GAB was the most appropriate to describe the respective sorption curves, whose behavior was completely sigmoidal type II. From these results, the net isosteric heat of adsorption was determined using the Clausius-Clapeyron, ranging from 52.5 to 46.6 (kJ/mol) a moisture content from 2 to 38% dry basis. Also, Gibb's energy ($\Delta G<0$) showed that the process will proceed spontaneously (exothermic process) which increased to higher moisture content (X_w). The differential entropy (ΔS) was less at 15°C, making the adsorption process thermodynamically favorable at low temperatures.

Keywords: Conservation, fruit, mathematical models, powder, stability

INTRODUCTION

Rocoto pepper (*Capsicum pubescens*), is a fruit native to Bolivia and Perú, is grown in the highlands of Mexico to Argentina (León, 2000). It is characterized by its flower purple and black seeds are morphologically medium-sized apple shaped and bright red (Yamamoto *et al*., 2013). Its pungency level ranges from 100,000 to 210,000 Scoville Hot Units (SHU) (López, 2003), whereby is attractive to hotter gastronomy. However, because of its high average moisture content of 89.5% (García *et al*., 2009), it has been chosen for various unit operations to diversify its presentation and extend the shelf life, from processing in sauces, brines, heat treatments and frozen, fried or blanched and dehydrated (Loizzo *et al*., 2015; Martínez-Girón and Ordóñez-Santos, 2015).

Dehydrated products offer many advantages over the fresh because the moisture is reduced and increase its stability and further storage space is reduced and transport is facilitated. In similar product, Genus Capsicum has been processed by the freeze-drying where the final product presented a greater reconstitution properties with better features in color and with minimal loss of volatile components (Shofian *et al*., 2011; Toontom *et al*., 2012; Topuz *et al*., 2011).

However the storage stability of freeze-dried products is limited by the high hygroscopicity (Serna-Cock *et al*., 2015), therefore studying the adsorption isotherms, the isosteric heat of adsorption, Gibbs energy and glass transition temperature of the freeze-dried food will establish the appropriate packaging and storage controls (Cortes *et al*., 2012).

The adsorption isotherms in dehydrated foods, describe the thermodynamic relationship between water activity and moisture balance of a food product at constant temperature and pressure, allowing to generate predictions of quality, stability, shelf life and calculate packaging requirements and other applications (Andrade *et al*., 2011; Aouaini *et al*., 2015). There are many mathematical models that have been used, which can describe these mechanisms of sorption in food: BET (Brunauer-Emmett - Teller), GAB (Guggenheim-Andersen-de Boer), Henderson, Caurie, Smith, Hasley,

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Oswin, Peleg, Chungamong others (Largo Ávila *et al*., 2014; Rodríguez-Bernal *et al*., 2015; Wu *et al*., 2015).

The thermodynamic properties of the dried products associated with adsorption behavior represent the energy required to break the intermolecular forces between water in the atmosphere and the food surface (Noshad *et al*., 2012). The differential heat of adsorption often referred to as the net isosteric heat of sorption, measured energy changes occurring during the sorption process where $\Delta H<0$ implies the existence of attractive forces in the process. Gibb's energy, spontaneity related process where $\Delta G<0$ represents a spontaneous process, while the differential entropy (ΔS) of a material is proportional to the number of sorption sites available at a specific energy level, associated with the molecular ordering system, if $\Delta S<0$, describes a structured system (Al-Muhtaseb *et al*., 2002; Largo Ávila *et al*., 2014; Martínez *et al*., 1998; Velázquez-Gutiérrez *et al*., 2015).

The aim of the study was to establish a study of the thermodynamics adsorption of the freezer-dried peppers variety "rocoto" at different temperature (15, 25 and 35°C) and water activities of the product. In *Capsicum pubescens*, studies have not been reported previously.

MATERIALS AND METHODS

Material: The raw material used was the pepper variety rocoto (*Capsicum pubescens*), for a total of 10 kg, harvested in the Department of Antioquia (Colombia/South America). The vegetal product was stored at 5°C and 90% RH. The fruits were removed and the stems were cleaved in particle size of 10 mm±2 long, 10 mm±2 wide and 5 mm±1 thick, including the epidermis; treatments contained the seed which gives 13.3% of the total weight. Each Experimental Unit (EU) was 425 g±0.5; then they placed on stainless steel trays the freeze-drying process.

Freeze-drying: The equipment used in the experimental runs was Labconco freezing dryer (7948040 Model). Initially, the raw material was cooled from 25°C to -40°C, with a cooling rate of 0.10°C/min, for a total time of 10.83 h. a heating rate of 0.04°C/min and a holding time between each of the segments of 1.2 h, with a final temperature of 30°C for 3 h, it was used for the sublimation step a total of 36.4 h. After the samples were dried, the sample was subjected to a grinding process (Pulverisette 19, Fritsch Germany), to 8,000 RPM for 5 min, obtaining an average particle size of 50 μm.

Physicochemical determinations: To determine the moisture content of the samples, it was used AOAC 931.04 method using a convection oven (Mermmet, UN160plus). The water activity (a_w) was measured at 25°C with a hygrometer dew point (Aqua LAB Decagon series 3TE) method according to AOAC 978.18 (AOAC, 1990).

Table 1: Relative Humidity (RH) of air in equilibrium with supersaturated saline solutions

	HR		
Salt	15°C	25°C	35°C
Lithium chloride (LiCl)	0.188	0.183	0.131
Potassium acetate (CH_3COOK)	0.285	0.252	0.218
Magnesium chloride ($MgCl_2$)	0.379	0.339	0.328
Potassium carbonate (K_2CO_3)	0.495	0.473	0.445
Copper chloride (CuCl)	0.717	0.709	0.692
Sodium Chloride (NaCl)	0.749	0.760	0.768
Potassium chloride (KCl)	0.847	0.836	0.842

Adsorption isotherms: To obtain the adsorption isotherms, the static gravimetric method was used (Largo Ávila *et al*., 2014), which consists of the determination of the moisture content of the sample after it has reached equilibrium with air of known Relative Humidity (RH). The RH values were obtained by different supersaturated salts in tightly closed containers (Table 1).

Freeze dried samples pepper were weighed to a value of approximately 2 g±0.31 and placed in triplicate in hermetically sealed glass jars a constant temperature of 15, 25 and 35°C using a climate chamber (ICH260C, Memmert). The weight of the sample was measured until to reach the equilibrium moisture content (differences in weights not exceeding 0.1%) Measurements were performed in triplicate. For environments with $a_w>0.7$, it was placed 0.5 g of thymol for to inhibit microbial growth in accordance with the methodology of Largo Ávila *et al*. (2014).

Sorption models: In Table 2 shows the theoretical description of the models used in this study:BET (Brunauer-Emmett-Teller), GAB (Guggenheim-Anderson-de Boer), Herdenson, Smith, Oswin, Peleg and Caurie, which are widely applied to food (Largo Ávila *et al*., 2014; Rodríguez-Bernal *et al*., 2015; Velázquez-Gutiérrez *et al*., 2015; Wu *et al*., 2015).

Thermodynamic properties: The Clausius-Clayperon equation for vapor-liquid equilibrium was applied to the experimental data in order to calculate the enthalpy change for different moisture contents associated with the sorption process (differential heat of adsorption) (Kedzierska and Pałacha, 2012; Muñio *et al*., 2015; Tsami, 1991; Varghese *et al*., 2012). The net isosteric heat sorption (q_{st}) or differential adsorption heat (ΔH) was obtained by the total heat of sorption (Q_{st}) in units of kJ/mol, the Gibbs free energy (ΔG) in units of kJ/mol and entropy (ΔS) in units of J/K.mol by Eq. (1), (2) and (3) respectively:

$$q_{st} = \Delta H = RT^2 \left[\frac{\partial lnP}{\partial T}\right]_{X_w} = Q_{st} + \lambda \quad (1)$$

Table 2: Mathematical models of sorption isotherms

Model	Mathematics equation
BET	$X_w = \frac{X_0 C a_w}{(1-a_w)(1+(C+1)a_w)}$ X_o: Moisture content corresponding to the monolayer product. *C*: Constant of the material related to the heat released in the sorption process.
GAB	$X_w = \frac{X_0 C K a_w}{(1-\mathrm{K}a_w)(1+(C+1)K a_w)}$ X_o: Moisture content corresponding to the monolayer product. *C*: Guggenheim constant related to the heat of adsorption of the monolayer in the GAB model. *k*: Correction constant related to the heat of adsorption of the monolayer in the GAB model.
HENDERSON	$X_w = 0{,}01\left[\frac{-\log(1-a_w)}{10^f}\right]^{\frac{1}{n}}$ *f and n:* Parameters in the model.
SMITH	$X_w = \mathrm{B} + \mathrm{A}\log(1-a_w)$ *A and B:* Constants in the model.
OSWIN	$X_w = \mathrm{A}\left[\frac{a_w}{1-a_w}\right]^B$ *A* and *B*: Constants in the model.
PELEG	$X_w = A{a_w}^B + C{a_w}^D$ *A, B, C* and *D:* Constants in the model.
CAURIE	$X_w = \exp\left[a_w \ln(R) - \frac{1}{4{,}5X_s}\right]$ *R:* Parameter in the model. X_s: Moisture content security provides maximum stability dehydrated food during storage (kg H2O / kg dry solid)

$$\Delta G = RTln\left(\frac{P}{P^o}\right) = RTln(a_w) \quad (2)$$

$$\Delta S = \frac{\Delta H - \Delta G}{T} \quad (3)$$

where,

- R = The universal gas constant (8.314 J/mol.K)
- λ = The latent heat of vaporization of pure water at 25°C (43.96 kJ/mol)
- T = The temperature adsorption in kelvin (K)
- P = The partial pressure of water vapor in equilibrium with the material at a given temperature
- P° = The vapor pressure of pure water
- a_w = Water activity (Prokopiuk *et al*., 2010)

Statistic analysis: The model parameters were estimated by software DATAFIT non-linear regression, version 9.0.59 (Oakdale Engineering). For each model it defined; the coefficient of determination (R^2), Mean Square Error (MSE) and the square root of the mean error (E_{RMS}) (Largo Ávila *et al*., 2014). For the goodness of fit, the highest value of R^2 and the lowest values of MSE and E_{RMS} was chosen:

$$\mathrm{MSE} = \left(\frac{\sum_{i=1}^{N}(M_{R,exp,i} - M_{R,pre,i})^2}{\mathrm{N-z}}\right) \quad (4)$$

$$E_{RMS} = \left[\frac{1}{N}\sum_{i=1}^{N}(M_{R,exp,i} - M_{R,pre,i})^2\right]^{1/2} \quad (5)$$

where, $M_{R,exp,i}$, y $M_{R,pre,i}$, are experimental and calculated for each model of the equilibrium moisture (X_w), N is the number of observations and z is the number of constants for each model.

RESULTS AND DISCUSSION

The initial moisture content of pepper rocoto dehydrated by freezing-drying was 7.62±0.67 (% dry basis) and water activity (a_w) of 0.28±0.01. Figure 1 shows the experimental isotherms at different temperatures, indicating the relationship between water activity and equilibrium moisture content.

Isotherms show a sigmoidal shape type II, normally found in nonporous foods such as vegetables rich in carbohydrates (Khawas and Deka, 2016; Rodríguez-Bernal *et al*., 2015; Téllez-Perez *et al*., 2015). It was observed that the X_w increased as the value of a_w increased where the product adsorbed small amounts of water at low and intermediate values

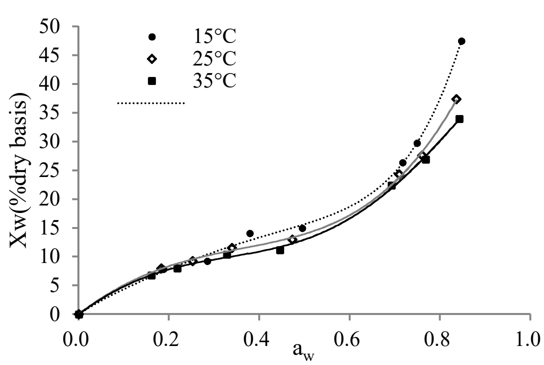

Fig. 1: Adsorption isotherms of pepper variety "rocoto" obtained by freezing drying

Table 3: Parameters of rocoto pepper powder obtained with various sorption mathematical models

Model	Variables	15°C	25°C	35°C
BET	X_o	0.07409	0.06415	0.05823
	C	93.1427	46.5934	45.4460
	R^2	0.99413	0.98991	0.97428
	CME	0.00013	0.00018	0.00040
	$E_{(RMS)}$	0.01074	0.01238	0.01854
GAB	X_o	0.08151	0.08143	0.08110
	K	0.97938	0.93944	0.91307
	C	21.2189	18.8732	17.3244
	R^2	0.99522	0.99891	0.99504
	CME	0.00011	0.00002	0.00009
	$E_{(RMS)}$	0.00885	0.00406	0.00814
HENDER-SON	F	(3.7634)	(4.0360)	(4.1995)
	N	1.12900	1.21084	1.25984
	R^2	0.96893	0.96754	0.95873
	CME	0.00100	0.00069	0.00077
	$E_{(RMS)}$	0.02670	0.02221	0.02348
SMITH	A	0.01095	0.02290	0.02579
	B	(0.2267)	(0.1863)	(0.1689)
	R^2	0.97123	0.98331	0.98314
	CME	0.00090	0.00025	0.00018
	$E_{(RMS)}$	0.02535	0.01336	0.01141
OSWIN	A	0.15955	0.15240	0.14554
	B	0.61707	0.53776	0.50776
	R^2	0.98777	0.99552	0.99273
	CME	0.00034	0.00010	0.00014
	$E_{(RMS)}$	0.01551	0.00825	0.00985
PELEG	A	0.25356	0.17914	0.43048
	B	0.71786	0.48512	3.32949
	C	0.97760	0.54789	0.09879
	D	8.20083	5.39200	0.15485
	R^2	0.99781	0.99907	0.99910
	CME	0.00012	0.00003	0.00003
	$E_{(RMS)}$	0.00709	0.00375	0.00346
CAURIE	Xs	0.06503	0.06799	0.06767
	R	21.58517	14.58250	13.45992
	R^2	0.96264	0.97235	0.97626
	CME	0.00098	0.00030	0.00016
	$E_{(RMS)}$	0.02652	0.01459	0.01079

(a_w<0.6), followed by a linear increase with increasing moisture adsorbing activity water (a_w>0.6), introducing the capillary condensation region (Timmermann *et al*., 2001). This behavior indicates that *Capsicum pubescens* obtained by freeze-drying require values of Relative Humidity (RH) no greater than 60% to ensure its stability under storage conditions.

It was noted that increases in temperature tend to diminish the moisture content (X_w) of the product keeping the water activity constant (a_w). Similar results have been reported for freezing dried food where increases in temperature results in a loss of moisture (Rodríguez-Bernal *et al*., 2015; Velázquez-Gutiérrez *et al*., 2015; Wu *et al*., 2015).

Sorption models: Table 3 shows parameters: R^2, MSE and $E_{(RMS)}$ of sorption models used, five of which showed a goodness of fit adjustment R^2>0.97 (BET, GAB, Smith, Oswin and Peleg). The GAB and Peleg model which showed better adjustments with regard to the experimental data, presenting the higher coefficients of determination for all temperatures tested and the lower values of MSE and $E_{(RMS)}$. Figure 2 shows the experimental adsorption isotherms and predicted, according to a mathematical model at different temperatures.

The advantage of GAB and BET models from the model Peleg, is providing the parameter X_o (moisture content in the monolayer), this being essential to determine stability of the food product (Andrade *et al*., 2011), although the GAB model is more effective for dehydrated foods whose a_w<0.4 (Dos Santos *et al*., 2015; Largo Ávila *et al*., 2014).

The value in both models for X_o, there is a decrease with increases of temperature, this behavior is possibly due to increased activation of water molecules induced by the temperature increase, reducing the active points on the surface of the food and so both a decrease in the X_w addition also occurs with increasing temperature of 25°C and 35°C with the observed parameter X_s (moisture content security) according to Caurie model (Cortes *et al*., 2012; Téllez-Perez *et al*., 2015; Wu *et al*., 2015).

The constant C relates the heat of sorption of the first layer, with values from 21.22 to 17.32 and the value *k*, represents a correction factor related to the heat of sorption of the multilayers, the results the product showed values from 0.97 to 0.91, where these

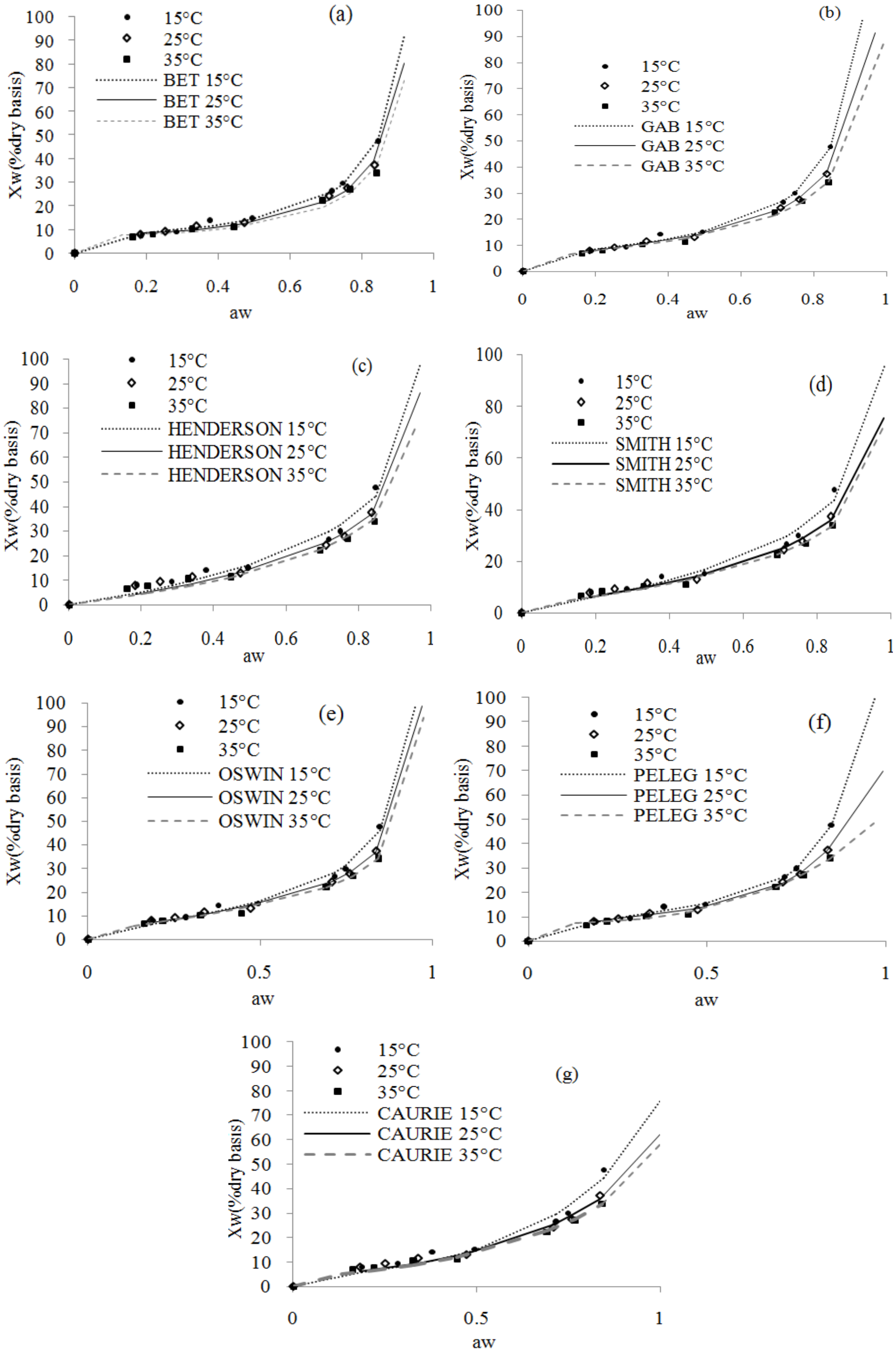

Fig. 2: Comparison of experimental models isotherms: (a): BET; (b): GAB; (c): Herdenson; (d): Smith; (e): Oswin; (f): Peleg (g): and Caurie

parameters decrease with the increasing temperature, similar results have been reported by Téllez-Perez *et al.* (2015) and where the *k* value should be less than the unit (Chirife *et al*., 1992). Strong interactions between adsorbent-adsorbate indicate an exotherm caused froman increase in the C parameter with decreasing

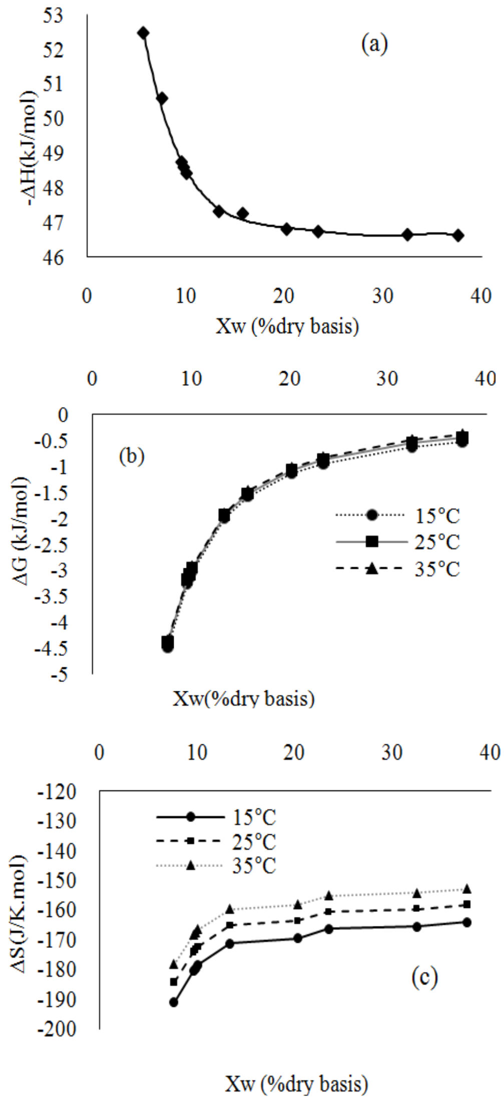

Fig. 3: Thermodynamic properties of pepper rocoto obtained by freezing drying; (a): ΔH; (a): ΔG; (c): y ΔS

temperatures (Dos Santos *et al*., 2015; Rodríguez-Bernal *et al*., 2015).

Thermodynamic properties: Figure 3a shows behavior q_{st} or ΔH for freezing-dried pepper using the GAB model with respect to moisture content. The result of $-\Delta H>0$ implies an exothermic nature of the adsorption process. The q_{st} (kJ/mol) tends to approach the value the latent heat of vaporization of free water (43.96 kJ/kg) with increasing water content of the product varying from 52.5 to 46.6 for moisture contents from 2 to 38 (% dry basis) respectively. Also, the results show that at low moisture content ($X_w<10\%$) possible resistance to water movement from the inside to the surface and indicative of more polar sites on the surface of the material (Largo Ávila *et al*., 2014; Rodríguez-Bernal *et al*., 2015; Ouafi *et al*., 2015). Additionally, it was found that the higher moisture content the q_{st} is closer to the heat of evaporation of free water due to a weakening of hydrogen bonds between the active sites (Cortes *et al*., 2012; Dos Santos *et al*., 2015). In similar studies, it has reported the exothermic nature of adsorption at freezing -dried products (Amaral *et al*., 2016; Rodríguez-Bernal *et al*., 2015; Wu *et al*., 2015).

Figure 3b and 3c show thermodynamic properties ΔG and ΔS respectively with respect to different moisture contents and temperatures. The tendency of the free energy of Gibb's ($\Delta G<0$) indicates the spontaneity of the adsorption of water to the product, which energy tends to zero as X_w increases, where an increase in temperature causes a diminishing in the adsorption capacity. In addition, these changes indicate that the lyophilized product is a hygroscopic product, showing that ΔG increases when there is a change from lower to higher X_w, due to spontaneity to capture water molecules. Similar results have been reported by other researchers (Cortes *et al*., 2012; Muñio *et al*., 2015; Wu *et al*., 2015). The physical explanation for the rapid increase in ΔH at low moisture contents may be that in the initial stages of sorption there are highly active polar sites on the surface of the food material, which are covered with water molecules to form a monomolecular layer (Goula *et al*., 2008).

The differential entropy ($\Delta S<0$) indicates a structured system, showing a strong dependence of moisture content. The entropy change is higher in temperature of 35°C because the kinetic energy of the interacting molecules during the process of water exchange is directly proportional to temperature, while at 15°C the entropy of the system is lower due to restricting the movement of water molecules, thereby making the adsorption process thermodynamically favorable at lower temperaturas (Domínguez *et al*., 2007).

Rocoto pepper freezing- dried showed a decrease in entropy reaching a minimum of 9 and 13% dry basis, then increased in magnitude as the moisture content increases. The decrease of the integral entropy possibly an increase in the restricted mobility of water molecules as the available sites are saturated and further increase implies that water molecules are free to form multilayers, the similar result was found by Velázquez-Gutiérrez *et al*. (2015).

CONCLUSION

Adsorption isotherms of freezing-dried pepper variety "rocoto" at 15, 25 and 35°C showed a sigmoid shape type II. The GAB model was the most suitable to describe sorption curves according with the temperatura and water activity range investigated.

The net isosteric heat of sorption and differential entropy values show a strong dependence of moisture content moisture content. The adsorption process thermodynamically is favorable at low temperaturas.

CONFLICT OF INTEREST

The authors declare that there is no conflict of interest with any financial organization regarding the material discussed in the manuscript.

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