

SORPTION ISOTHERMS AND THERMODYNAMIC ANALYSIS OF SEED FRUITS USED TO OBTAIN VEGETABLE OIL

K.S. SILVA, J.T. ROMERO and M.A. MAURO

Department of Food Engineering and Technology, Institute of Biosciences, Language and Physical Sciences (IBILCE), UNESP – São Paulo State University Rua Cristóvão Colombo 2265, 15054-000 São José do Rio Preto, SP, Brazil. e-mail: keiolasouzas@yahoo.com.br

Abstract - Sorption isotherms of papaya, melon and grape seeds were determined and thermodynamic properties compared. The experiments were carried out at different temperature using the gravimetric method. The Henderson and GAB models were the best which represented the experimental data. Papaya and grape seeds are more stable with larger moisture content than melon seeds at 30°C. Grape seeds presented more resistance to lose water during the dehydration. The differential enthalpy and entropy decreased with increasing moisture content and satisfied the compensation theory. It was found that the sorption process investigated was enthalpy-driven.

Keywords— seeds, grape seeds, melon seeds, papaya seeds, thermodynamics.

I. INTRODUCTION

Much of the fruit production is destined for processing candies, extracts, juices and pulps. Each processing, tons of waste are generated and discarded, causing problems to the environment and to the companies. Some studies emphasize the use of seeds from industrial discharges to obtain vegetable oils (Arvanitoyannis *et al.*, 2006; Jorge and Malacrida, 2008; Jorge *et al.*, 2009). Besides being consumed in the food, this vegetable oil can be used in the chemical industry, pharmaceutical and biodiesel production. Malacrida (2009) noted high lipid content in Papaya seeds (*Carica papaya* L.) and melon seeds (*Cucumis melo inodorus*) when compared to the content found in soybean seeds and indicated the studied seeds as a good source of oil. The study showed that the papaya and melon seeds presented higher protein content than that found in rice, corn, oats and wheat. According to Crews *et al.* (2006), grape seed oil has a high content of unsaturated fatty acids and phytosterols, suitable for a diet to reduce cholesterol levels. For a large scale production is necessary to know some characteristics of seeds and their stability attributes, as well as the process variables, because to obtain vegetable oil is necessary that the seeds are dehydrated. The knowledge of the existent relations between the temperature and the air relative moisture is very important to control microbial growth and ensure seed quality during storage, because these tend to exchange moisture with the atmosphere surrounding them. This exchange of moisture content can be increased or reduced according to the hygroscopicity of the seeds and the gradient of potential hydric between the air atmospheric and the seeds (Rizvi, 1986).

The relationship between the water activity and equilibrium moisture content of a product subject to a specific temperature are expressed graphically by the curves of sorption isotherms (Rizvi, 1986). These curves are obtained at constant temperature and pressure and vary with the physical and chemical structure of food (Chirife and Iglesias, 1978). When the product is a solid, the water interactions occur not only in aqueous solution, but also depend on factors such as capillarity and hydration forces, responsible for binding more strongly the water to insoluble macromolecules of food. For this reason the relation between the equilibrium moisture and the water activity in solid foods is usually determined experimentally for each product. From the graphs of sorption isotherms point it is possible to determine thermodynamic functions such as enthalpy differential, which provides informations on the state where water is present in a biological material (Fasina *et al.*, 1997) and the differential entropy, which informs the number of sorption sites for a given level of energy inherent in biological materials (Madamba *et al.*, 1996). This manner it is possible to predict the required energy consumption for the dehydration process as well as the more stable conditions of seeds to the storage.

According to Ferro-Fontan *et al.* (1982), some foods present a linear correlation between the enthalpy and entropy for water sorption. This linearity is represented by the compensation theory and confirmed by several researchers (Madamba *et al.*, 1996; Telis *et al.*, 2000; McMinn *et al.*, 2005; Fasina, 2006). The slope of the straight represents the temperature at which all reactions in series proceed at the same rate: the isokinetic temperature (T_B).

This work aimed to determine and model the sorption isotherms of papaya seeds, melon seeds and grape seeds using several mathematical models, to determine and compare some thermodynamic properties of the studied seeds and to evaluate the application of the enthalpy-entropy compensation theory.

II. MATERIALS AND METHODS

A. Materials

Sorption isotherms of seeds of grape (*Vitis vinifera*), papaya (*Carica papaya* L.) and melon (*Cucumis melo inodorus*) were accomplished. The seeds were maintained in cold chamber (5°C) until the experiment. Saturated aqueous salt solutions were prepared using NaOH, LiCl, $KC_2H_3O_2$, $MgCl_2$, K_2CO_3 , $Mg(NO_3)_2$, $NaNO_2$, NaCl, KCl, $BaCl_2$ and $CuSO_4$ (all analytical grade) and commercial formaldehyde (40%) was used for cleaning the material and preventing microbial spoilage of samples.

Table 1: Mathematical models applied to the experimental sorption data.

Models	Equation
GAB (Van den Berg and Bruin, 1981)	$X = \frac{X_m \cdot C \cdot K \cdot a_w}{(1 - K \cdot a_w)(1 - K \cdot a_w + C \cdot K \cdot a_w)} \quad (1)$
Peleg (Peleg, 1993)	$X = (A \cdot a_w^B) + (C \cdot a_w^D) \quad (2)$
Oswin (Oswin, 1946)	$X = A \left[\frac{a_w}{(1 - a_w)} \right]^B \quad (3)$
Henderson (Henderson, 1952)	$X = \left(\frac{\ln(1 - a_w)}{-A} \right)^{\frac{1}{B}} \quad (4)$

where a_w is water activity, dimensionless; X is equilibrium moisture (% dry basis); X_m is moisture content of monolayer (% dry basis); A , B , C , D and K are parameters of isotherm equations.

B. Samples

The seeds were separated from fruits waste by hand, washed quickly with distilled water and dried in tray at room temperature. The seeds were homogenized and destined to the experiment.

C. Sorption isotherms

Sorption isotherms were plotted for the seeds, based on static gravimetric methods proposed by Jowitt *et al.* (1987). Ten saturated aqueous salt solutions were prepared corresponded to water activity intervals between 0.02 to 0.97. Duplicate samples were weighed into small plastic receptacles and placed on tripods in the jars, which were then tightly closed and placed in a temperature-controlled chamber. The interval of studied temperature was from 30°C to 70°C. The required equilibration time was 4 to 5 weeks based on the change in weight expressed on a dry basis, which did not exceed 0.1% (0.001g/g dry solids). The equilibrium moisture content was measured gravimetrically in triplicate, drying to constant weight using a vacuum oven at 60°C.

D. Data analysis

Sorption Isotherms

For the mathematical description of sorption isotherms were adjusted four mathematical models to the experimental data (Table 1) using the non-linear regression module of Statistica 7.0 software (Statsoft, Tulsa, OK, USA). Following the methodology proposed by Peleg (1993), regressions were repeated with various initial estimated values both above and below those that had been calculated to confirm whether or not convergence were reliable.

The goodness of fit of the different models was evaluated with the regression coefficient (R^2) and the mean relative deviation modulus (M_e). Based on Eq. (5), Lomauro *et al.* (1985) considered mean relative percent deviation values below 10% a reasonable adjustment for the proposal practices.

$$M_e = \frac{100}{n} \sum_{i=1}^n \frac{|(M_i - M_{pi})|}{M_i} \quad (5)$$

where M_i is experimental moisture content values (kg water kg⁻¹ dry solids), M_{pi} is moisture content values predicted by the model (kg water kg⁻¹ dry solids) and n is number of experimental data.

Thermodynamic properties

The differential enthalpy of water sorption (Δh) or net isosteric heat of sorption, is defined as the difference between the total differential enthalpy of sorption (ΔH) point it and the molar enthalpy of vaporization of pure water (kJ/mol) (ΔH_{vap}) and can be determined from Eq. (6), which is derived from the Clausius – Clapeyron equation, applied to food and pure water (Rizvi, 1986).

$$\left. \frac{\partial \ln(a_w)}{\partial 1/T} \right|_x = \frac{\Delta h}{R} \quad (6)$$

where R is the gas constant (8.314 J.mol⁻¹.K⁻¹), a_w the water activity and T the temperature (K).

Assuming that the sorption differential enthalpy be independent of temperature changeness, $\ln(a_w)$ was plotted versus $1/T$ for a specific moisture content of the material and determined the slope of the curve, which equals ($\Delta h/R$) and the linear coefficient of the curve ($\Delta s/R$) where Δs is the differential entropy (J.mol⁻¹.K⁻¹) (Tsami *et al.*, 1990; McMinn and Magee, 2003). The model that best fitted the experimental data was used to determine the value for each moisture content in order to determine the dependence of a_w on the moisture content (X), at each temperature. This way, the differential enthalpy and entropy of sorption can be determined from moisture sorption data by using the Eq. 7 (Fasina *et al.*, 1997):

$$(\ln a_w)_x = \frac{\Delta h}{RT} - \frac{\Delta s}{R} \quad (7)$$

According to Ferro-Fontan *et al.* (1982), some foods present a linear relationship between the enthalpy and entropy for water sorption. This linear relationship is represented by compensation theory (Eq. 8) and confirmed by several other authors (Madamba *et al.*, 1996; Telis *et al.*, 2000; McMinn *et al.*, 2005; Fasina, 2006). The slope of the straight represents the temperature at which all reactions in series proceed at the same rate: the isokinetic temperature (T_B).

$$\Delta h = T_B (\Delta s) + \Delta G_B \quad (8)$$

where ΔG_B is the free energy at T_B .

The isokinetic temperature was compared with harmonic mean temperature to confirm the compensation theory, as recommended by Krug *et al.* (1976). According to the author, if the temperatures are different, the compensation theory can be applied for the studied product. The Eq. 9 defines the harmonic mean temperature, where n represents the temperatures number used.

$$T_{hm} = \frac{n}{\sum_{i=1}^n (1/T)} \quad (9)$$

The Eq. 10 expresses an approximate (1- α)100% percent confidence interval for isokinetic temperature, where T_B and $\text{Var}(T_B)$ are calculated according to Eqs. 11 and 12, respectively.

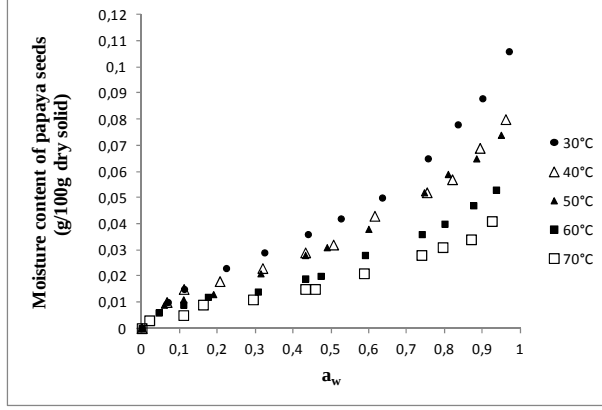


Fig. 1. Experimental values of equilibrium moisture content (dry basis) as a function of water activity for papaya seeds at different temperatures.

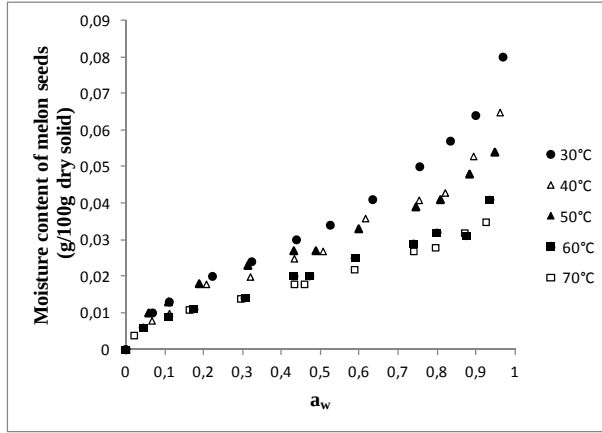


Fig. 2. Experimental values of equilibrium moisture content (dry basis) as a function of water activity for melon seeds at different temperatures.

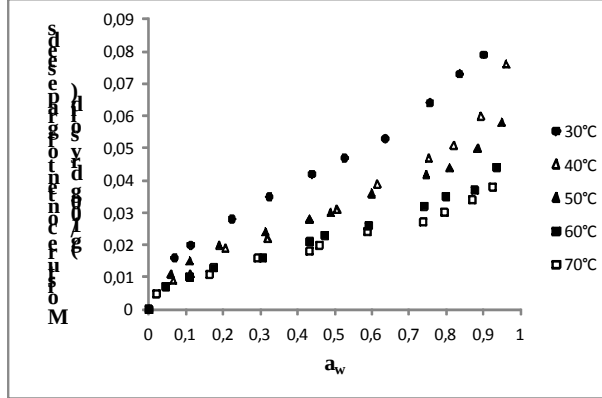


Fig. 3. Experimental values of equilibrium moisture content (dry basis) as a function of water activity for grape seeds at different temperatures.

$$T_B = T_B \pm t_{m-2, \alpha/2} \sqrt{\text{Var}(T_B)} \quad (10)$$

$$T_B = \frac{\sum (\Delta H - \overline{\Delta H})(\Delta S - \overline{\Delta S})}{\sum (\Delta S - \overline{\Delta S})^2} \quad (11)$$

$$\text{Var}(T_B) = \frac{\sum (\Delta H - \Delta G_B - T_B \Delta S)^2}{(m-2) \sum (\Delta S - \overline{\Delta S})^2} \quad (12)$$

where m is the number of data pairs of enthalpy and entropy; $\overline{\Delta H}$ is the mean differential enthalpy (J.mol^{-1});

$\overline{\Delta S}$ is the mean differential entropy (J.mol^{-1}) and $\overline{\Delta G_B}$ is the Gibbs free energy at isokinetic temperature (J.mol^{-1}).

III. RESULTS AND DISCUSSION

The sorption isotherms of papaya, melon and grape seeds obtained experimentally are presented in the Fig. 1, 2 and 3, respectively.

The isotherms of all seeds showed the typical sigmoid shape of Type II, according to the BET classification, describing the seeds as no porous or macroporous materials with low sorption energy (Condon, 2006). The same format was observed for quinoa seeds (Miranda *et al.*, 2012), pumpkin seeds (Mayor *et al.*, 2005), pea seeds (Chen, 2003) and lentil seeds (Nikolay, 2000).

Observe that the effect of temperature in sorption isotherms is very pronounced and as the temperature increases occurs a fall in the equilibrium moisture content of seeds, resulting in less hygroscopic product. This behaviour, generally, ascribed to a reduction in the number of active sites, due to the physical and chemical changes induced by temperature (Rizvi, 1986). The reduction of these sites would result less energy for the interaction with the water molecules resulting in less hygroscopic seeds (Ascheri *et al.*, 2009).

For the grape and melon seeds, the Henderson model presented the best fit to the experimental data showing higher values of regression coefficient (R^2) and smaller relative percent deviation (M_e) (below of 10%), already for papaya seeds, the GAB model presented the best fit to the experimental data. The results of nonlinear regression analysis of experimental data, (R^2) values and the mean relative percent deviation values (M_e) are presented in Table 2.

Mayor *et al.* (2005) noted GAB, Peleg and Henderson models were the best fit the experimental data for pumpkin seeds. For tomato seeds, Sogi *et al.* (2003) observed that Henderson model was the best fit to the experimental data. Francisco *et al.* (2007), although, noted Oswin and Peleg models were the best fit for Seed of two bean cultivars. The Peleg model, for presenting four parameters, almost ever result in an excellent fit to the sorption isotherms data, thus the resultant parameters of this model are empirical.

The GAB model is much used to foods because it presents physical meaning. The moisture content corresponding to an adsorbed monolayer (X_m), provided by this model has a remarkable importance in food storage and deterioration study, because represents the quantity of water that is strongly bounded to the actives sites and aid to define the chemical and physical stability.

Observe in Table 2 the moisture of monolayer obtained for each seed tend to reduce with temperature. The increase in temperature provides an increase on disorder of water molecules resulting in the breaking of the intermolecular bounds between water molecules and the sorption sites present in seeds and following decreasing the monolayer moisture content (Hossain *et al.*, 2001).

The papaya and grape seeds showed bigger and similar monolayer moisture (about de 3.1g/100g dry solid) than melon seeds at 30°C (2.34g/100g dry solid), indicating that the first seeds hold bigger stability at high moisture (Table 2). However, from 60°C was not observed more difference between monolayer moisture of seeds, probably due to chemical and physical changeness provided by temperature. According to Vishwakarma *et al.* (2011), a product with moisture content equal to the moisture of monolayer can be storage for long periods with a minimum quality loss. Mayor *et al.* (2005) observed moisture monolayer values for pumpkin seeds at 25°C around 5.65g/100g dry solid.

To determine the value of a_w for each equilibrium moisture content, was chosen the Henderson model, because it was the best fit for the majority seeds. The differential enthalpy (Δh) was calculated according to the Eq. 7. Due to the bigger value of differential enthalpy (values less negatives) (Fig. 4), note that grape seeds present bigger resistance to lose water and thus, requires a larger spend energetic for its dehydration. This occurs due to the water molecules in grape seeds being strongly bounded in the monolayer form.

The differential entropy values (Δs) were determined through linear regression using Eq. 7. The differential entropy in function of equilibrium moisture content is presented in Fig. 5. Observe a decreasing of differential entropy values with increasing in moisture of seeds that can be related.

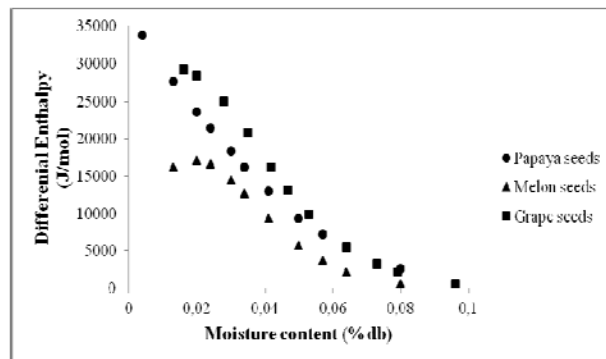


Fig. 4. Differential enthalpy of papaya seeds, melon seeds and grape seeds as a function of equilibrium moisture content.

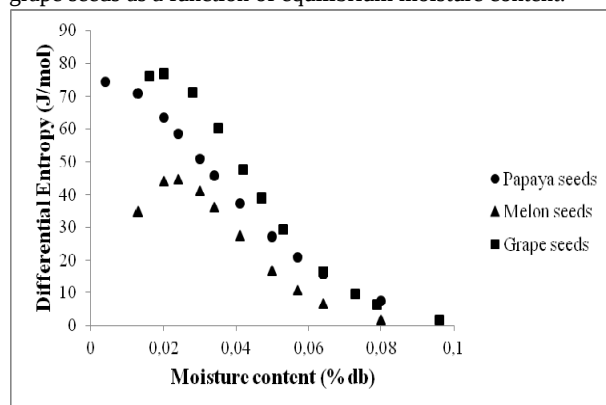


Fig. 5. Differential entropy of papaya seeds, melon seeds and grape seeds as a function of equilibrium moisture content.

Table 2: Estimated values of coefficients, regression coefficient (R^2) and relative percent deviation (M_e) obtained for selected sorption models applied to experimental sorption data for papaya seed, melon seed and grape seed.

Mathematical models	Parameters, R^2 and M_e	Papaya				
		T (°C)				
		30	40	50	60	70
GAB	X_m	0.031	0.024	0.030	0.017	0.0136
	C	8.153	11.281	5.000	9.514	7.304
	K	0.747	0.739	0.671	0.752	0.741
	R^2	0.999	0.997	0.995	0.994	0.994
	M_e	1.528	4.793	5.208	7.664	10.634
Peleg	A	0.525	0.422	0.252	0.000	0.301
	B	4.481	3.589	2.103	0.369	3.035
	C	0.011	0.002	0.000	0.287	0.000
	D	0.631	0.503	0.396	2.584	0.525
	R^2	0.999	0.996	0.999	0.998	6.723
	M_e	1.629	3.326	2.826	4.556	0.994
Oswin	A	0.041	0.033	0.032	0.022	0.017
	B	0.300	0.301	0.328	0.350	0.371
	R^2	0.945	0.957	0.943	0.970	0.977
	M_e	20.617	14.953	18.642	12.272	13.623
Henderson	A	143.683	275.064	213.768	319.368	402.632
	B	1.679	1.774	1.674	1.632	1.579
	R^2	0.995	0.994	0.989	0.992	0.994
	M_e	3.600	5.363	6.760	8.700	8.951
Melon						
GAB	X_m	0.023	0.020	0.021	0.016	0.014
	C	12.512	13.232	20.051	13.861	19.599
	K	0.734	0.728	0.648	0.638	0.654
	R^2	0.999	0.992	0.998	0.981	0.998
	M_e	1.718	5.133	2.555	6.436	4.108
Peleg	A	0.586	0.692	0.514	0.007	0.000
	B	6.244	9.553	5.863	0.667	0.470
	C	0.009	0.010	0.002	1.024	0.312
	D	0.629	0.670	0.495	116.540	3.671
	R^2	0.998	0.993	0.997	0.989	0.998
	M_e	2.214	5.207	2.035	6.338	1.462
Oswin	A	0.033	0.027	0.028	0.020	0.019
	B	0.280	0.292	0.253	0.282	0.278
	R^2	0.960	0.967	0.970	0.955	0.977
	M_e	15.253	13.705	9.415	13.238	10.549
Henderson	A	373.01	550.516	2657.15	2856.74	4711.37
	B	1.852	1.870	2.330	2.151	2.244
	R^2	0.999	0.993	0.997	0.980	0.999
	M_e	1.411	4.402	2.387	4.654	1.703
Grape						
GAB	X_m	0.032	0.022	0.023	0.017	0.014
	C	16.974	12.362	22.295	16.305	24.993
	K	0.686	0.744	0.645	0.656	0.676
	R^2	0.998	0.996	0.998	0.995	0.995
	M_e	1.940	4.027	1.779	4.206	5.629
Peleg	A	0.575	0.687	0.523	0.380	0.467
	B	5.946	8.850	5.926	4.244	5.393
	C	0.007	0.014	0.001	0.001	0.001
	D	0.544	0.690	0.479	0.515	0.476
	R^2	0.999	0.997	0.998	0.996	0.997
	M_e	1.650	4.431	2.101	2.953	3.596
Oswin	A	0.047	0.030	0.030	0.022	0.020
	B	0.247	0.303	0.249	0.279	0.278
	R^2	0.957	0.973	0.974	0.971	0.984
	M_e	15.646	13.687	8.092	10.592	8.244
Henderson	A	539.17	329.60	2717.98	2445.89	4337.68
	B	2.155	1.783	2.387	2.169	2.260
	R^2	0.999	0.998	0.998	0.996	0.996
	M_e	1.019	3.313	2.028	2.599	3.797

Table 3: Temperature isokinetic, Gibbs free energy at isokinetic temperature and regression coefficient obtained for papaya seeds, melon seeds and grape seeds.

Seeds	$T_B(K)$	$\Delta G_B (J.mol^{-1})$	R^2
Papaya seeds	423.41 ± 27.05	-1948.80	0.971
Melon seeds	370.22 ± 7.58	-383.31	0.966
Grape seeds	387.87 ± 9.70	-603.64	0.995

Melon seeds showed smaller differential entropy (values more negatives), indicating high mobility water molecules and, consequently, less difficulty for dehydration.

The entropy values correspondent to the monolayer values (Table 2) of papaya, melon and grape seeds were: 50.96; 44.73; 60.28 J/mol.K, respectively.

The correlation between Δh and Δs for the tree studied seeds, established through Eq. 8, introduced linear behaviour, indicating the existence of compensation theory for papaya, melon and grape seeds. Isokinetic temperature (T_B), Gibbs free energy at isokinetic temperature (ΔG_B) and R^2 are showed in Table 3.

Harmonic mean temperature (T_{hm}), calculated according to the Eq. 5, was 322.38K. According to Leffler (1955), the process is enthalpy-driven when $T_B > T_{hm}$ and entropy-controlled when $T_B < T_h$. Note that all seeds showed $T_B > T_{hm}$ indicating enthalpy-driven process (Table 3), in other words, water vapor sorption mechanisms of papaya, melon and grape seeds are controlled by energetic interaction and not by seeds microstructure. Thys *et al.* (2010) noted the same behaviour for *Araucaria angustifolia* seeds, thus isokinetic temperature calculated by authors ($421 \pm 17K$) was very close to the ones which were obtained for papaya seeds.

The clarification about the hygroscopicity of the papaya, melon and grape seeds and its water vapor sorption mechanisms, obtained in this study, is very important to control microbial growth and to ensure seed quality during storage.

IV. CONCLUSIONS

Between studied models (Henderson, GAB, Peleg and Oswin), The Henderson model was the best to represent the experimental data obtained to construct of sorption isotherms of grape seeds and melon seeds. For papaya seeds, though, the GAB model was the best fit to experimental data. Papaya and grape seeds are stabler with larger moisture content than melon seeds at 30°C. Grape seeds present bigger enthalpy differential value and bigger differential entropy value, indicating, consequently, more resistance to lose water during dehydration of product. Plots of differential enthalpy versus differential entropy satisfied the enthalpy–entropy compensation theory (isokinetic theory). The results suggest that the sorption process of papaya seeds, melon seeds and grape seeds are enthalpy-driven.

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