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Thermodynamic properties of water sorption isotherms of grape seed

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A b s t r a c t. In this study the moisture sorption isotherm of grape seed was determined by using a static gravimetric method at 35-65°C and 0.108-0.821 water activity range. The sorption isotherms were found to be typical sigmoid shape of most food materials. Five models including the Brunauer-Emmett-Teller (2-parameter), Guggenheim, Anderson and De Boer (3-parameter), Oswin (2-parameter), Ferro-Fontan (3-parameter) and Peleg (4-parameter) models were considered to fit the experimental data. The Ferro-Fontan and Peleg equations (at three temperatures 35, 45, 65°C) having R^2 greater than 0.97 and lower values of standard error of estimate and deviation modulus gave the best fit of the experimental data throughout the entire range of water activity. The net isosteric heat of sorption, calculated by Calusius-Clapeyron equation on experimental data, was found to be a polynomial and exponential function of equilibrium moisture content within the temperature range investigated.

K e y w o r d s: moisture sorption isotherm, thermodynamic properties, grape seed

INTRODUCTION

Grapes (*Vitis vinifera*) originated in Western Asia and Europe. It accounts for about 16% of the global fruit production. Among the major grape producing countries, Iran with the production of 3 mln t ranks the seventh after Turkey (FAO, 2009). Grape seed, a by-product of grape processing, is a well-known oilseed containing about 8-20% oil, 11% protein, and 10-20% polyphenolic compounds like tannins, sugars, minerals and other substances (Campos *et al.,* 2008).

Drying grape seed is a critical processing step in the preparation of the seeds for oil extraction. Moisture reduction is necessary to increase shelf-life and to prevent microbial and chemical deterioration. Drying also facilitates handling conditions and reduces storage costs (García-Pérez *et al.,* 2008). In order to describe the process of drying and its effect on water activity (a_w), knowledge of the relationship between equilibrium moisture content (EMC) and water activity/equilibrium relative humidity (ERH) is essential. This relationship, known as moisture sorption isotherm, depends on structure, composition, as well as pressure and temperature (Amiri-Chayjan and Esna-Ashari, 2010, Chowdhury *et al.,* 2006). It is useful for modelling, designing and optimization of the drying process. The water sorption isotherms can also be used to calculate the net heat of sorption which provides the energy requirements associated with the sorption behaviour. The differential heat of sorption is an indicator of the state of water adsorbed by the solid particles, and its knowledge is important in designing drying unit operation (Chowdhury *et al.,* 2006; Goula *et al.,* 2008).

Numerous mathematical models have been used to describe the moisture sorption behaviour of foods. Van den Berg and Bruin (1981) gave more than 200 equations that had been developed theoretically, semi-theoretically and empirically to model the sorption isotherms of different biological materials. Some of those models are based on theories on the sorption mechanism, others are purely empirical or semi-empirical. In general, the models can be divided into several categories: kinetic models based on a monolayer (Mod-BET model), kinetic models based on a multilayer and condensed film (GAB), semi-empirical (Ferro-Fontan, Henderson, and Halsey), and empirical models (Smith, Peleg, and Oswin).

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The main objective of this study was to provide experimental and modelled sorption isotherm data of grape seed at various temperatures (35, 45, 55 and 65°C).

MATERIALS AND METHODS

Black grapes (*Vitis vinifer* L. *cv*. Seiahe Shiraz) were purchased form Shiraz orchards (Beyzaa, Fars province, southern Iran). Separation of seeds from grapes was done by hand before being used for experimental procedures. The grape seeds were then dried using a cabinet dryer set at 55°C with an air flow rate of 1.5 m s^{-1}. General composition of grape seeds was measured according to the AOAC methods as shown in Table 1. The moisture sorption isotherm measurement of grape seeds was performed according to the static gravimetric method developed in the COST 90 project. The a_w for the applied salts at each temperature were taken from Labuza *et al.* (1985) and Rizvi (2005) as shown in Table 2. The samples were allowed to equilibrate until there was not more than 1% difference in weight (±0.001 g) between three successive measurements. The moisture content of the equilibrated samples was determined by the vacuum oven method at 65°C.

The experimental data obtained corresponding to the water activity and the equilibrium moisture content at the temperatures studied were adjusted to several regression models (Labuza *et al.,* 1985; Rizvi, 2005). The Brunauer-Emmett-Teller model (BET) (Eq. (1)), Guggenheim, Anderson and De Boer model (GAB) (Eq. (2)), Peleg (Eq. (3)), modified Ferro-Fontan (Eq. (4)) and Oswin (Eq. (5)) equations were applied to fit the experimental data. In order to determine the monolayer moisture values of the samples, which is an important parameter in food deteriorations, BET (a_w up to 0.4) and GAB (a_w up to 0.9) models were used:

$$M = \frac{M_0 C a_w}{(1-a_w)(1+Ca_w-a_w)}, \tag{1}$$

$$M = \frac{M_0 CK' a_w}{(1-K'a_w)(1-K'a_w+CK'a_w)}, \tag{2}$$

$$M = K_1 a_w^{n1} + K_2 a_w^{n2}, \tag{3}$$

$$M = \left(K \ln\left(\frac{\gamma}{a_w}\right)\right)^{\frac{-1}{r}}, \tag{4}$$

$$M = C\left(\frac{a_w}{1-a_w}\right)^n, \tag{5}$$

where: C, K', K, K_1, K_2, n, n_1, n_2 and r are model constants, M is the equilibrium moisture content based on dry materials, M_0 is mono layer moisture content and a_w is the water activity. Fitting experimental data to model equations was performed using the 'Solver package' of Excel program (Microsoft Office, 2003).

To evaluate the goodness of the fit of each model, statistical parameters were applied, such as standard error of estimate (*SEE*), coefficient of determination (R^2), residual sum of squares (*RSS*), the mean relative percentage deviation modulus (M_e) and residual plot defined as:

$$RSS = \sum_{i=1}^{n} (M_{i,\exp} - M_{i,pre})^2, \tag{6}$$

$$SEE = \sqrt{\sum_{i=1}^{n} \frac{(M_{i,\exp} - M_{i,pre})^2}{n}}, \tag{7}$$

T a b l e 1. General composition (%) of dried grape seeds used in this research

Moisture content	7.0±0.5
Protein	7.65±0.15
Fat	20.4±0.4
Polysaccharide	63.85±3.55
Ash	1.1±0.2

T a b l e 2. Water activities (a_w) of saturated salt solutions at 35, 45, 55 and 65°C

Salt	35°C	45°C	55°C	65°C	K_1*	K_2*
LiCl	0.108	0.102	0.098	0.093	500.95	3.85
CH_3COOK	0.215	0.197	0.181	0.168	861.39	4.33
NaI	0.347	0.327	0.307	0.290	643.01	3.14
$NaNO_2$	0.628	0.601	0.576	0.556	435.96	1.88
NaCl	0.750	0.750	0.748	0.747	228.92	1.04
KCl	0.821	0.790	0.760	0.740	367.58	1.39

*Constants of temperature-water activity equation.

$$M_e = \frac{100}{n} \sum_{i=1}^{n} \frac{\left| M_{i,\exp} - M_{i,pre} \right|}{M_{i,\exp}}, \tag{8}$$

where: $M_{i,\,\exp}$ and $M_{i,\,pre}$ are experimentally observed and predicted by the model values of the EMC, respectively, and n is the number of data points. The correlation coefficient, R^2, gives a measure of the proportion of variability attributed to the model. The standard error of estimate, *SEE*, gives an indication of the precision of the experiment estimation. The model which has the smallest *RSS*/*SEE* and the highest R^2 was considered to be the best fitted equation (Chowdhury *et al.,* 2006; Foster *et al.,* 2005; Jamali *et al.,* 2006). Another parameter, the mean relative percentage deviation modulus, M_e, is also an important index to select the best fitted equation with a modulus value below 10% indicative of a good fit of practical data on a model equation (Al-Muhtaseb *et al.,* 2004). Plotting residuals against a_w may also be considered to evaluate the closeness of the fit. If the residuals are uniformly scattered around the horizontal value of zero showing no systematic tendency towards a clear pattern, the model fits well (Aviara *et al.,* 2006).

The net isosteric heat of sorption (q_{st}) is defined as the difference between the total heat of sorption in food and the heat of vaporization of water at the system temperature, which indicates the energies for water molecules binding at a particular hydration level (Tsami *et al.,* 1990). For estimating the net iosteric heat of sorption the Clausius-Clapeyron Eq. (9) can be used as follows:

$$\left(\frac{\partial \ln a_w}{\partial (1/T)} \right)_{\Gamma} = -\frac{q_{st}}{\mathrm{R}}, \tag{9}$$

where: q_{st} is the net isosteric heat of sorption at constant moisture content (kJ mol^{-1} H_2O), a_w is water activity, T is the absolute temperature (K), R is the universal gas constant (8.314 J mol^{-1} K), and $\Gamma = n_l/\mathrm{n_A}$ and is proportional to the moisture content in food and that the number of moles of adsorbent, $\mathrm{n_A}$ (if such a thing exists), is constant and proportional to the surface area of the food ($\mathrm{n_l}$ is number of moles of adsorbate (water)) (Rizvi, 2005). Through this equation, q_{st} may be established by plotting ln(a_w) at a specific moisture content (estimated by the Ferro-Fontan model) against $1/T$ and measuring the slope (Roman *et al.,* 1982).

The empirical exponential relationship between the isosteric heat of sorption and moisture content has been proposed by Tsami *et al*. (1990):

$$q_{st} = q_0 \exp(-M/M_0), \tag{10}$$

where: q_0 (kJ mol^{-1}) is the net isosteric heat of sorption of the first molecule of water in the food, and M_0 is a characteristic moisture content of the food material on dry basis. For estimating the constants q_0 and M_0, Eq. (10) was fitted to the values of q_{st}, obtained by applying Eq. (9) to the experimental isotherms (Tsami *et al*., 1990).

RESULTS AND DISCUSSION

The experimental EMC data for grape seed at 35, 45, 55, 65°C over the a_w range of 0.108-0.821 are given in Table 3. These values at each a_w reperesent the mean value of three replication. From this Table it can be seen that at a constant temperature the EMC of samples increased with increase in a_w. The experimental data ranged between 0.211 and 0.479 (g water g^{-1} dry matter) for a_w range between 0.108 and 0.821 at 35°C. Indeed, grape seeds have 21% oil and compared with non-oily seeds have lower EMC at a given relative humidity. It has been reported that the presence of fat and oil in agricultural products contributes to the lowering of their sorption capacity. This behaviour has been also seen in melon seed (Aviara *et al*., 2002) and peanut (Bianco, 2001).

The experimental data were used to determine the sorption isotherms at different temperatures as shown in Fig. 1. These isotherms reflect the common sigmoidal shape pattern,

Table 3. Equilibrium moisture content (EMC, g water g^{-1} dry matter) at various water activity (a_w) of grape seed at different temperatures

Temperature (°C)	Water activity (a_w)	Equilibrium moisture content (g water g^{-1} dry matter)
35	0.108	0.211±0.005
	0.215	0.265±0.008
	0.347	0.288±0.007
	0.628	0.355±0.009
	0.750	0.403±0.005
	0.821	0.479±0.006
45	0.102	0.210±0.005
	0.197	0.255±0.010
	0.327	0.274±0.008
	0.601	0.340±0.005
	0.750	0.399±0.009
	0.790	0.470±0.009
55	0.098	0.199±0.006
	0.181	0.236±0.006
	0.307	0.262±0.003
	0.576	0.332±0.009
	0.748	0.392±0.006
	0.760	0.450±0.007
65	0.093	0.186±0.007
	0.168	0.226±0.006
	0.290	0.252±0.008
	0.556	0.327±0.009
	0.747	0.377±0.006
	0.740	0.446±0.008

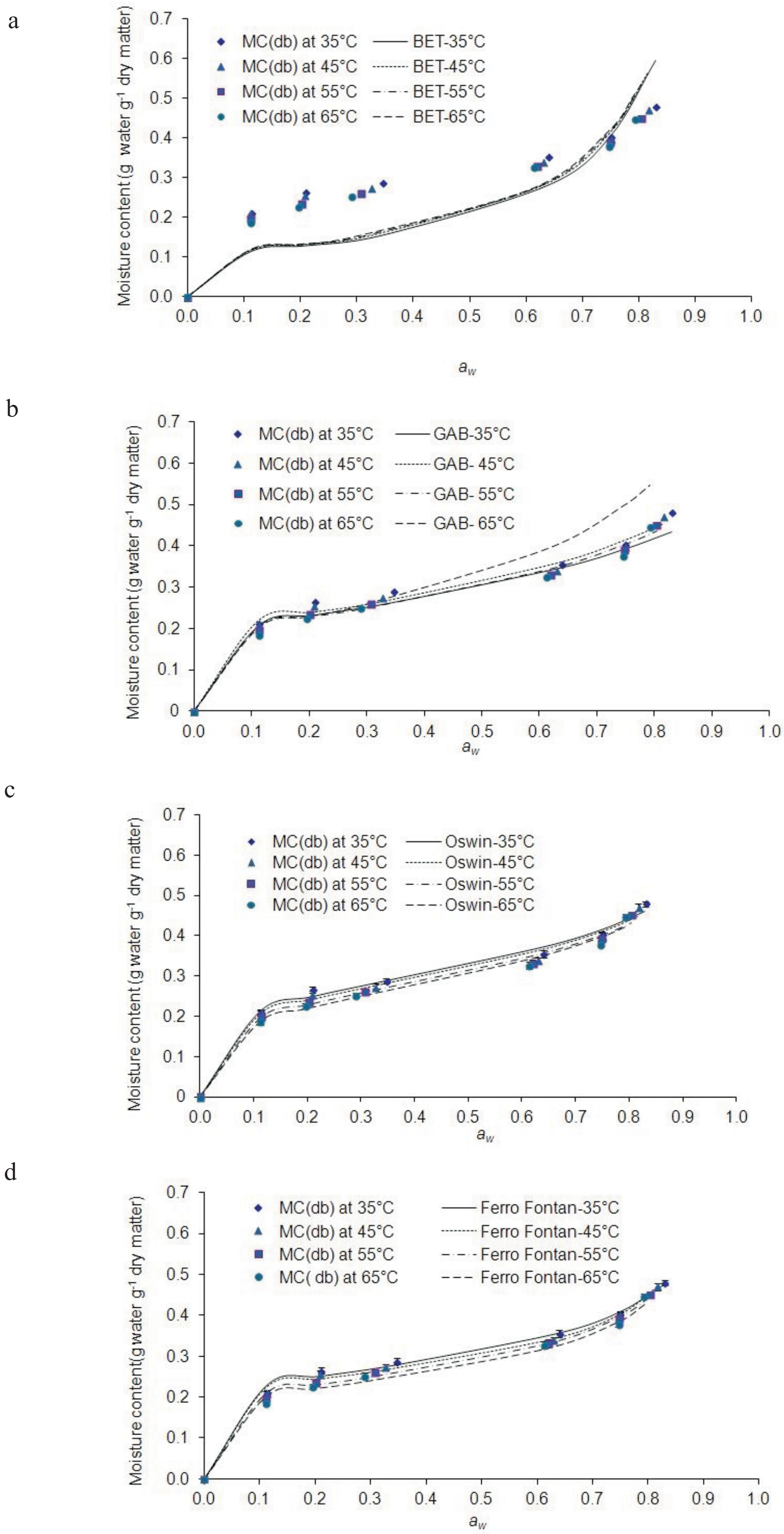

Fig. 1. Moisture sorption isotherm of grape seed at different temperatures. The symbols are experimental data and the lines are from the equations obtained by fitting the experimental data to: a – BET, b – GAB, c – Oswin, d – Ferro-Fontan, and e – Peleg equations. Each bar is ±SD.

e

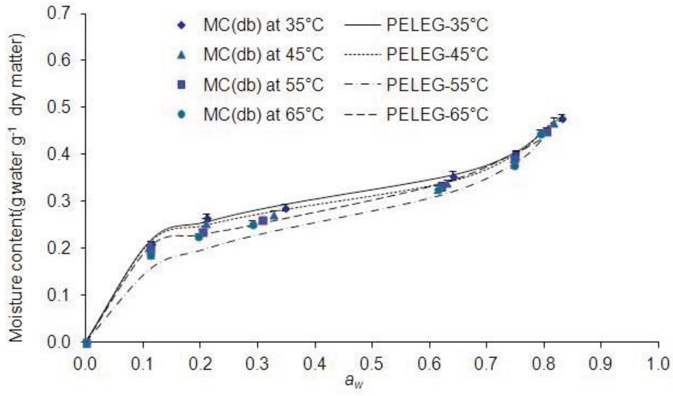

Fig. 1. Continuation.

T a b l e 4. Estimated BET, GAB, Peleg, Ferro-Fontan, Oswin constants and monolayer moisture levels for grape seed at different temperatures

Temperature (°C)	BET*		GAB			Peleg				Ferro-Fontan			Oswin	
	M_0 (%)	C	M_0 (%)	K	C	K_1	K_2	n_1	n_2	r	K	γ	n	C
35	19.65	402.65	21.12	134.9	0.62	0.389	0.604	0.270	9.20	3.85	0.278	0.941	0.21	0.329
45	19.59	140.30	20.94	402.7	0.66	0.367	0.713	0.248	8.74	4.20	0.267	0.896	0.21	0.323
55	19.08	88.12	20.48	160.6	0.66	0.366	0.763	0.390	9.11	3.69	0.257	0.915	0.22	0.313
65	18.85	60.40	20.26	94.6	0.79	0.312	0.316	3.770	0.20	3.76	0.246	0.899	0.23	0.306

*Up to a_w 0.4.

typical for isotherms from seeds. In the first segment (with low RH) of the S-shaped sorption isotherm curves, grape seeds absorbed relatively lower amounts of moisture, with larger amounts in the second segment (at higher RH). Similar behaviour has been reported for other foods (Togrul and Arslan, 2007). According to Van den Berg and Bruin (1981), a sigmoid moisture sorption isotherm can be divided into three parts: ranges I (a_w = 0-0.22), II (a_w = 0.22-0.73) and III (a_w = 0.73-1.0). At higher a_w (ranges II and III) water absorption would take place at newly created pores of the already swollen structure and so it would be markedly influenced by the stability of the micro-porous structures. The data also showed that at a constant a_w the EMC of grape seeds decreased with increasing temperature. This trend may be due to a reduction in the total number of active sites for water binding as a result of physical and/or chemical changes in the product induced by temperature. According to Mulet *et al*. (2002), at increased temperatures the attractive forces between molecules decreased due to an increase in kinetic energy of water molecules, thus decreasing the EMC. The effect of temperature, especially temperatures higher than room temperature, on the moisture sorption isotherm of foods is of great importance as they are exposed to a range of temperatures during storage and processing. However, there was no marked difference in the EMC of grape seeds with increasing temperature.

The results of fitting the sorption equations to the experimental data are shown in Table 4. In order to confirm the equations ability for predicting the equilibrium moisture content, *RSS*, *ESS*, R^2 and M_e (%) values corresponding to each sorption isotherm as a function of temperature are given in Table 5. As the applicability of the BET equation is generally restricted to a_w values below 0.45, it cannot be considered as a suitable model in fitting the data. Among the three other models (Peleg, Oswin and GAB) which can be applied throughout the entire range of water activity, the smallest *RSS* ($0.01\text{-}7\times10^{-4}$), *SEE* (0.07-1.3) and higher values of R^2 (98.3-99.3%) at all the temperatures studied were obtained for the Ferro-Fontan and PELEG equations (except for 55°C), respectively. Figure 1 also confirmed that the Ferro-Fontan equation gives the closest fit to the experimental data at all the temperatures studied, followed by the Peleg model (except for the isotherm at 55°C). The usefulness of models may also be assessed by considering the mean relative percentage deviation modulus (M_e) which should be below 10% to be an indication of a good fit. The Ferro-Fontan model gives the lowest Me values ranging from 2.4 to 4.3% (with average value of 3.35%), compared with 2.8 to 3.6% (with average value of 3.2%), 3.5 to 11.3% (with average value of 7.4%) and 0.98 to 9.81% (with average value of 5.39%) for the Oswin, GAB and Peleg models, respectively. However, the BET model, giving M_e

T a b l e 5. Coefficient of determination R^2, standard error of estimate (*SEE*), residual sum of square (*RSS*), mean relative percentage deviation moduli (M_e) and the nature of residual plot obtained for several models applied to experimental sorption data of sample at different temperatures

Model	*RSS*				*SEE*				R^2				M_e				Residual plot			
	Temperature (°C)																			
	35	45	55	65	35	45	55	65	35	45	55	65	35	45	55	65	35	45	55	65
BET*	0.0005	0.0003	0.0001	0.0001	0.012	0.009	0.005	0.004	0.989	0.993	0.998	0.999	0.035	0.026	0.016	0.012	S	S	S	S
GAB	0.004	0.001	0.0007	0.028	0.026	0.014	0.011	0.069	0.974	0.974	0.984	0.976	0.064	0.044	0.035	0.113	R	R	R	S
Peleg	0.0001	0.00006	0.004	0.001	0.004	0.003	0.0280	0.014	0.997	0.998	0.999	0.989	0.013	0.009	0.098	0.040	R	R	S	R
Ferro-Fontan	0.0007	0.0004	0.0003	0.001	0.011	0.008	0.007	0.013	0.986	0.991	0.993	0.983	0.033	0.0258	0.024	0.043	R	R	R	R
Oswin	0.001	0.001	0.0008	0.001	0.013	0.015	0.011	0.014	0.978	0.971	0.982	0.974	0.032	0.036	0.028	0.034	R	R	R	R

*Up to a_w 0.4.

values of 1.2% to 3.5% (with average value of 2.35%), was only adequate for reperesenting the sorption isotherms of grape seeds with a_w from 0 to 0.4 (and not the whole range). Figure 2 shows the residuals (the difference between the experimental and predicted values of the EMC) versus a_w for the Ferro-Fontan, Peleg, Oswin and BET models at 55°C. Since among the models evaluated only the Ferro-Fontan model had a random distribution at all the temperatures studied, this model can be used for the evaluation of water sorption behaviour of grape seeds. However, for BET, GAB, Oswin and Peleg (only at 55°C) the residual plots show a systematic pattern. Similar patterns are seen for other temperatures. Overall, the examination of the results in Tables 4, 5 and Figs 1-2 indicated that the Ferro-Fontan model describes best the experimental data for grape seeds throughout the entire range of a_w and temperatures.

The monolayer water content (M_0) corresponds to the moisture level at which the rate of chemical reactions in food is very low since the water molecules are bound strongly to the surface of the monolayer (Arslan and Togrul, 2006). This critical moisture content can be determined by the BET and GAB model and is presented in Table 4 at different temperatures. In the temperature range of 35-65°C, the values of the monolayer moisture content of grape seeds varied from 18.85 to 19.65% (dry basis) using the BET model, and from 20.26 to 21.12% (dry basis) when the GAB model was used. As seen, the values of monolayer moisture content calculated by the BET model were lower than those calculated by the GAB model since the BET model is related to the water sorption in the first layer and the GAB model in the multi-layer region. This was in accordance with the research of Timmermann *et al*. (2001). It is also seen that M_0 decreases with increasing temperature, irrespective of the equation used. This may be attributed to the reduction of hydrogen bonding degree of food polymers with increasing temperature, and so decreasing the availability of active sites for water binding and monolayer moisture content (Mulet *et al*., 2002). Similar results have been found for the moisture sorption of African star apple and mango (Falade and Awroh, 2004) and fig (Ansari *et al*., 2011). There is another parameter in the BET model, namely the sorption energy constant (C), which can be used for the classification of sorption isotherms according to the Brunauer classification. As the values of C for the grape seeds at all the temperatures studied are higher than 2, their sorption isotherms can be classified as Type II. This behaviour has also been observed in other fruits such as apple (Falade and Awroh, 2004), grape (Kaymak-Ertekin and Gedik, 2004) and spray-dried tomato pulp (Goula, 2008).

Isosteric heat of sorption (also called latent heat of vaporization) in foodstuffs is of practical interest in drying operations, handling, storage and processing. The most common method for measurement of the differential heat of sorption is the application of the Clausius-Clapeyron equation (Tolba, 2004). Sorption isotherms estimated using the

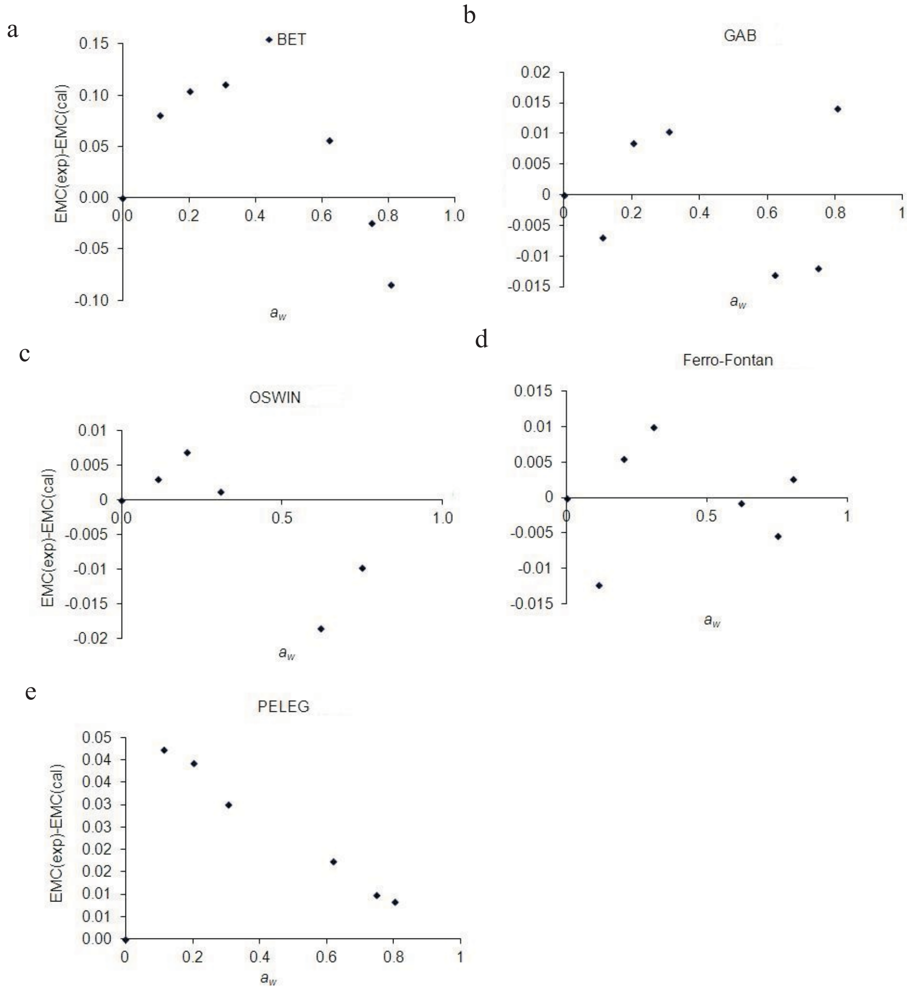

Fig. 2. Residual plots of: a – BET, b – GAB, c – Oswin, d – Ferro-Fontan and e – Peleg models for the adsorption data of grape seed at 55°C.

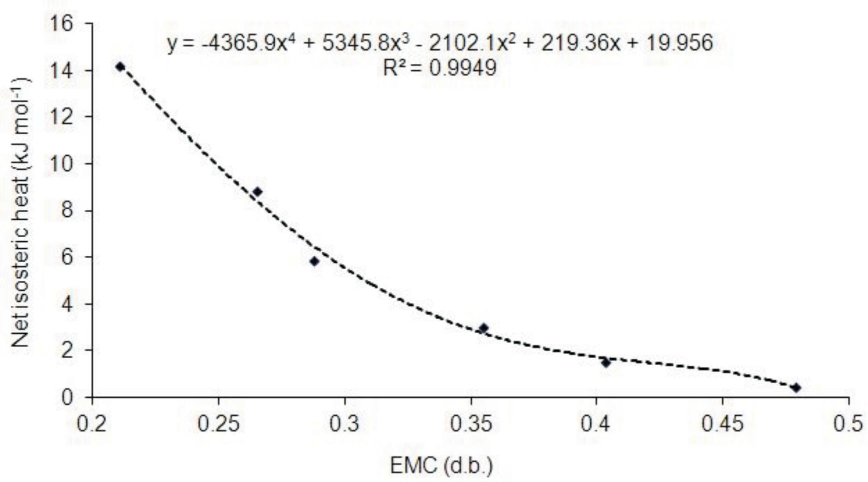

Fig. 3. Isosteric heat of sorption of grape seed as a function of EMC obtained using polynomial function.

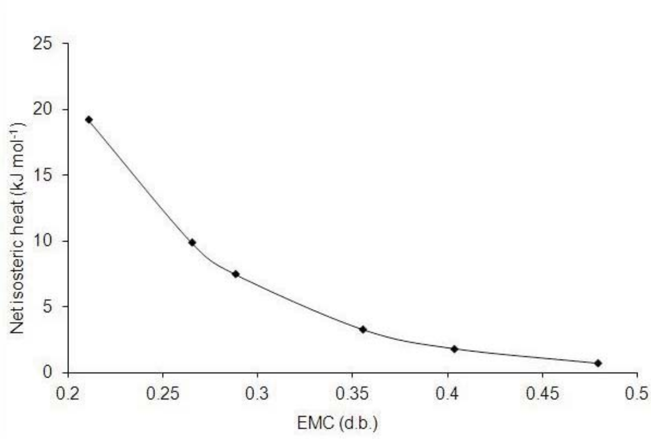

Fig. 4. Isosteric heat of sorption of grape seed as a function of EMC obtained using linear regression between ln(q) and EMC.

Ferro-Fontan model for each temperature (Figs 1, 2 and Table 5) were used to interpolate water activity values at different experimental temperatures for a constant moisture content. Figure 3 indicates the effect of moisture content on isosteric heat of sorption of grape seeds. The curve shows that the heat of sorption decreased with increasing moisture content, initially rapidly up to 0.35 g moisture 100 g^{-1} dry solids, and later slowly, as observed in many other food systems (Ansari *et al*., 2011; Tolba, 2004).

A polynomial function (with R^2 of 99.49%) was used to describe the relationship between isosteric heat of sorption and EMC (Fig. 3).

$$q_{st} = -4365.9M^4 + 5345.8M^3 - 2102.1M^2 + 219.36M + 19.956 \text{ (kJ mol}^{-1}\text{)}, \quad (11)$$

where: M is the moisture content (d.b. %) and q_{st} is net isosteric heat of sorption (kJ mol^{-1}). This type of empirical expression was similar to the correlations obtained in the literature for some other products such as tow mint (Ethmane *et al*., 2008) and dried date powder (Hassan, 2000).

The decrease in the isosteric heat with increase in moisture content may be attributed to the fact that sorption initially occurs on the most active sites (such as hydrophilic polar groups). With increase in the moisture content, as these sites become occupied, sorption occurs on the less active sites (such as hydrophobic hydration sites) resulting in lower heats of sorption (Jayendra *et al*., 2005).

Using linear regression between ln(q) and M, one can estimate the exponential function between net isosteric and moisture content (Eq. (12)):

$$q = 251.313 \exp(-12.19\, M) \, . \quad (12)$$

Linear regression with $R^2 = 0.9867$ and estimated q_0 value of 251.313 kJ mol^{-1}, and M_0 of 0.0819 (dry basis) is shown in Fig. 4.

CONCLUSIONS

1. The moisture sorption isotherm of grape seeds follows a sigmoid shape typical for most foods. Temperature affected the sorption behaviour, so that the EMC decreased with increasing temperature at constant a_w.

2. The semi-empirical three-parameter Ferro-Fontan model (within the whole temperature range studied) and the empirical four-parameter Peleg equation (at 35, 45 and 65°C) were found to be the best appropriate equations for experimental sorption data of grape seeds, respectively.

3. The net isosteric heat of sorption was found to be a polynomial function of equilibrium moisture content which increased with decreasing moisture content.

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