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Modelling of the Sorption Isotherms and Determination of the Isosteric Heat of Split Pistachios, Pistachio Kernels and Shells

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Abstract

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We aimed to measure the equilibrium moisture content of the Ohadi variety of pistachio shells, pistachio kernels and split pistachios using gravimetric methods at 25, 40, 55, and 70°C and saturated salt solutions with water activities ranging from 0.08 to 0.97. Then, 11 mathematical models were used to select the best model for data description. The GAB model, which had the maximum value for the correlation coefficient (R^2) and minimum values for the chi-squared test (χ^2) and root mean square error (RMSE), was identified as the best model for split pistachios and pistachio kernels. The Caurie model, meanwhile, was identified as the best model for pistachio shells. In this test, there was no significant hysteresis between the desorption and adsorption curves. Furthermore, the isosteric heat of adsorption and desorption were also determined using thermodynamic equations (e.g., the Clausius-Clapeyron equation).

Keywords: drying; gravimetric method; isosteric heat; water activity

Pistachios (*Pistacia vera* L.) are cultivated in Mediterranean, American and Middle Eastern countries. Iran annually produces over 300 000 tons of pistachios, which is 47% of total global production (FAO 2008). In Iran, the pistachio accounts for about 40% of agricultural exports and 8–11% of non-oil exports (Tavakolipour 2011).

Due to the seasonal nature of pistachio harvesting, vast quantities of pistachio may be stored for months after harvesting. During the storage period, if the ambient moisture is not maintained carefully in cold storage, the moisture-loving characteristic of pistachios leads to significant changes in the moisture content of the product (Kashani Nejad *et al.* 2002). Therefore, the quality of the stored product depends on the amount of moisture, moisture migration and moisture adsorption through the food material during storage (Mujumdar & Devahastin 2000). Consequently, for proper storage, it is necessary to determine the sorption isotherms of the product. A sorption isotherm is the relationship between equilibrium moisture content and moisture content under constant temperature and pressure (Garbalińska *et al.* 2017), and the moisture content difference between adsorption and desorption values is known as hysteresis because of the differing abilities of samples to adsorb and desorb water (Tejada-Ortigoza *et al.* 2017). Moreover, sorption isotherms are very important for modelling the drying process, equipment design and optimisation, forecasting the shelf life stability, computation of moisture changes during storage and the selection of packaging material (Tunç & Duman 2007; Farahnaky *et al.* 2016).

Some reports of sorption isotherm research in the pistachio are available in the literature. Karatas and Battalbey *et al.* (1991) measured the adsorption and desorption isotherms of the Gaziantep pistachio at 20°C. Ayranci and Dalgic (1992) determined the sorption isotherms of different varieties of raw pistachio and pistachio protein isolates and Yan-

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niotis and Zaramboutis (1996) measured the sorption isotherms of pistachios at 15, 25, and 45°C.

Maskan and Gogus? (1997) determined the adsorption and desorption properties of pistachios at 10, 20, and 30°C. They demonstrated that parameters such as monolayer moisture content and temperature affected the water adsorption (Maskan & Karatas 1997). Using standard gravimetric methods, Yazdani *et al.* (2006) measured the sorption isotherms of raw, shelled and powdered Amiri pistachios. The results indicated that the Smith model was the best model for describing the behaviour of this pistachio variety. They also measured the isosteric heat of sorption using the Clausius-Clapeyron equation (Yazdani *et al.* 2006). Hayoglu and Gamli (2007) conducted similar studies on pistachio paste. They considered the Peleg model as an appropriate model, and they also found that, with increased isosteric heat, the absolute moisture content of the sample is reduced (Hayoglu & Gamli 2007). Tavakolipour and Kalbasi (2008) determined the sorption isotherms of the Kerman pistachio to be 15, 25, 35, and 40°C. They also found that the Smith model was the best model for describing the sorption properties of the pistachio at these temperatures and reported significant hysteresis between the adsorption and desorption isotherms (Tavakolipour & Kalbasi 2008).

The net isosteric heat of adsorption is useful for estimating the energy required for the drying process and for obtaining important information about the state of water in the food. The net isosteric heat of sorption is referred to as the latent heat of water vaporisation, representing the bound water of the food. The energy required for vaporisation gradually increases with a decrease in the moisture content (Benado & Rizvi 1985).

In this study, we aimed to determine the sorption isotherms and hysteresis of split pistachios, pistachio kernels and pistachio shells of the Ohadi variety, to identify the best mathematical model for describing the adsorption and desorption behaviours of water and to determine the isosteric heat of this pistachio variety.

MATERIAL AND METHODS

Experimental method. The fresh *Ohadi* pistachios were selected from the Kerman region of Iran. The samples were stored at room temperature prior to tests. The initial moisture content of the samples was 36.17 g/100 g (Wb). Pistachio composition was determined to include the following substances in the following amounts: lipid 33.15 ± 0.002, carbohydrate 1.14 ± 0.02, protein 1.88 ± 0.002, ash 0.94 ± 0.17, and fibre 26.72 ± 0.1 g/100 g (AOAC 2000).

Table 1. The water activities of salts at the tested temperatures

Salt	Experimental temperature (°C)			
	25	40	55	70
KOH	0.08	0.063	0.0507	0.0416
CH_3COOK	0.22	0.21	0.188	0.17
$MgCl_2$	0.33	0.31	0.299	0.278
$Mg(NO_3)_2$	0.53	0.5	0.439	0.395
$SrCl_2$	0.70	0.71	0.719	0.728
KCl	0.84	0.821	0.807	0.795
K_2SO_4	0.97	0.96	0.957	0.95

The equilibrium moisture contents of the split pistachios, pistachio kernels and pistachio shells were determined using the standard gravimetric method. Saturated solutions of KOH, CH_3COOK, $MgCl_2$, $Mg(NO_3)_2$, $SrCl_2$, KCl, and K_2SO_4 were used to achieve a constant relative moisture content at 25, 40, 55, and 70°C. Table 1 shows the water activities of the salt solutions at the tested temperatures (AOAC 1978).

The samples were prepared for adsorption and desorption isotherm measurement in the following way: For adsorption isotherm measurement, the samples were dried in the air oven at 80°C for 48 h and for desorption isotherm measurement, the samples were placed in a jar filled with pure water up to the mark before transfer to jars for isotherm measurement.

Experimental equipment included seven jars, which were filled with the saturated salt solution to 1/3 of their capacity. The samples were placed in jars without contacting the saturated solution, then they were placed in an incubator at constant temperature until they reached a constant weight (± 0.001 g) (Peng *et al.* 2007), a period of approximately two weeks. Then, the samples were extracted from the jar and weighed. They were dried in an oven at a constant temperature of 105°C for 8 h to determine their dry weight. The experiments were carried out in three replications.

Mathematical modelling. Twelve models were used to predict the sorption isotherms (Table 2). The experimental data were used to select the appropriate models through non-linear regression with Matlab R2016a.The criteria for selecting the best model included correlation coefficient (Equation 1), root mean square error (Equation 2) and the chi-squared test (Equation 3):

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Table 2. Models fitted to the experimental data of pistachios

Names of model	Model
GAB (van Den Berg 1981)	$w = \frac{w_m CKa_w}{(1 - Ka_w)(1 - Ka_w + CKa_w)}$
BET (Brunauer *et al.* 1940)	$w = \frac{w_m Ca_w}{(1 - a_w)(1 - a_w + Ca_w)}$
Halsey (Halsey 1948)	$w = \frac{-A}{(T \ln a_w)}$
Henderson (Henderson 1952)	$w = A\,[-\ln(1 - a_w)]^B$
Oswin (Oswin 1946)	$w = A\left[\frac{a_w}{(1 - a_w)}\right]^B$
Smith (Smith 1947)	$w = A + B\log(1 - a_w)$
Peleg (Peleg 1993)	$w = K_1 a_w^{n_1} + K_2 a_w^{n_2}$
Caurie (Caurie 1970)	$w = e^{(K + Ca_w)}$
Anderson (Anderson 1946)	$w = \frac{ABCa_w}{1 + (B - 2)Ca_w + (1 - B)C^2 a_w}$
Kuhn (Kuhn 1964)	$w = \left(\frac{A}{\ln a_w}\right) + B$
Chung and Pfost (Chung & Pfost 1967)	$w = A\ln\left[\frac{B}{\ln a_w}\right]$

$$R^2 = \frac{\sum_{i=1}^{N}(W_{pre.i} - W_{ave})^2}{\sum_{i=1}^{N}(W_{exp.i} - W_{ave})^2} \quad (1)$$

$$RMSE = \left[\frac{1}{N}\sum_{i=1}^{N}(W_{exp.i} - W_{pre.i})^2\right]^{½} \quad (2)$$

$$\chi^2 = \frac{\sum_{i=1}^{N}(W_{exp.i} - W_{pre.i})^2}{N - n} \quad (3)$$

where: W_{exp}, W_{pre} – moisture contents of the experimental and predictive models; N, n – number of observations and model constants (Ghodake *et al.* 2007)

Isosteric heat and adsorption entropy. When water is removed from food, heat is absorbed, despite the increased osmotic pressure and water activity gradient. Sorption isosteric heat-differential-enthalpy is an indicator of the state of the water retained in the solid materials. Net sorption isosteric heat (ΔH_S) is the difference between total heat absorbed by food (ΔH_d) and water vaporisation heat (ΔH_{vap}) associated with the sorption process, which is calculated from experimental data using the Clausius-Clapeyron equation (Equation 4):

$$\left[\frac{d(\ln a_w)}{d(1/T)}\right]_M = -\frac{\Delta H_S}{R} \quad (4)$$

Where the isosteric heat of sorption is defined as molecules absorbed at a particular moisture content (Equation 5):

$$\Delta H_S = \Delta H_d - \Delta H_{vap} \quad (5)$$

By integrating and entering the boundary conditions (Equations 6 and 7):

$$\int_{a_{w1}}^{a_{w2}} d(\ln a_w) = -\frac{\Delta H_S}{R}\int_{T_1}^{T_2}(d(1/T) \quad (6)$$

$$\ln\left(\frac{a_{w2}}{a_{w1}}\right) = \frac{\Delta H_S}{R}\left[\frac{1}{T_1} - \frac{1}{T_2}\right] \quad (7)$$

where: a_{w1}, a_{w2} – water activity of T_1 and T_2; R – gas constant (8.314 kJ/mol·K) (Santanu-Basu *et al.* 2006)

The isosteric heat of sorption is an important parameter during drying. Its value in drying equilibrium should be considered at the end of the drying process, since it has a magnitude equal to the latent heat of vaporisation (Talla *et al.* 2005).

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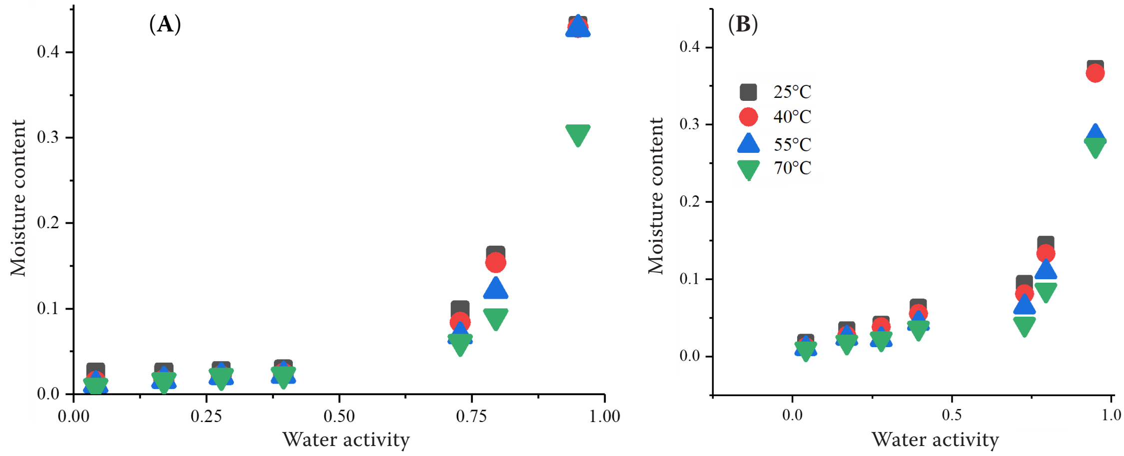

Figure 1. Adsorption (**A**) and desorption (**B**) isotherms of pistachios at selected temperatures

The differential enthalpies (ΔS_d) were calculated from isothermal data at different temperatures. In this way, the relationship between isosteric heat (ΔH_d) and sorption differential enthalpy (ΔS_d) is as follows:

$$d(\ln a_w)_x = (\Delta H_d/RT) - (\Delta S_d/R) \tag{8}$$

By plotting ln a_w versus the inverse of temperature, the particular moisture content, W and ΔH_d were obtained from the slope, and ΔS_d was obtained from the intercept. Use of these equations at different moisture contents demonstrates the dependence of ΔH_d and ΔS_d on moisture content (Rouquerol *et al.* 2014).

RESULTS AND DISCUSSION

Moisture sorption isotherms. Figure 1 shows the adsorption and desorption isotherms of the samples at experimental temperatures. These isotherms are type II (Perry *et al.* 1984).

Analysis of variance (ANOVA) indicated that the effect of temperature on moisture content was significant ($P \geq 0.5$). As can be seen in Figure 1, with increased temperature, the moisture content of the samples decreased. Nikoly (2000) demonstrated that with increasing temperature, some water molecules are activated, which increases their energy level leading to them breaking apart. This means that active sites for water adsorption are reduced due to the physical and chemical changes of the product during heating.

The results also indicated that at a constant temperature, with increased water activity, the moisture content increases. Similar results were reported by Basunia and Abe (2001) for rice, by Ghodake *et al.* (2007) for green, black and wet tea and by Lahsasni *et al.* (2003) for pears.

Figure 2 shows the adsorption and desorption isotherms of the pistachio at 55°C. The same behaviour was observed at 25, 40, and 70°C. ANOVA and data variance analysis using the least significant difference indicated that the hysteresis effect was not significant ($P \geq 0.5$). Similar results were reported by Menkov *et al.* (2000) for the pea and by Al-Muhtaseb *et al.* (2004) for starch.

Mathematical modelling of experimental data. Estimated parameters of the respective models are given in Tables 3–5. The criteria for selecting model to describing the behaviour of the samples were maximum correlation coefficient (R^2) and the minimum RMSE and χ^2. Therefore, the GAB model was considered as the best model for split pistachios and pistachio kernels, and the Caurie model was identified as an appropriate model for pistachio shells.

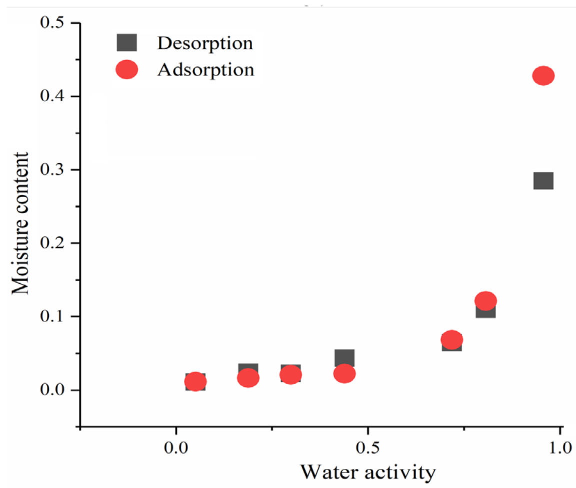

Figure 2. Adsorption and desorption isotherms of pistachios at 55°C

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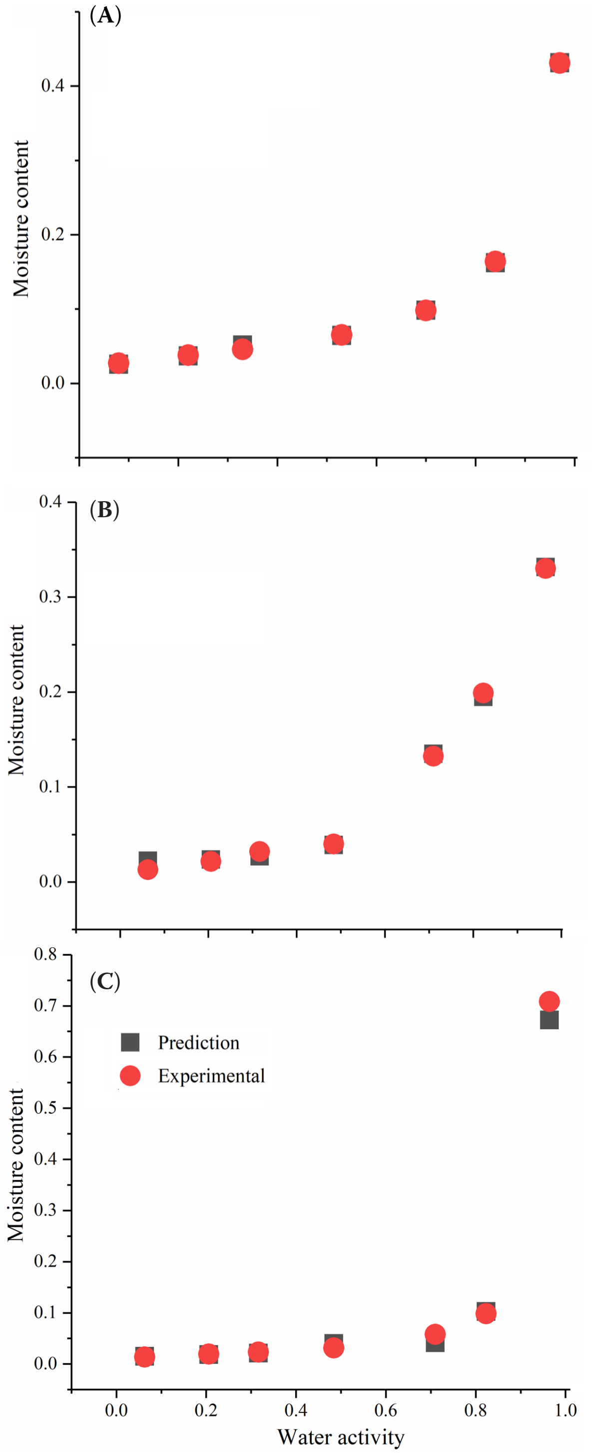

Figure 3. Comparison of predicted and measured data for split pistachios (**A**), shells (**B**), and kernels (**C**)

The equilibrium moisture content of the split pistachios, pistachio kernels and pistachio shells determined in the laboratory during adsorption was compared with the values predicted by the best model (Figure 3). A similar graph was also plotted for desorption (data not shown).

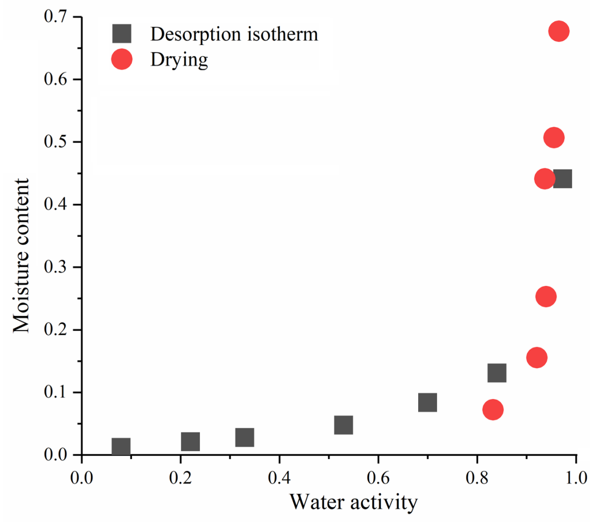

Figure 4. Comparison of the desorption isotherm and pistachio kernel drying at 70°C

Some researchers have selected other mathematical models for describing the desorption and adsorption of the pistachio (Maskan & Gogus 1997; Yazdanip *et al.* 2006; Hayoglu & Gamli 2007; Tavakolipour & Kalbasi 2008), because most existing models are not defined and valid for the high levels of water activity in these nuts. Since the moisture contents of the pistachio in at these levels of water activity are also high, this leads to high error and low correlation coefficients, which, in turn, results in mistakes in choosing the best model.

To evaluate the changes in water activity during pistachio drying, the samples were placed in a cabinet dryer, and the moisture content and water activity of the samples during the drying process were meas-

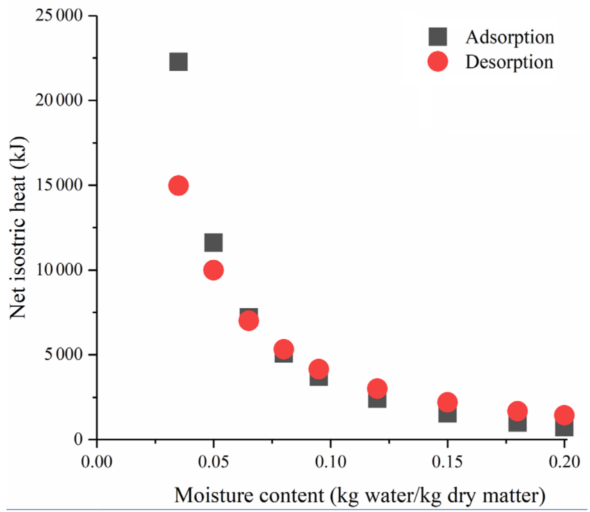

Figure 5. Adsorption and desorption isosteric heat at different temperatures

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Table 3. Performance of selected models for sorption isotherms

Model	Constants	Adsorption (°C)				Desorption (°C)			
		25	40	55	70	25	40	55	70
Split pistachios									
GAB	w_m	0.034	0.029	0.024	0.016	0.033	0.031	0.024	0.016
	C	14.822	12.807	28.113	50.802	33.228	12.263	28.673	170.677
	K	0.935	0.956	0.959	0.991	0.952	0.962	0.982	0.992
	RMSE	0.001	0.0001	0.0001	0.0001	0.00004	0.0002	0.0003	0.0002
	R^2	0.997	0.999	0.998	0.994	0.999	0.999	0.999	0.999
Pistachio kernels									
GAB	w_m	0.034	0.007	0.016	0.015	0.049	0.048	0.025	0.023
	C	3.871	0.363	77.146	84.572	33.228	12.263	28.673	170.677
	K	0.909	1.597	0.997	1.007	0.671	0.680	0.922	0.776
	RMSE	0.000	0.006	0.0002	0.0005	0.004	0.004	0.001	0.0004
	R^2	0.998	0.987	0.998	0.997	0.992	0.985	0.997	0.995
Pistachio shells									
Caurie	C	7.299	7.918	8.490	8.8038	3.576	3.594	3.916	4.975
	K	–6.938	–8.569	–6.167	–7.4375	–3.658	–4.572	–5.052	–6.039
	RMSE	0.003	0.002	0.001	0.0009	0.001	0.0001	0.0001	0.0008
	R^2	0.992	0.997	0.997	0.9974	0.995	0.999	0.999	0.996

ured and compared with values obtained from the sorption isotherms. The results indicated that the water activity of the samples was over 0.8, even with moisture content of less than 0.06, and none of the existing models were predictive of the behaviour of the samples during the drying process (Figure 4).

Isosteric heat and sorption entropy. The sorption isosteric heat of the pistachios was obtained by applying the Clausius-Clapeyron equation to the experimental data. The results indicated that the lower the moisture content of the sample, the more sorption isosteric heat was required (Figure 5). Since in the early stages of adsorption, many polar active sites exist on the product surface, and as water molecules are attached to these sites as a monolayer, the energy required for removing water is very high. However, with increased moisture content the affinity of the molecules for the sample is reduced and the sorption isosteric heat is decreased (Moreira *et al.* 2008).

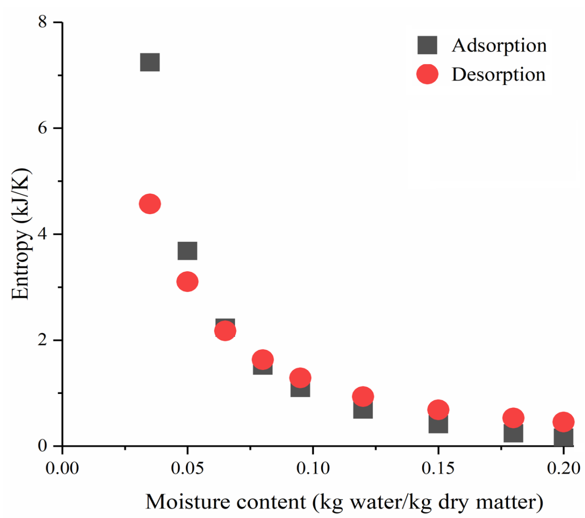

Figure 6. Sorption and desorption isosteric heat at different temperatures

Furthermore, comparison of the heat required for desorption and adsorption at different levels of moisture indicates that the desorption isosteric heat is higher than the adsorption isosteric heat.

Entropy as a function of the moisture content (Figure 6) suggests that, similar to sorption isosteric heat, entropy is also strongly dependent on the moisture content.

Similar trends with respect to isosteric heat and entropy were also observed in pistachio kernels and pistachio shells.

CONCLUSIONS

In this study, the sorption isotherms of split pistachios, pistachio kernels and pistachio shells of the

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Ohadi variety were determined at four temperatures. The results indicated that temperature has an effect on adsorption isotherms, and that at fixed water activity, moisture content decreases with increasing temperature. At fixed moisture content, the water activity increased with increasing temperature.

Mathematical modelling of the experimental data also showed that the GAB model was the best model for split pistachios and pistachio kernels, and that the Caurie model was the best model for describing the adsorption and desorption behaviour of pistachio shells. The isosteric heat and entropy of the adsorption and desorption was also reduced with increased moisture.

Comparison of the data from the drying process and from sorption isotherms of the samples showed that the water activity was over 0.8 even with moisture contents of less than 0.06 and that none of the existing models were able to describe water desorption behaviour during the drying process.

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