Bulgarian Journal of Agricultural Science, 20 (No 3) 2014, 515-522
Agricultural Academy

SORPTION ISOTHERMS AND NET ISOSTERIC HEAT OF SORPTION FOR PLUM OSMOTICALLY PRE-TREATED WITH TREHALOSE AND SUCROSE SOLUTIONS

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Abstract

AKTAS, T., P. ULGER, F. DAGLIOGLU and F. H. SAHIN, 2014. Sorption isotherms and net isosteric heat of sorption for plum osmotically pre-treated with trehalose and sucrose solutions. *Bulg. J. Agric. Sci.*, 20: 515-522

Net isosteric heat of sorption can be used to determine the energy requirements of drying processes. Trehalose pre-treatments have better effects on some quality properties such as colour, shrinkage and cell reconstruction of dried agricultural materials. In this study, changing of moisture sorption isotherms and net isosteric heat of sorption for plum were determined at 30, 40, 50, 60°C. Sorption isotherms were determined using standard static gravimetric method. The net isosteric heat was determined by the application of the Clausius-Clapeyron equation to obtained sorption isotherms. Plum samples were pre-treated osmotically with trehalose and sucrose solutions with 20 and 50 Brix concentrations.

Key words: plum, sorption isotherms, net isosteric heat, osmotic treatment

Abbreviations: M - Equilibrium moisture content (Decimal, d.b.); N - number of experimental data; n - number of constant in models; RMSE - Root mean square error; EF - modelling efficiency; M_0 - mono-layer moisture content (d.b.); a_w - water activity; c, k, n - empirical parameters; C, K - empirical GAB parameters; q_{st} - net isosteric heat of sorption (kJmol^{-1}); Q_{st} - isosteric heat of sorption (kJmol^{-1}); R - universal gas constant (kJmol^{-1}K^{-1}); ΔH_{vap} - the heat of vaporisation (kJmol^{-1} water); T - absolute temperature (K)

Subscripts: pre – predicted; exp – experimental

Introduction

Plum (*Prunus domestica*) is the most numerous and diverse group of fruit tree species, but the extent of fundamental investigations concerning the prunes production is not appropriate to its importance (Živkovic et al., 2011). Preservation is necessary to give an added value to this agricultural product. Studies and developments of new preservation methods are continuously carried out in order to improve the quality of the final product. Pre-treatment of osmotic sugar solutions is a commonly used application in processing of fruit to improve the final product quality before air dying (Muntada et al., 1998; Nieto et al., 2001).

Moisture sorption isotherms describe the relationship between the equilibrium moisture content and the water activity at constant temperature and pressure. For food materials these isotherms give information about the sorption mechanism and the interaction of food biopolymers with water. The moisture sorption isotherms are extremely important in modelling the drying process, design and optimization of drying equipment, predicting shelf-life stability, calculating moisture changes which may occur during storage and selecting appropriate packaging material (Kaymak-Ertekin and Gedik, 2004).

The net isosteric heat of sorption can be used to estimate the energy requirements for drying processes and supply important information about the state of water in food products.

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The level of material moisture content at which the net isosteric heat of sorption approaches the latent heat of vaporization of water is often taken as an indication of the amount of ‘bound-water’ existing in the food. The heat of vaporization of sorbed water may increase to values well above the vaporization of pure water as food is dehydrated to low moisture levels (Yazdani et al., 2006).

In this research, the moisture sorption isotherms of plum halves osmotically pre-treated using sucrose solution that is used commonly for fruit dehydration and trehalose solution that is used recently for vegetables dehydration before air drying (Noriyuki, 2001; Aktas et al., 2007) were determined. The object of this research is to obtain experimental equilibrium desorption and adsorption isotherms of plum halves pre-treated with sugar solutions with 20 and 50 Brix concentrations, in the temperature range from 30°C to 60°C, to propose a mathematical model for prediction of their sorption behaviour as a function of temperature and pre-treatment, to evaluate the constants of sorption isotherm models and to calculate the net isosteric heat of sorption from the experimental data.

Material and Methods

Stanley plums (*Prunus domestica L.)* were acquired from Tekirdag Vineyard Research Institute that produces some fruits varieties in addition to grapes. Obtained fruits were stored at 4°C until the moment of the drying experiments.

Sample Preparation and Pre-treatments

Plums were cut into 2 halves and their stones were removed to determine the sorption isotherms. Osmotic dehydration procedures were applied as 200 g fresh material/400 g solution. Sterilized distilled water was used to prepare all solutions that were mentioned as follows to avoid contamination and other confounding factors. As osmotic solutions, 20 Brix sucrose, 50 Brix sucrose, 20 Brix trehalose and 50 Brix trehalose solutions were used. Samples were immersed and stirred with 80 rpm by using a shaker in the solutions for 30 min and drained (not rinsed). Sorption isotherms of notreated samples were also determined for comparison. Fresh samples that were notreated and sugar treated were used to obtain desorption isotherms. Osmotically dehydrated and notreated samples were dried at 70°C in hot air plum until the weight loss stopped to determine the adsorption isotherms.

Experimental Procedure

The desorption and adsorption isotherms were determined according to standard, static-gravimetric method. This method is based on the use of saturated salt solutions to maintain a fixed water activity (a_w). Although this method requires a long time for hygroscopic equilibrium to be attained, it has the advantage of presenting a more restricted domain of moisture content variation (Yazdani et al., 2006). Silica gel and five saturated salt solutions selected to give different water activity in the range of 0.105–0.836 were used. The salt solutions used and their corresponding water activity values at different temperatures were given in Table 1 (Rahman, 1995; Kaymak-Ertekin and Gedik, 2004). Water activity values of silica gel at maintained temperatures (Table 1) were determined using Testo 650 device combined with humidity measurment probe.

Plum halves were placed on airtight glass dishes which have two separate sections that contain saturated salt solution and sample. The salt solutions were prepared and allowed to stand one week in closed dishes before each experiment and were stirred once per day. The airtight dishes were placed in the temperature controlled cabin (MMM Medcenter, Venticell) that maintained the required temperature. Small amount toluene was placed inside the dish to prevent mould growth due to long period for equilibrium (Labuza, 1984). Samples were weighed periodically until a constant weight was reached. This condition was achieved when the difference between successive weighing was less than 0.001 g/day. The time for equilibrium was about 10 days or more depending on relative humidity and temperature. The moisture contents of the samples were determined using the AOAC method (AOAC, 1980).

Fitting the Models

The experimental sorption data of all samples at four different temperatures were fitted to four sorption equations (with three two-parameter and one three parameter namely GAB) shown in Table 2. BET, GAB, Oswin and Henderson equations were chosen because they are most widely used to fit experimental sorption data of various food materials especially

Table 1
Water activity values of the silica gel and saturated salt solutions at four temperatures used in the experiments (adapted from Rahman, 1995; Kaymak-Ertekin and Gedik, 2004)

Materials	Temperature (°C)			
	30	40	50	60
Silica gel	0.12	0.12	0.114	0.105
$MgCl_2$	0.324	0.316	0.305	0.293
K_2CO_3	0.432	-	-	0.432
$Mg(NO_3)_2$	0.514	0.484	0.454	0.440
NaCl	0.751	0.747	0.744	0.745
KCl	0.836	0.823	0.812	0.803

Table 2
Different models for describing of sorption isotherms

Name of the models	Equations
BET, Brunauer et al. (1938)	$M = \frac{M_0 c a_w}{[(1-a_w)+(c-1)(1-a_w)a_w]}$
Oswin, Oswin (1946)	$M = k(a_w/1-a_w)^n$
Henderson, Henderson (1952)	$1-a_w = \exp(-kTM^n)$
GAB, Van den Berg (1985)	$M = \frac{M_0 C K a_w}{[(1-Ka_w)(1-Ka_w+CKa_w)]}$

for fruits and vegetables. The parameters of the sorption models were estimated from the experimental results using the nonlinear regression analysis and the reduced χ-square, root mean square error (RMSE) and modelling efficiency (EF) were used as the primary criterion to select the best model to account for variation in the sorption data of the samples. Reduced χ-square is used to determine the goodness of the fit. The lower values of the reduced χ-square, the better is of the fit. The RMSE gives the deviation between the predicted and experimental values and it is required to reach zero. The EF also gives the ability of the model and its highest value is 1. These statistical values can be calculated as follows (Ertekin and Yaldiz, 2004, Hossain and Bala, 2002):

$$\chi^2 = \frac{\sum_{i=1}^{N}(M_{\exp,i} - M_{pre,i})^2}{N-n}, \qquad (1)$$

$$RMSE = \sqrt{\frac{\sum_{i=1}^{N}(M_{pre,i} - M_{\exp,i})^2}{N}}, \qquad (2)$$

$$EF = \frac{\sum_{i=1}^{N}(M_{\exp,i} - M_{i,\exp_{mean}})^2 - \sum_{i=1}^{N}(M_{pre,i} - M_{\exp,i})^2}{\sum_{i=1}^{N}(M_{\exp,i} - M_{i,\exp_{mean}})^2}, \qquad (3)$$

here, $M_{exp,i}$ is the ith experimentally observed equilibrium moisture content, $M_{pre,i}$ is the ith predicted equilibrium moisture content, N is the number of observations and n is the number constants in the sorption models and $M_{expmean}$ is the mean value of experimental equilibrium moisture content.

Determination of Isosteric Heat of Sorption

The isosteric heat of sorption was obtained from the sorption isotherm data. This method based on the Clausius-Clapeyron equation, and this equation is often used to evaluate the effect of temperature on the isotherm. The equation is characterized by its simplicity and accuracy and is applicable over a wide temperature range (Phomkong et al., 2006). Clausius-Clapeyron equation is given below (Yazdani et al., 2006):

$$d(\ln a_w) / d(1/T) = -(q_{st}/R), \qquad (4)$$

in which:

$$q_{st} = Qs - \Delta H_{vap}, \qquad (5)$$

where a_w is the water activity, q_{st} is the net isosteric heat of sorption (kJ mol-1), Q_{st} is the isosteric heat of sorption (kJ mol^{-1}), ΔH_{vap} is the heat of vaporisation (kJ mol^{-1} water), R is the universal gas constant (kJ mol^{-1} K^{-1}) and T is absolute temperature (K). The value of q_{st} was calculated from the slope of the plot between values of $ln(a_w)$ and $1/T$ at constant moisture.

Results and Discussion

Experimental Results of Sorption Isotherms

The experimental adsorption and desorption data obtained for selected four temperatures and water activity for plum samples that were no treated and pre-treated using sugar solutions were presented in Table 3.

The results showed that the equilibrium moisture content of all samples increased with water activity at selected temperatures. This may be due to the fact that vapour pressure of water present in foods increases with that of the surroundings (Shivhare et al., 2004). Also equilibrium moisture content of all samples increased with decreasing temperature at constant water activity. Similar results for many plants and foods materials have been reported in the literatures (Lahsasni et al., 2003; Kaymak-Ertekin and Gedik, 2004; Hossein et al., 2001).

The hysteresis effect was observed according to curves of desorption and adsorption. At constant water activity, the equilibrium moisture content of desorption is higher than

Table 3
Experimental equilibrium moisture content of plum samples at selected temperature and water activities

Temp., °C	a_w	Desorption data					Adsorption data				
		No-treated	20 Brix sucrose	50 Brix sucrose	20 Brix trehalose	50 Brix trehalose	No-treated	20 Brix sucrose	50 Brix sucrose	20 Brix trehalose	50 Brix trehalose
30	0.12	0.181	0.182	0.188	0.167	0.165	0.179	0.136	0.157	0.149	0.149
	0.324	0.190	0.200	0.214	0.203	0.197	0.189	0.159	0.168	0.165	0.157
	0.432	0.212	0.222	0.267	0.228	0.220	0.203	0.178	0.174	0.176	0.209
	0.514	0.267	0.294	0.276	0.242	0.252	0.260	0.254	0.219	0.202	0.225
	0.751	0.350	0.327	0.344	0.308	0.346	0.341	0.324	0.278	0.271	0.271
	0.836	0.510	0.520	0.508	0.450	0.495	0.379	0.396	0.361	0.319	0.320
40	0.12	0.179	0.170	0.186	0.165	0.161	0.161	0.136	0.152	0.138	0.146
	0.316	0.188	0.190	0.206	0.191	0.189	0.173	0.143	0.164	0.163	0.155
	0.432	0.212	0.206	0.212	0.226	0.215	0.201	0.174	0.173	0.173	0.189
	0.484	0.245	0.239	0.256	0.234	0.248	0.250	0.224	0.190	0.191	0.218
	0.747	0.323	0.32	0.323	0.305	0.293	0.315	0.259	0.259	0.261	0.255
	0.823	0.355	0.368	0.416	0.350	0.364	0.352	0.273	0.361	0.327	0.318
50	0.114	0.166	0.166	0.151	0.156	0.160	0.147	0.138	0.107	0.118	0.143
	0.305	0.186	0.172	0.167	0.175	0.181	0.173	0.157	0.162	0.136	0.154
	0.432	0.191	0.202	0.221	0.203	0.190	0.180	0.168	0.168	0.149	0.179
	0.454	0.239	0.236	0.249	0.232	0.220	0.211	0.186	0.184	0.154	0.208
	0.744	0.295	0.310	0.313	0.295	0.258	0.279	0.242	0.255	0.226	0.241
	0.812	0.333	0.400	0.364	0.333	0.322	0.319	0.268	0.332	0.270	0.318
60	0.105	0.152	0.165	0.136	0.155	0.149	0.141	0.162	0.121	0.136	0.134
	0.293	0.159	0.190	0.153	0.171	0.158	0.154	0.170	0.149	0.155	0.152
	0.432	0.184	0.200	0.187	0.198	0.190	0.157	0.184	0.184	0.157	0.175
	0.440	0.215	0.215	0.239	0.209	0.218	0.204	0.227	0.235	0.184	0.200
	0.745	0.270	0.264	0.280	0.238	0.256	0.240	0.250	0.274	0.230	0.231
	0.803	0.298	0.282	0.306	0.266	0.286	0.281	0.260	0.305	0.265	0.286

the adsorption one for all samples for all tested temperatures. Desorption and adsorption curves obtained for 30, 40, 50 and 60 °C temperature and different treatments were given in Figure 1. Hysteresis between desorption and adsorption of plum halves at all temperatures can be explained by considering a rigid structure pore connected to its surrounding by a small capillary. During adsorption, the capillary begins to fill as a result of the rising in water activity, while the pore is still empty. When the partial pressure of the vapour in air becomes greater than the vapour pressure of the liquid in the capillary, the moisture will move into the pore. For desorption, the pore is initially full of liquid at saturation. This liquid can escape only when the pressure of the surrounding air becomes lower than the vapour pressure of liquid inside the capillary. As the system of pores has generally a large range of capillary diameters, it results in differences between adsorption and desorption processes (Yazdani et al., 2006).

Fitting of Sorption Models to Experimental Results

Four known mathematical models, BET, Oswin, Henderson and GAB, were used to correlate the experimental data. According to the reduced χ-square, root mean square error (RMSE) and modelling efficiency (EF), Henderson model gave the close fit to the experimental data for the sorption of all samples when model parameters and comparison criteria were found by taking into account all temperature together (Table 4). The GAB model gave the close fit to the experimental data for the sorption of most of samples at four every temperature condition (Table 5). The GAB equation is satisfactory in predicting the equilibrium moisture content of many agricultural materials as stated by Tsami et al. (1990).

The Net Isosteric Sorption Heat Results

The net isoteric heat of sorption calculated by applying the Clasusius–Clapeyron equation for plum halves were given in

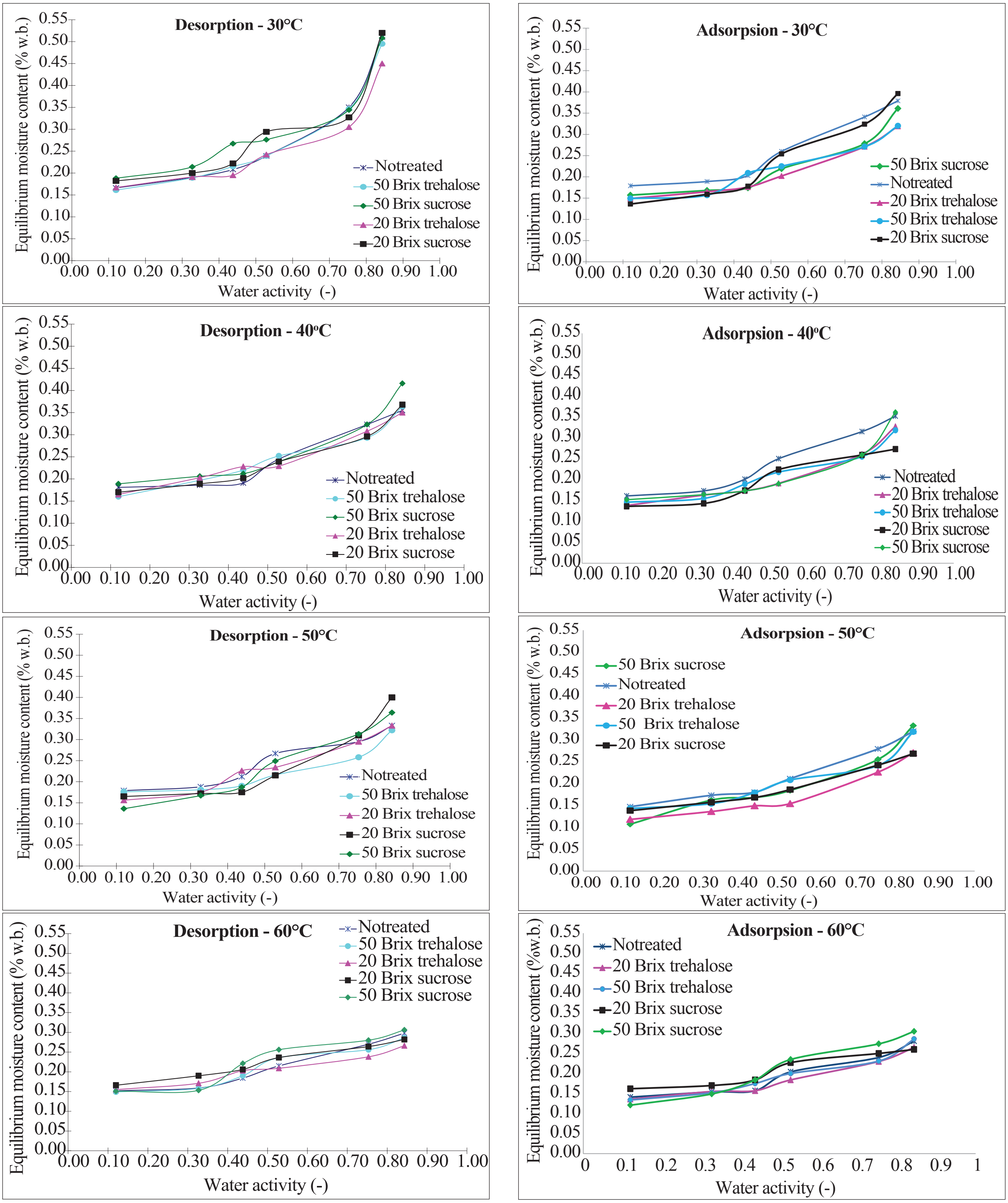

Fig. 1. Variation of desorption (a, b, c, and d) and adsorption (e, f, g, and h) isotherms versus water activity at different temperatures

Figure 2a and 2b. The net isoteric heat of sorption, q_{st}, values were calculated from the slope of the plot between the values of ln(a_w) and 1/*T* at constant moisture content. As seen in Figure 2a and 2b, the isoteric heat has a strong dependence

Table 4
Predicted model parameters and comparison criteria for desorption and adsoprtion models that were taking into account temperatures for plum samples

Model	Desorption data					Adsorption data				
	No-treated	20 Brix sucrose	50 Brix sucrose	20 Brix trehalose	50 Brix trehalose	No-treated	20 Brix sucrose	50 Brix sucrose	20 Brix trehalose	50 Brix trehalose
BET										
M_0	0,081243	0,083381	0,085168	0,077103	0,078715	0,074577	0,067506	0,071293	0,065198	0,067808
C	2210502	2072124	3048045	1260999	6741257	2278197	6954944	2935475	3665324	1846129,
χ2	0,0056	0,0057	0,0059	0,0059	0,0055	0,005278	0,004815	0,003299	0,003815	0,004460
RMSE	0,3501	0,3554	0,3588	0,3603	0,3471	0,340763	0,325474	0,269397	0,289717	0,313257
EF	0,9411	0,9373	0,9342	0,9396	0,9443	0,9489	0,9591	0,9709	0,9683	0,9610
Oswin										
K	0,583687	0,593343	0,614477	0,613557	0,58055	0,224649	0,202992	0,207786	0,197747	0,206767
N	-0,182102	-0,175686	-0,184079	-0,262384	-0,202987	0,251926	0,254599	0,290948	0,241087	0,237232
χ2	0,003797	0,004153	0,003604	0,003032	0,003596	0,000843	0,001219	0,000481	0,000305	0,000313
RMSE	0,289026	0,302284	0,281596	0,258266	0,281283	0,136170	0,163792	0,102882	0,081875	0,083044
EF	0,9598	0,9546	0,959464	0,968976	0,9634	0,9918	0,9896	0,9958	0,9975	0,9973
Henderson										
K	0,589123	0,563233	0,489999	1,088636	0,647073	1,020637	1,227895	1,097003	2,535024	2,539031
N	2,577529	2,589642	2,526239	2,960258	2,592560	2,837845	2,768583	2,760318	3,184992	3,278265
χ2	0,001784	0,001448	0,000928	0,000695	0,000968	0,000507	0,001004	0,001056	0,000456	0,000511
RMSE	0,198117	0,178494	0,142849	0,123627	0,145928	0,105612	0,148651	0,152454	0,100144	0,106065
EF	0,9881	0,9842	0,9896	0,9929	0,9902	0,9951	0,9915	0,9907	0,9962	0,9955
GAB										
M_0	0,143321	0,145639	0,148459	0,146672	0,140602	0,138928	0,125354	0,121470	0,123622	,131302
C	1909142	7198064	941102,0	1161910	1197817	792964,5	542001,7	1097248	1651603	1722,025
K	0,748097	0,753913	0,755729	0,697434	0,739798	0,713422	0,715413	0,770447	0,700619	,686728
χ2	0,001386	0,001582	0,001445	0,000949	0,001430	0,000730	0,001160	0,000383	0,000201	0,000276
RMSE	0,174637	0,186543	0,178328	0,144523	0,177362	0,126721	0,159747	0,091808	0,066557	0,077864
EF	0,9853	0,9827	0,983743	0,990285	0,9855	0,9929	0,9902	0,9966	0,9983	0,9976

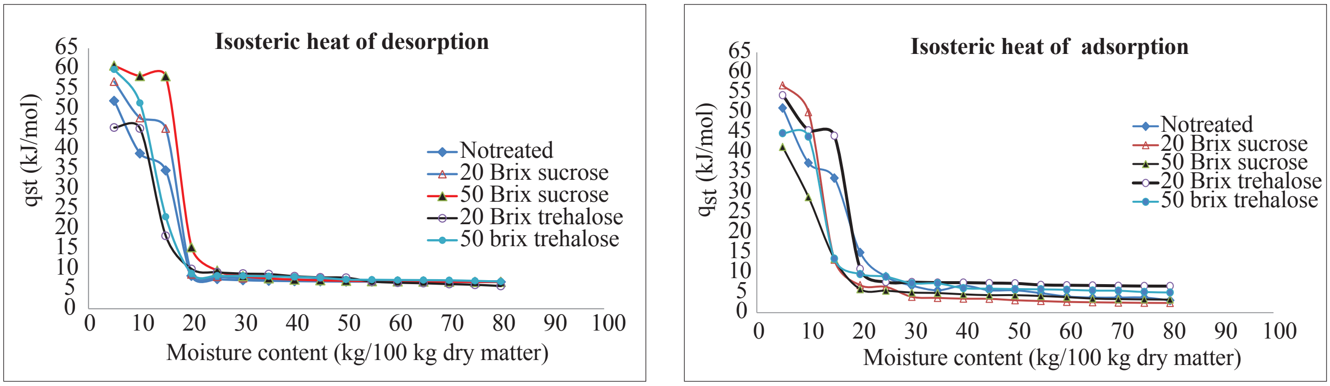

Fig. 2. Variation of net isosteric heat of desorption (a) and adsorption (b) with moisture content for pretreated plum samples

on moisture content, with the energy required for sorption increasing at low moisture contents either for desorption or for adsorption. This reflects the differing strength of water binding; initial occupation of highly active polar sites on the surface (with the greatest interaction energy), followed by the progressive filling of the less available sites with lower bonding activation energies (Yazdani et al., 2006).

As the moisture content increases, the heat of sorption tends to that of pure water; an indication of the moisture existing in the free form. It is observed from Figure 2a and 2b that the net isoteric heat increased whiles the moisture content decreased. It can be seen that from the data in Figure 2, adsorption and desorption data shows that, at a specific moisture content, the isoteric heat of desorption is higher than the

Table 5
Estimated parameters and comparison criteria of the GAB model for plum samples

Sample	Desorption				Adsorption			
	30°C	40°C	50°C	60°C	30°C	40°C	50°C	60°C
Notreated								
M_0	0,140064	0,156279	0,150449	0,137899	0,153457	0,148515	0,134395	0,127127
C	2348942	5507705	1723812	1009742	4617773	353,9130	2009025	2284039
K	0,853511	0,683762	0,669918	0,665764	0,718335	0,707390	0,707488	0,664109
X^2	0,000773	0,073922	0,000251	0,000168	0,000241	0,000267	0,000097	0,000323
RMSE	0,055610	0,543772	0,031657	0,025922	0,031050	0,032659	0,019719	0,035919
EF	0,9929	0,9991	0,9981	0,9989	0,9979	0,9980	0,9994	0,9981
20 Brix sucrose								
M_0	0,147663	0,149594	0,139513	0,160321	0,132705	0,141490	0,102731	0,157096
C	2360820,	2152319,	1796869,	601,8817	83,02702	75,75329	3994806,	507,6205
K	0,832550	0,718431	0,784095	0,534993	0,797766	0,607208	0,752524	0,500943
X^2	0,001838	0,000069	0,000406	0,000026	0,000428	0,000403	0,000036	0,000276
RMSE	0,085753	0,016637	0,040281	0,010214	0,041385	0,040154	0,011950	0,033197
EF	0,9824	0,9994	0,9968	0,9998	0,9969	0,9976	0,9998	0,9982
50 Brix sucrose								
M_0	0,161703	0,156329	0,159654	0,154237	0,127343	0,118819	0,119314	0,159586
C	3132610	2605878	64,83940	55,67088	7334625	7028077	59,84572	31,49498
K	0,791104	0,738425	0,690603	0,627566	0,759834	0,790262	0,773206	0,608317
X^2	0,001114	0,000412	0,000330	0,000500	0,000244	0,000483	0,000248	0,000477
RMSE	0,066750	0,040584	0,036327	0,044738	0,031219	0,043959	0,031494	0,043660
EF	0,9883	0,9963	0,9975	0,9967	0,9981	0,9969	0,9985	0,9970
20 Brix trehalose								
M_0	0,144219	0,158867	0,149818	0,157718	0,127439	0,120346	0,126359	0,123651
C	895296,5	234,4388	230,5360	225,7472	7781247	2028372	1541610	1610989
K	0,788711	0,659552	0,675820	0,494451	0,713200	0,753914	0,647774	0,647483
X^2	0,000758	0,000037	0,000127	0,000081	0,000043	0,000100	0,000028	0,000122
RMSE	0,055078	0,012186	0,022567	0,018017	0,013144	0,019953	0,010629	0,022115
EF	0,9935	0,9997	0,9991	0,9995	0,9997	0,9994	0,9998	0,9993
50 Brix trehalose								
M_0	0,138586	0,155818	0,144781	0,145394	0,142452	0,131663	0,127368	0,130851
C	1755378,	205,8927	7474156	252,6759	199,9096	637663,8	1222240	270,1999
K	0,848015	0,678704	0,649459	0,606171	0,659553	0,695317	0,708353	0,647885
X^2	0,000454	0,000248	0,000273	0,000853	0,000206	0,000260	0,000413	0,000853
RMSE	0,042623	0,031527	0,033039	0,058427	0,028725	0,032259	0,040656	0,058427
EF	0,9959	0,9980	0,9981	0,9987	0,9986	0,9983	0,9974	0,9984

corresponding adsorption value. For example the highest net isoteric desorption heat was found as 60.65 kJ/mol for samples treated using 50 brix sucrose while its value for adsorption was found 41.36 kJ/mol . The highest net isoteric adsorption heat was found as 56.78 kJ/mol for samples treated using 20 brix sucrose. It means that the required energy for moisture desorption was found as maximum for the samples treated using high concentration sucrose and minimum required energy to get to this moisture into the samples was found for samples treated using low concentration solution. According to results on the net isoteric adsorption heat, higher concentration sucrose and trehalose treated samples and no treated samples can get moisture easier (with lower energy) than pretreated samples as seen in Figure 2b.

Trehalose pre-treatments decreased the net isoteric desorption heat of plum halves as seen in Figure 2. On the other hand using of sucrose with high concentration was increased this value compared with other applications and no treatment.

Conclusions

The moisture adsorption and desorption isotherms of treated using sucrose and trehalose solutions with 20 and 50 brix concentration at four temperatures namely 30,40, 50 and 60°C and different water activity values were determined using the gravimetric static method. The equilibrium moisture content of all samples decreased with increasing of temperature.

The hysteresis phenomenon was observed. Among the sorption models chosen to fit sorption curves, the GAB equation described better the sorption isotherms of the samples for all temperatures separately while Henderson equation described better the sorption isotherms of the samples by taking into account temperatures together.

The heat of sorption of plum halves decreases with an increase in moisture content. The net isosteric heat of desorption was found higher than the net isoteric heat of adsorption. Trehalose pretreatments decreased the net isoteric desorption heat of plum halves slices while it increased the net isoteric adsorption heat.

Acknowledgements

This project is being supported by The Scientific & Technological Research Council of Turkey (TUBITAK) (TOVAG-105O544).

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Received January, 23, 2013; accepted for printing December, 2, 2013.