

Water sorption thermodynamic properties applied to persimmon skin and pulp

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Abstract

Moisture equilibrium data of persimmon skin and pulp were determined using the static gravimetric method. Adsorption and desorption isotherms were obtained in the range of 20–70°C, to water activities (a_w) from 0.02 to 0.85. The application of the GAB model to the experimental results, using direct nonlinear regression analysis, provided a good agreement between experimental and calculated values. The net isosteric heat of sorption was estimated from equilibrium sorption data, using the Clausius–Clapeyron equation. Isosteric heats of sorption were found to increase with increasing temperature and could be well adjusted by an exponential relationship. The enthalpy–entropy compensation theory was applied to sorption isotherms and plots of ΔH versus ΔS for skin and pulp provided the isokinetic temperatures, indicating an enthalpy controlled sorption process. © 2000 Elsevier Science B.V. All rights reserved.

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1. Introduction

The persimmon (*Diospyros kaki*) is native to East Asia, most likely China. It has been cultivated in the Mediterranean basin for more than a century. Europeans refer to the persimmon by the Japanese name *kaki*. World production is about 1 200 000 t annually from an area of ≈ 235 000 ha, with China producing around 57% and Japan 27% of the crop. Smaller, but expanding persimmon industries are being developed in Italy (70 000 t), Korea (50 000 t), Brazil (45 000 t), Israel (10 000 t), Spain (8000 t), the U.S. (7000 t),

New Zealand, and Australia. There are over 400 species of persimmon that vary widely in shape and color, although they are broadly classified into two major groups: the non-astringent and astringent varieties. The astringency is due to the high soluble tannin content of the fruit, which diminishes during the natural ripening process. When fully matured the fruit has a tough, glossy, orange-red skin and a yellow-orange flesh, which is very sweet and juicy. The persimmon is mainly eaten fresh, but can be frozen, canned or dried and can be stored for up to 6 months in modified or controlled atmospheres. During drying the tannin cells coagulate so removing the astringency and the sugars in the fruit exude to the surface where they crystallize, thus producing a sweet, candied product [1].

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During dehydration of whole fruits, as in the case of grapes, prunes, persimmons, cherries, etc., the skin acts as a semipermeable membrane that controls the water transfer rate from the fruit to external medium. According to Riva and Masi [2] and Gabas [3], water permeation through grape skin is the governing step of the drying process and the main resistance to water transport is attributed to the cellular layer and the soft wax fraction that cover the fruit. Additional resistance to water diffusion is due to the fruit pulp. Gabas et al. [4] found significant differences between thermodynamic properties and water sorption isotherms for grape skin and pulp treated in a separated way. These results lead us to adopt the same experimental treatment to the persimmon, which skin is also covered by a waxy layer.

Knowledge of water sorption isotherms and isosteric heat of sorption is of essential importance to various food processes such as drying, storage and packaging. They give information about the sorption mechanism and interactions between food components and water. They also help to establish the final moisture content and permit the estimation of energy requirements of drying processes.

Several researchers have determined water sorption isotherms for various products and temperatures and selected ranges of water activity [4–8]. Empirical and semi-empirical equations have been proposed to correlate the equilibrium moisture content with water activity of food products. The Guggenheim–Anderson–deBoer (GAB) equation has been suggested as the most versatile sorption model available in the literature and has been applied successfully to various dehydrated foods [8,9]. It is based on the Brunauer–Emmett–Teller (BET) theory and involves three coefficients which have physical significance, two of them being functions of temperature [10].

A promising theory which has been widely considered to investigate physical and chemical phenomena involved in water sorption [11–13] is enthalpy–entropy compensation or isokinetic theory, which was originally applied by Bell [14]. The isosteric heat of sorption is a useful quantity, particularly in the design of drying processes, as heats of sorption rise well in excess of the heat of vaporization of water as food is dehydrated to low moisture contents [15]. Changes in enthalpy (ΔH) may provide a measure of the energy changes occurring upon mixing of water molecules

with sorbent during sorption processes. Entropy changes (ΔS) may also be associated with the binding or repulsive forces in the system. Gibbs free energy (ΔG) may be indicative of the affinity of sorbents for water and provides a criterion whether water sorption occurs as a spontaneous process. Aguerre et al. [13] applied enthalpy–entropy compensation to derive a two parameter sorption equation which takes into account the effect of temperature on water sorption isotherms of some food products. Ferro-Fontan et al. [16] suggested the existence of a linear relationship between enthalpy and entropy for water sorption in some foods.

The purpose of the present work was to model the effect of temperature on water sorption of skin and pulp of persimmon using the isokinetic theory, and to analyze the implications of the use of one or both parts of the fruit for design and optimization of food process and storage.

2. Mathematical development

2.1. GAB model

The GAB is a relatively simple model with a small number of parameters, which have physical meaning, and can be applied to a high range of water activities. It is mathematically expressed as [15]:

$$X = \frac{(C-1) K a_w X_m}{1 + (C-1) K a_w} + \frac{K a_w X_m}{1 - K a_w} \quad (1)$$

where X_m is the moisture content (dry basis) corresponding to an adsorbed monolayer (BET) and C and K are constants related to the temperature effect.

$$C = C_0 \exp\left(\frac{\Delta H_c}{RT}\right) \quad (2)$$

$$K = k_0 \exp\left(\frac{\Delta H_k}{RT}\right) \quad (3)$$

where ΔH_c and ΔH_k are functions of the heat of sorption of water: $\Delta H_c = H_m - H_n$ and $\Delta H_k = \lambda - H_n$. In the above expressions C_0 and k_0 are adjusted constants for the temperature effect, H_m and H_n the heats of sorption of the monolayer and the multilayer of water respectively, and λ the heat of condensation of pure water.

The parameters can be estimated by fitting the mathematical model to the experimental data, using nonlinear regression.

2.2. Thermodynamic properties

The net isosteric heat of sorption or enthalpy of sorption (q_{st}) is defined as the difference between the total heat of sorption (Q_{st}) and the heat of vaporization of water. It can be determined from the following equation, which is derived from the Clausius–Clapeyron equation [15]:

$$\frac{\partial \ln(a_w)}{\partial(1/T)} \Big|_X = -\frac{Q_{st}-\lambda}{R} = -\frac{q_{st}}{R} \quad (4)$$

The isosteric heat of sorption is a differential molar quantity derived from the temperature dependence of the isotherm. This approach assumes that the net isosteric heat is invariant with temperature and the application of this method requires the measurement of sorption isotherms at two or more temperatures [17].

The change in molar differential entropy of desorption can be calculated from Gibbs–Helmholtz equation as used by [15]:

$$\Delta S = \frac{\Delta H - \Delta G}{T} \quad (5)$$

where the free energy is calculated as

$$\Delta G = RT \ln a_w \quad (6)$$

The effect of a change in water sorption on free energy is usually accompanied by changes on both the enthalpy and the entropy. Substituting Eq. (6) in Eq. (5) and rearranging, it results in:

$$\ln a_w = \frac{\Delta H}{RT} - \frac{\Delta S}{R} \quad (7)$$

Then, the isosteric heat of sorption or enthalpy of sorption and entropy can be calculated from Eq. (7) by plotting $\ln a_w$ versus $1/T$ for certain values of the material moisture content and then determining the slope ($-\Delta H/R$) and the linear coefficient ($\Delta S/R$). This procedure is repeated for many values of X , in order to detect the dependence of q_{st} on the moisture content. Several researchers used the above procedure for the determination of q_{st} . Tsami [18] calculated the heat of sorption of water for dried fruits, Kiranoudis et al. [7]

for some vegetables, Madamba et al. [11] for garlic and McLaughlin and Magee [17] for potatoes.

A plot of ΔH versus ΔS from values of Eq. (7) for the skin and pulp of persimmon can be evaluated for each set of sorption data. These values can be correlated according to the following equation:

$$\Delta H = T_B(\Delta S) + \Delta G_B \quad (8)$$

The isokinetic temperature has an important physical meaning as it represents the temperature at which all reactions in the series proceed at the same rate. Since there is a high degree of linear correlation between enthalpy and entropy, the compensation theory was assumed to be valid for sorption [11]. Krug et al. [19,20] recommended a test for the compensation theory, which involves comparing the isokinetic temperature with the harmonic mean temperature T_{hm} that is defined as:

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

An approximate $(1 - \alpha)$ 100% confidence interval for T_B may be calculated from:

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

where:

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

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

m is the number of $(\Delta H, \Delta S)$ data pairs, $(\overline{\Delta H})$ the average enthalpy, and $(\overline{\Delta S})$ the average entropy.

3. Materials and methods

3.1. Raw material

Samples of persimmon of the Rama Forte variety were kept in a cold room at 7°C prior to their use. The samples were pretreated in a solution of 1.5% ethyl-oleate for 30 s at 50°C, and then rinsed in current water. After that, the persimmons were peeled and the pulp cut in small pieces. Sorption isotherms were determined for skin and pulp separately.

3.2. Sorption isotherms

The equilibrium moisture content of skin and pulp at several temperatures was determined by the static gravimetric method [21], following adsorption and desorption procedures, in the temperature range of 20–70°C. Adsorption isotherms were obtained with samples dehydrated at 60°C to constant weight. Nine saturated salt solutions were prepared corresponding to a range of water activities from 0.02 to 0.85. Each solution was transferred into separated jars in an amount to occupy a space of about 1.5 cm depth at the bottom. 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 required equilibration time was 4–5 weeks based on the change in weight expressed on a dry basis, which did not exceed 0.1% (0.001 g/g dry solids). The equilibrium moisture content was determined by the vacuum oven method [22].

4. Results and discussion

4.1. Sorption isotherms

Equilibrium moisture contents versus water activity for persimmon skin and pulp in the range of temperatures examined are shown in Table 1. The moisture content at each water activity represents the mean value of two replications, each replicate being the mean of five samples.

The behavior of the curves for desorption at 30, 50 and 70°C is shown in Figs. 1 and 2 for skin and pulp, respectively. It can be seen that the equilibrium moisture contents increase with decreasing temperature at constant water activity. This behavior is generally ascribed to a reduction in the number of active sites due to chemical and physical changes induced by temperature; the extent of decrease, therefore, depends on the nature or constitution of the food [15]. The same temperature dependence was observed for both persimmon skin and pulp, although a slight difference can be detected between equilibrium moisture contents of these materials in the case of desorption isotherms (Table 1). Gabas et al. [4], on the other hand, verified that the temperature dependence

Table 1
Experimental equilibrium water contents (kg water/kg dry matter) for persimmon skin and pulp

Temperature (°C)	Water activity	Component persimmon			
		Adsorption		Desorption	
		Skin	Pulp	Skin	Pulp
20	0.070	0.0132	0.0132	0.0132	0.0132
	0.112	0.0217	0.0217	0.0217	0.0217
	0.245	0.0519	0.0519	0.0519	0.0519
	0.331	0.0755	0.0755	0.0755	0.0755
	0.446	0.1145	0.1145	0.1168	0.1259
	0.547	0.1628	0.1630	0.1661	0.1793
	0.655	0.2445	0.2445	0.2567	0.2811
	0.753	0.3718	0.3718	0.3904	0.4276
	0.852	0.6472	0.6349	0.6796	0.7301
30	0.068	0.0102	0.0102	0.0102	0.0102
	0.111	0.0172	0.0172	0.0172	0.0172
	0.223	0.0384	0.0384	0.0384	0.0384
	0.323	0.0626	0.0626	0.0626	0.0626
	0.438	0.0986	0.0986	0.1006	0.1084
	0.525	0.1365	0.1367	0.1393	0.1504
	0.634	0.2105	0.2105	0.2210	0.2421
	0.756	0.3610	0.3610	0.3790	0.4151
	0.834	0.5641	0.5533	0.5923	0.6363
40	0.066	0.0078	0.0078	0.0078	0.0078
	0.111	0.0138	0.0138	0.0138	0.0138
	0.206	0.0288	0.0288	0.0288	0.0288
	0.319	0.0521	0.0521	0.0521	0.0521
	0.432	0.0840	0.0840	0.0857	0.0925
	0.506	0.1132	0.1133	0.1155	0.1246
	0.615	0.1784	0.1784	0.1874	0.2052
	0.753	0.3380	0.3380	0.3549	0.3887
	0.820	0.4988	0.4893	0.5237	0.5627
50	0.059	0.0055	0.0055	0.0055	0.0055
	0.110	0.0111	0.0111	0.0111	0.0111
	0.189	0.0213	0.0213	0.0213	0.0213
	0.314	0.0432	0.0432	0.0432	0.0432
	0.432	0.0732	0.0732	0.0747	0.0806
	0.489	0.0934	0.0935	0.0952	0.1028
	0.599	0.1514	0.1514	0.1590	0.1742
	0.746	0.3075	0.3075	0.3229	0.3537
	0.809	0.4473	0.4388	0.4697	0.5046
60	0.045	0.0033	0.0033	0.0033	0.0033
	0.110	0.0091	0.0091	0.0091	0.0091
	0.175	0.0160	0.0160	0.0160	0.0160
	0.307	0.0354	0.0354	0.0354	0.0354
	0.432	0.0636	0.0636	0.0649	0.0700
	0.473	0.0764	0.0765	0.0780	0.0842
	0.590	0.1310	0.1310	0.1376	0.1507
	0.740	0.2787	0.2787	0.2927	0.3205
	0.800	0.4025	0.3948	0.4226	0.4541

Table 1 (Continued)

Temperature (°C)	Water activity	Component persimmon			
		Adsorption		Desorption	
		Skin	Pulp	Skin	Pulp
70	0.020	0.0011	0.0011	0.0011	0.0011
	0.110	0.0075	0.0075	0.0075	0.0075
	0.162	0.0121	0.0121	0.0121	0.0121
	0.294	0.0280	0.0280	0.0280	0.0280
	0.432	0.0552	0.0547	0.0563	0.0602
	0.458	0.0624	0.0618	0.0636	0.0680
	0.587	0.1157	0.1157	0.1215	0.1331
	0.739	0.2580	0.2580	0.2709	0.2967
	0.795	0.3688	0.3545	0.3872	0.4077

was different for skin and pulp of grape of the Italy variety. Their data showed that grape skin isotherms were almost independent of temperature, while those obtained for grape pulp clearly exhibited a temperature dependence.

A marked intersection of the isotherms and the inverse temperature effect was not observed for persimmon fruit, but there was a tendency for this ‘crossing-over’ at higher water activities. According to Ayranci et al. [5], products which have high sugar content, such as dried apricots, dried figs and raisins showed intersection of the curves, since more

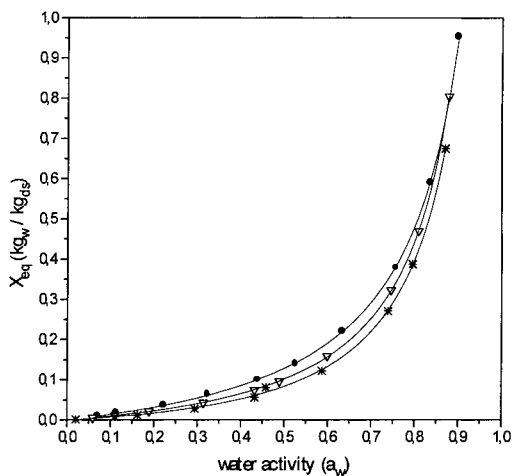


Fig. 1. Desorption isotherms of persimmon skin at different temperatures: ●, $T = 30^{\circ}\text{C}$; ▽, $T = 50^{\circ}\text{C}$; *, $T = 70^{\circ}\text{C}$; —, GAB Model.

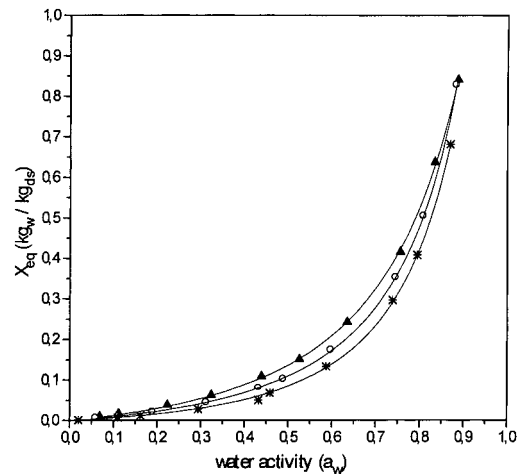


Fig. 2. Desorption isotherms of persimmon pulp at different temperatures: ▲, $T = 30^{\circ}\text{C}$; ○, $T = 50^{\circ}\text{C}$; *, $T = 70^{\circ}\text{C}$; —, GAB Model.

sugar was being dissolved and thus more water was being held by these food products at higher temperatures. They also proposed that isotherm crossing behavior may not be observed for fresh fruits since sugar should already be in dissolved form throughout the whole water activity range. However, Gabas et al. [4] found an isotherm-crossing behavior for grapes even in desorption and in water activities as low as 0.75.

The results of direct nonlinear regression analysis of fitting the GAB equation to the experimental points are shown in Table 2. The fit determination coefficient, r^2 , was found to be satisfactory and is also included in this table.

4.2. Sorption thermodynamic properties

The net isosteric heats of sorption were determined by applying the Eq. (4) obtained from adsorption and desorption isotherms. The results for persimmon skin and pulp are given in Table 3 and clearly show a marked increase in isosteric heats with decreasing moisture contents. The elevated net heats of sorption of water at low moisture contents are an indication of strong water–food component interactions in the fruit. As the moisture content increases, the available sites for sorption of water reduce, resulting in lower values of q_{st} .

Table 2
Estimated GAB parameters for persimmon skin and pulp

Persimmon component	X_m (% dry basis)	C_0 ($\times 10^3$)	ΔH_c (kJ/mol)	k_0	ΔH_k (kJ/mol)	r_c^2	r_k^2
<i>Adsorption data</i>							
Pulp	10.9	0.91	18.4	1.13	-0.344	0.99	0.94
Skin	11.2	0.70	19.0	1.09	-0.264	0.99	0.99
<i>Desorption data</i>							
Pulp	11.6	2.59	16.2	1.14	-0.355	0.99	0.95
Skin	11.5	1.06	18.0	1.10	-0.270	0.99	0.99

Similar effects of moisture content on the heats of sorption were reported by Saravacos et al. [23] for sultana raisins and by Mazza and LeMaguer [24] for yellow globe onion. According to Wang and Brennan [6] the decrease in the isosteric heats with higher amounts of sorbed water can be quantitatively explained by considering that, initially, sorption occurs on the most active available sites giving rise to high interaction energy. As these sites become occupied, sorption occurs on the less active ones, resulting in lower heats of sorption.

It can be also observed in Table 3 that the values of net isosteric heats of desorption are a little higher than those of adsorption, mainly for persimmon pulp, indicating that energy involved in the desorption process is greater than that in adsorption process.

Iglesias and Chirife [25] compared the isosteric heats of water adsorption and desorption for various kinds of food. In general, heats of desorption were significantly higher than adsorption at low moisture

contents. On further sorption, the difference decreased and practically disappeared at high moisture contents. Several theories have been advanced to explain the phenomenon of hysteresis. It is believed that it is not possible to give a simple explanation of this phenomenon in foods, since each food is a complex combination of various constituents, which not only can sorb water independently, but can also interact among themselves giving rise to different water-sorbing capacities. Also in Table 3, it is shown that the differences between heats of sorption for skin and pulp are more evident in the case of desorption, which can be important for the drying process of persimmon.

An empirical exponential relationship between the isosteric heat of sorption and the moisture content, as proposed by Tsami et al. [26] was fitted to the results, according to Eq. (13).

$$q_{st} = q_0 \exp(-X_{eq}/X_0) \quad (13)$$

where q_0 is the isosteric heat of sorption at the first water molecule, X_{eq} the equilibrium moisture content, and X_0 is taken as the characteristic moisture content of a food material.

Table 4 shows the constants q_0 and X_0 and the values of the fit determination coefficient (r^2), which were satisfactory. In general, the calculated values of these

Table 3
Net isosteric heat of sorption for persimmon skin and pulp

X (% dry basis)	Adsorption q_{st} (kJ/mol)		Desorption q_{st} (kJ/mol)	
	Pulp	Skin	Pulp	Skin
4	10.4	10.5	11.0	10.5
8	6.6	6.5	7.2	6.7
10	5.4	5.3	6.0	5.5
15	3.5	3.4	4.0	3.6
20	2.4	2.4	2.8	2.5
25	1.8	1.7	2.1	1.8
30	1.3	1.3	1.6	1.4
40	0.8	0.7	1.0	0.8
50	0.5	0.4	0.7	0.5
60	0.3	0.3	0.4	0.3
70	0.2	0.1	0.3	0.2
80	0.1	0.1	0.2	0.1

Table 4
Estimated parameters of the empirical Eq. (13) for permission

Permission component	q_0 (kJ/mol)	X_0 (% dry basis)	r^2
<i>Adsorption data</i>			
Pulp	15.7	10.1	0.99
Skin	15.8	9.9	0.99
<i>Desorption data</i>			
Pulp	15.9	11.2	0.99
Skin	15.6	10.3	0.98

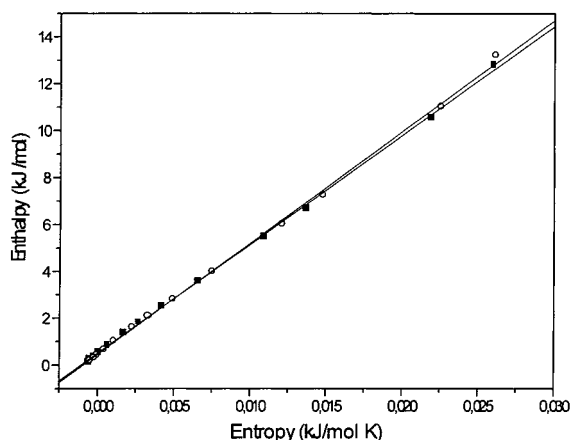


Fig. 3. Enthalpy–entropy relationship for water desorption in persimmon skin and pulp: ■, skin; ○, pulp; —, Eq. (8).

constants were slightly higher for desorption than for adsorption. A similar variation of these constants was observed by Tsami et al. [26] for sultana raisins, figs, prunes and apricots.

From the straight lines calculated by Eq. (7) at different moisture contents of the persimmon, the values of ΔH and ΔS were obtained. A plot of enthalpy versus entropy for desorption is shown in Fig. 3. As the law of compensation could be applied in the range of moistures studied, the results were correlated in accordance with Eq. (8). The parameter T_B that is called the isokinetic temperature was calculated by linear regression for all data sets within a 95% confidence interval. The harmonic mean temperature (T_{hm}) was calculated from Eq. (9). According to Krug et al. [19,20] a linear chemical compensation pattern exists only if $T_B \neq T_{hm}$. The isokinetic temperature for adsorption and desorption of persimmon skin was found to be 458 ± 7 and 464 ± 7 K, as long as for pulp it was found to be 458 ± 8 and 475 ± 10 K. The adjusted r^2 value was 0.99 for all experimental data. The harmonic mean temperature was calculated as 317 K, value significantly different from all values of T_B described above, confirming the suitability of the isokinetic theory for water sorption of persimmon skin and pulp. According to Leffler [27], if $T_B > T_{hm}$ the process is enthalpy driven, while if the opposite condition is observed ($T_B < T_{hm}$), the process is considered to be entropy-controlled.

5. Conclusions

The GAB isotherm model was found to be adequate for the description of the experimental data obtained for persimmon skin and pulp, and the temperature had a similar effect on sorption isotherms of both materials. The equilibrium moisture content increased with decreasing temperature at constant water activity. The net isosteric heat of sorption was higher as the moisture content was reduced, the heat of desorption being little higher than that of adsorption at low moisture contents. An empirical exponential relationship could describe the heat of sorption dependence on the material moisture content. The enthalpy–entropy compensation or the isokinetic theory could be successfully applied to water sorption by persimmon skin and pulp and suggests that this process could occur by enthalpy-controlled mechanisms.

6. Symbols

a_w	water activity
C	constant in GAB model Eq. (1)
ΔG	change in Gibbs free energy (J/mol)
ΔH	change in enthalpy (J/mol)
K	constant in GAB model Eq. (1)
n	number of isotherms
q_{st}	net isosteric heat of sorption (kJ/mol)
R	universal gas constant (8.314 J/mol K)
r_c^2	fit determination coefficient for parameters ΔH_c and C_0 in Table 2
r_k^2	fit determination coefficient for parameters ΔH_k and k_0 in Table 2
ΔS	change in specific entropy (J/mol K)
T	absolute temperature (K)
T_B	estimated isokinetic temperature (K)
Var	statistical variance
X	moisture content
X_m	constant in GAB model Eq. (1)

Superscript

— Average

Subscripts

B isokinetic

ds	dry solids
eq	equilibrium
hm	harmonic mean
m	first layer
n	multilayers
w	water

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