



Equilibrium Moisture Content and Heat of Desorption of Some Vegetables

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ABSTRACT

The equilibrium moisture content of potato, carrot, tomato, green pepper and onion was determined within the range of 10 to 90% water activity at three different temperatures (30, 45 and 60°C), using the standard static gravimetric method developed by the European Cooperation Project COST 90. The GAB equation was fitted to the experimental data, using the direct nonlinear regression analysis method and the agreement between experimental and calculated values was satisfactory. The net isosteric heat of desorption of water was determined from the equilibrium desorption data, using the Clausius–Clapeyron equation. An empirical exponential relationship between the net isosteric heat of sorption and the moisture content was proposed and validated.

INTRODUCTION

The equilibrium moisture content and net isosteric heat of desorption are essential properties in the analysis and design of various food processes such as preservation, drying, storing, packaging and mixing. The moisture sorption isotherm data are used to calculate the driving force (potential) in many mass transfer operations and also to evaluate the optimum residual moisture content of the resulting products, in order to increase their stability. The net isosteric heat of sorption can be used to estimate the energy requirements of dehydration processes. The level of material moisture content at which the net isosteric heat of sorption approaches the latent heat of vaporisation of water is often taken as an indication of the amount of 'bound water' existing in the food

(Duckworth, 1972). The values of this isosteric heat can be increased to such levels, when the material moisture content decreases below this critical 'bound water' limit, that they must be taken into consideration when striving for reasonable heat transfer balances in process calculations (Rizvi, 1986).

A static gravimetric method for the experimental determination of desorption isotherms was developed and standardised in the European COST 90 project (Wolf *et al.*, 1984). Moisture sorption data for vegetables published in the literature have been obtained at various levels of temperature and water activity values (Gane, 1948, 1950; Makower & Dye, 1956; Nemitz, 1961; Taylor, 1961; Saravacos & Stinchfield, 1965; Lewicki & Brzozowski, 1973; Lewicki & Lenart, 1975; Alcazaz *et al.*, 1977; Lomauro *et al.*, 1985; Carbonell *et al.*, 1986; Aguerre *et al.*, 1989; Diamante & Murno, 1990; Samaniego-Esquerra *et al.*, 1991; Timmermann & Chirife, 1991; Wang & Brennan, 1991). Moreover, a large amount of sorptional data have been presented for various vegetables by Iglesias and Chirife (1982).

Several empirical and semiempirical equations have been proposed for the correlation of the equilibrium moisture content with the water activity of food products. Among them the GAB (Guggenheim-Anderson-de Boer) equation has been applied successfully to various foods (Van den Berg, 1985), and it is recommended by the European Project COST 90 on Physical Properties of Foods (Wolf *et al.*, 1984). It is based on the BET (Brunauer-Emmett-Teller) theory and generally involves three coefficients which have physical significance. Normally, temperature effects can be expressed by introducing a temperature dependence into two of these coefficients. In many cases, an additional dependence of the third coefficient on temperature has been recognised (Iglesias & Chirife, 1984; Weisser, 1985).

Heat of desorption can be determined by means of calorimetric measurements or from moisture sorption data (Tsami *et al.*, 1990a). The second method is more convenient since equilibrium moisture content data are determined routinely. Values of isosteric heats of desorption, obtained by means of the second method, have been reported in the literature for several foods, including vegetables and fruits (Makower, 1945; Becker & Sallans, 1956; Bushuk & Winkler, 1957; Rodriguez-Arias *et al.*, 1963; Saravacos & Stinchfield, 1965; Chung & Pfof, 1967; Iglesias & Chirife, 1976; Chirife *et al.*, 1977; Tsami *et al.*, 1990a; Wang & Brennan, 1991).

The objective of the present study was to provide experimental desorption isothermal data for five commercial vegetables (potato,

carrot, tomato, green pepper and onion), to verify the adequacy of the GAB equation, to calculate the net isosteric heat of desorption of water from the equilibrium desorption data using the Clausius–Clapeyron equation, and to propose an exponential relationship for the dependence of the net isosteric heat of desorption on the material moisture content.

MATERIALS AND METHODS

Experimental procedure

The desorption isotherms were determined, using the standard static gravimetric method developed by the European Cooperative Project COST 90 (Spiess & Wolf, 1983).

The experimental apparatus consisted of three thermostatically controlled water baths accurate to $\pm 0.2^\circ\text{C}$, each containing 10 one-litre airtight glass jars (hygrostats). Each hygrostat contained a saturated salt solution selected to give defined constant water activity inside the jar. Each salt solution was prepared according to the specifications presented in Table 1 (Greenspan, 1977). Five weighing bottles were placed in each hygrostat.

The accuracy and reliability of the experimental apparatus were checked by measuring the sorption isotherm of a standard method reference material, specifically MCC (microcrystalline cellulose, Merck Avicel PH101, Darmstadt, Germany). The mean value of five measurements at 10 water activities differ from the reference values by

TABLE 1
Preparation of Hygrostats According to COST-90 Project

<i>Jar</i>	<i>Salt</i>	<i>Anhydrous salt (g)</i>	<i>Water (g)</i>
1	LiCl	150	85
2	CH ₃ COOK	200	65
3	MgCl ₂	200	25
4	K ₂ CO ₃	200	90
5	Mg(NO ₃) ₂	200	30
6	NaBr	200	80
7	SrCl ₂	200	50
8	NaCl	200	60
9	KCl	200	80
10	BaCl ₂	200	70

no more than the critical difference: 0.498 kg water/100 kg dry solids. Moreover, the standard experimental error is less than the critical value of 0.060 kg water/100 kg dry solids (Wolf *et al.*, 1984).

Commercial samples of the following Greek vegetables were used in these experiments: potato (80% moisture content, wet basis), carrot (90% moisture content, wet basis), tomato (97% moisture content, wet basis), green pepper (94% moisture content, wet basis), and onion (85% moisture content, wet basis). Representative samples were taken from the bulk of the products. The samples were cut into thin slices of 1 to 2 mm. In order to prevent chemical browning, the samples were blanched in hot water at 80°C for 5 min, treated with ascorbic acid solution (2%, w/w) for 5 min, and potassium sorbate solution (2%, w/w) for 10 min. The samples were then distributed into five weighing bottles for each glass jar. A small quantity of thymol was placed in each hygrosat in order to prevent fungal activity (Wolf *et al.*, 1985). Five replications of the same experiment were carried out.

Equilibrium was reached within 15 days maximum for all the samples (different water activities and different temperatures). It was considered that equilibrium was obtained when the sample weight difference between two successive days was less than the balance accuracy (Saravacos *et al.*, 1986).

The total time for removal, weighing and putting back in the hygrosats was about 30 s (as recommended by the Cooperative Project). This minimizes atmospheric moisture sorption during weighing.

The equilibrium moisture content was determined, using a vacuum oven at 70°C and 50 torr for 6 h (AOAC, 1980).

Modelling equations

A detailed search of the literature (Van den Berg & Bruin, 1981) concluded that the most efficient equation for the prediction of the sorption isotherms was the GAB equation. It 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 (Van den Berg, 1985; Weisser, 1985).

The GAB equation is normally written in the following form (Maroulis *et al.*, 1988):

$$X = X_m C k a_w / [(1 - k a_w)(1 - (1 - C) k a_w)] \quad (1)$$

In this expression, X is the equilibrium material moisture content (kg/kg, dry basis), a_w is the water activity, X_m is the monolayer moisture con-

tent (kg/kg, dry basis), while C and k are related to the temperature effect through the equations (Van den Berg, 1985):

$$C = C_0 \exp(\Delta H_c/RT) \quad (2)$$

$$k = k_0 \exp(\Delta H_k/RT) \quad (3)$$

where T is the absolute temperature (K), R is the universal gas constant (J/mol K), and ΔH_c and ΔH_k are functions of the heat of sorption of water (J/mol): $\Delta H_c = H_m - H_n$ and $\Delta H_k = H_1 - H_n$ (Van den Berg, 1985; Weisser, 1985). In these expressions C_0 and k_0 are constants, H_m and H_n are the heats of sorption of mono- and multi-molecular layers of water (J/mol), respectively, while H_1 is the heat of condensation of water vapour (J/mol).

The parameters were estimated by fitting the mathematical model to the experimental data, using the method of direct nonlinear regression. This method estimates the parameters X_m , C_0 , ΔH_c , k_0 and ΔH_k by fitting the equation resulting from substituting of eqns (2) and (3) into eqn (1) to all the experimental data. This procedure exhibits several advantages over indirect nonlinear regression (Maroulis *et al.*, 1988). When, for example, successive regressions are involved, the first estimation may introduce significant uncertainties into the second and third regressions. Thus, the application of the indirect regression method depends strongly on the confidence regions of the constants obtained from the first approximation (Maroulis *et al.*, 1988). Linear regression, on the other hand (the GAB model can be transformed into a linear model, and linear regression can be applied), can give highly erroneous results and should be avoided (Schaer & Rugg, 1985). When there exist differences in the variance of the data (S_i^2 is dependent on the water activity), the direct nonlinear weighted regression method should be used (Samaniego-Esquerria *et al.*, 1991).

Consequently, the parameters are estimated by minimization of the residual sum of squares (SST) (Least squares method; Beck & Arnold, 1977):

$$SST = \sum_{i=1}^N \sum_{j=1}^{n_i} (\psi_{ij} - y_{ij})^2 \quad (4)$$

where ψ_{ij} is the experimental value of the dependent variable (equilibrium water content) of the j th replicate of the i th experiment, y_i is the calculated value of the model for the i th experiment, n_i is the number of replicates in the i th experiment, and N is the total number of experiments.

The residual sum of squares, SST, consists of the lack of fit sum of squares, SSR, and the pure error sum of squares, SSE, i.e.,

$$\text{SST} = \text{SSR} + \text{SSE} \quad (5)$$

where

$$\text{SSR} = \sum_{i=1}^N n_i (\psi_{ij} - y_i)^2 \quad (6)$$

$$\text{SSE} = \sum_{i=1}^n n_i S_i \quad (7)$$

where

$$\psi_i = \sum_{j=1}^{n_i} \psi_{ij} / n_i \quad (8)$$

and

$$S_i^2 = \sum_{j=1}^{n_i} (\psi_{ij} - y_i)^2 / n_i \quad (9)$$

The standard deviation between experimental and calculated values, S_R , and the standard experimental error, S_E , can then be calculated from the following equations:

$$S_R^2 = \text{SSR} / (N - p) \quad (10)$$

$$S_E^2 = \text{SSE} / (M - N) \quad (11)$$

$$M = \sum_{i=1}^N n_i \quad (12)$$

where p is the number of parameters.

A model is considered acceptable if the standard deviation, S_R , between experimental and calculated values is close to the standard experimental error (S_E).

The net isosteric heat of desorption of water (q_{st}) is defined as the total heat of desorption of water from the material minus the heat of vaporization of water.

It can be determined from the following equation, which is derived from the Clausius–Clapeyron equation (Rizvi, 1986; Tsami *et al.*,

1990b):

$$q_{st} = -R \, d \ln a_w / d(1/T) \quad (13)$$

where a_w is the water activity, T is the absolute temperature, and R is the universal gas constant.

The net isosteric heat of desorption can be calculated from eqn (6) by plotting the sorption isotherm as $\ln a_w$ versus $1/T$ for certain values of the material moisture content and then determining the slope, which is equal to $-q_{st}/R$. It is assumed here that q_{st} is invariant with temperature and that the application of this method requires the measurement of sorption isotherms at more than two temperatures.

The quantity q_{st} decreases considerably when the moisture content is increased. An empirical exponential relationship between the net isosteric heat of sorption and the moisture content has been proposed (Tsami *et al.*, 1990b):

$$q_{st} = q_0 \exp(-X/X_0) \quad (14)$$

In this expression, X is the equilibrium moisture content (kg/kg, dry basis), q_0 is the net isosteric heat of sorption of the first molecule of water in the food (J/mol) (clearly, $q_{st} \rightarrow q_0$ as $X \rightarrow 0$), and X_0 is a characteristic moisture content of the food material (kg/kg, dry basis), at which this net isosteric heat of desorption has been reduced by 63%. The constants q_0 and X_0 are estimated by fitting eqn (14) to the values of q_{st} , obtained by applying eqn (14) to experimental isotherms.

RESULTS AND DISCUSSION

Equilibrium moisture content

The results of the experimental measurement of the equilibrium moisture content of potato, carrot, tomato, green pepper and onion at three temperatures and 10 water activities are shown in Table 2. The equilibrium moisture content at each water activity represents the mean value of five replications (eqn (8)). The standard deviation of each experimental point S_i (eqn (9)) is also given in Table 2.

The experimental data were compared with similar data found in the literature and the results are considered satisfactory. In the case of potato (see Fig. 1), the experimental data obtained agree with those presented in the literature, being closer to the data produced by Saravacos and Stinchfield (1965) and Gane (1950). In the case of carrot, onion and tomato, a disagreement between experimental data obtained and those

TABLE 2
Equilibrium Moisture Content (% dry basis) of Vegetables

Water activity	Potato		Carrot		Tomato		Green pepper		Onion	
	Mean value	Standard deviation	Mean value	Standard deviation	Mean value	Standard deviation	Mean value	Standard deviation	Mean value	Standard deviation
	30°C									
0.113	6.4	0.5	7.4	1.0	13.4	1.1	11.4	0.4	10.9	0.7
0.216	8.4	0.2	12.4	2.0	18.4	1.7	16.5	0.5	15.9	0.6
0.324	10.2	1.3	16.2	1.5	21.2	2.2	20.2	2.0	20.2	0.5
0.432	12.0	1.0	21.0	1.9	24.5	2.3	25.5	1.8	24.5	2.0
0.514	13.6	1.0	25.1	2.1	27.6	1.3	28.6	2.3	27.6	2.3
0.560	14.5	1.8	27.5	2.1	29.5	1.7	30.5	1.3	30.0	1.4
0.691	18.2	1.4	35.2	3.2	38.2	2.4	39.2	1.3	37.7	2.5
0.751	22.2	1.7	39.2	3.7	43.2	2.2	45.2	3.1	42.7	2.1
0.836	28.9	1.6	47.9	5.7	53.9	3.6	54.9	3.2	50.9	2.9
0.900	32.0	2.7	51.0	2.3	59.0	2.5	60.1	4.0	57.0	3.9
	45°C									
0.112	4.8	1.2	6.3	1.5	9.8	2.3	7.8	1.5	7.8	0.3
0.195	6.8	1.1	9.8	1.9	13.8	2.6	11.8	0.5	11.8	0.5
0.311	8.9	1.6	12.4	1.5	17.4	1.3	14.4	1.1	15.4	0.6
0.432	10.4	1.1	15.9	1.9	20.9	3.3	17.9	0.8	17.9	1.9
0.469	10.6	2.1	16.6	2.1	21.6	2.3	19.6	1.3	18.6	2.3

0-520	11.3	1.2	19.3	2.0	23.8	1.5	21.8	1.2	20.8	1.2
0-640	14.1	1.5	25.6	2.3	28.6	4.5	26.6	1.4	27.1	1.6
0-745	17.5	1.7	30.5	3.7	35.0	3.2	34.0	2.1	33.0	1.1
0-817	21.1	2.3	36.1	4.2	41.1	3.7	39.1	2.2	38.1	2.0
0-880	24.5	2.8	41.0	8.4	46.0	4.7	45.0	4.1	43.0	3.1
60°C										
0-110	3.0	0.9	4.0	1.8	7.0	1.0	5.0	1.3	4.5	1.0
0-160	4.3	1.7	5.3	2.0	9.3	1.2	6.3	1.5	6.8	0.6
0-293	5.5	1.2	8.5	1.5	12.5	1.2	9.5	1.4	10.0	0.5
0-432	6.2	1.5	11.2	1.1	14.9	1.5	12.9	0.7	12.9	1.8
0-440	6.9	1.9	11.4	2.1	14.7	1.3	12.7	1.3	13.2	2.3
0-497	7.2	1.2	12.7	1.2	15.7	1.6	14.2	1.2	14.7	1.3
0-580	8.8	1.8	15.3	1.2	18.8	2.2	17.8	1.2	18.3	2.4
0-745	11.3	2.2	21.3	3.4	25.3	3.2	23.3	3.1	23.3	1.8
0-803	13.5	2.8	24.5	5.8	28.5	2.9	26.5	5.3	26.0	2.8
0-840	14.9	3.6	26.9	6.0	30.9	3.0	28.9	4.1	28.9	3.3

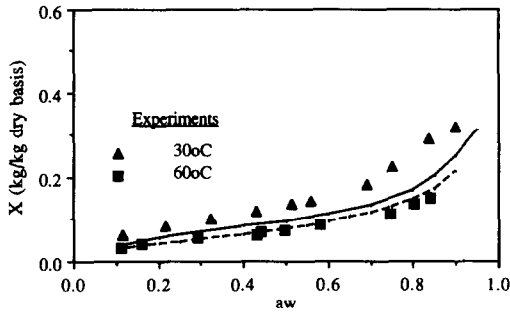


Fig. 1. Comparison of experimental data to those from the literature for potato. Literature data: —, 30°C (Saravacos & Stinchfield, 1965); ---, 60°C (Wang & Brennan, 1991).

of the literature was found for lower values of water activities (Gane, 1948; Gane, 1950; Nemitz, 1961; Taylor, 1961; Lewicki & Brzozowski, 1973; Lewicki & Lenart, 1975; Alcazaz *et al.*, 1977; Samaniego-Esquerria *et al.*, 1991). In the case of pepper, all the experimental values that were measured were found higher than those presented in the literature (Nemitz, 1961; Carbonell *et al.*, 1986).

The results of direct nonlinear regression analysis of fitting the GAB equation to the experimental points are shown in Table 3. In this table, the standard experimental error, S_E , and the standard deviation between the experimental and the calculated values, S_R , are also included (see eqns (11) and (10)). In all cases the standard deviation between the experimental and the calculated values is very close (or smaller, such as in the case of carrot) to the standard experimental error, which indicates that the GAB equation is satisfactory in predicting the equilibrium moisture content of vegetables.

The experimental points, as well as the prediction of the GAB equation for each temperature employed and for each vegetable examined, are shown in Fig. 2.

The results of fitting the GAB model for vegetables were compared to those for fruits presented on Table 4 (Tsami *et al.*, 1990b). The estimated values of the parameter X_m for vegetables (except for the potato), which varied between 18.2 and 21.2 kg/kg (dry basis) were found to be greater than those for fruits, which varied between 10.5 and 15.0 kg/kg (dry basis). This parameter is the monolayer moisture content, and it is a measure of sorption possibility of the material. The vegetables examined can be considered as more hygroscopic than fruits. The values ΔH_k and ΔH_c represent the mean values of the heat of

TABLE 3
Estimated GAB Parameters for Vegetables

Vegetable	X_m (% dry basis)	C_0 ($\times 10^3$)	ΔH_c (KJ/mol)	k_0 ($\times 10^2$)	ΔH_k (KJ/mol)	S_E (% dry basis)	S_R (% dry basis)
Potato	8.7	1.86	34.1	5.68	6.75	1.89	2.50
Carrot	21.2	5.94	28.9	8.03	5.49	3.39	2.56
Tomato	18.2	1.99	34.5	5.52	6.70	2.10	3.47
Green pepper	21.1	1.46	33.4	5.56	6.56	1.91	2.84
Onion	20.2	2.30	32.5	5.79	6.43	1.74	2.45

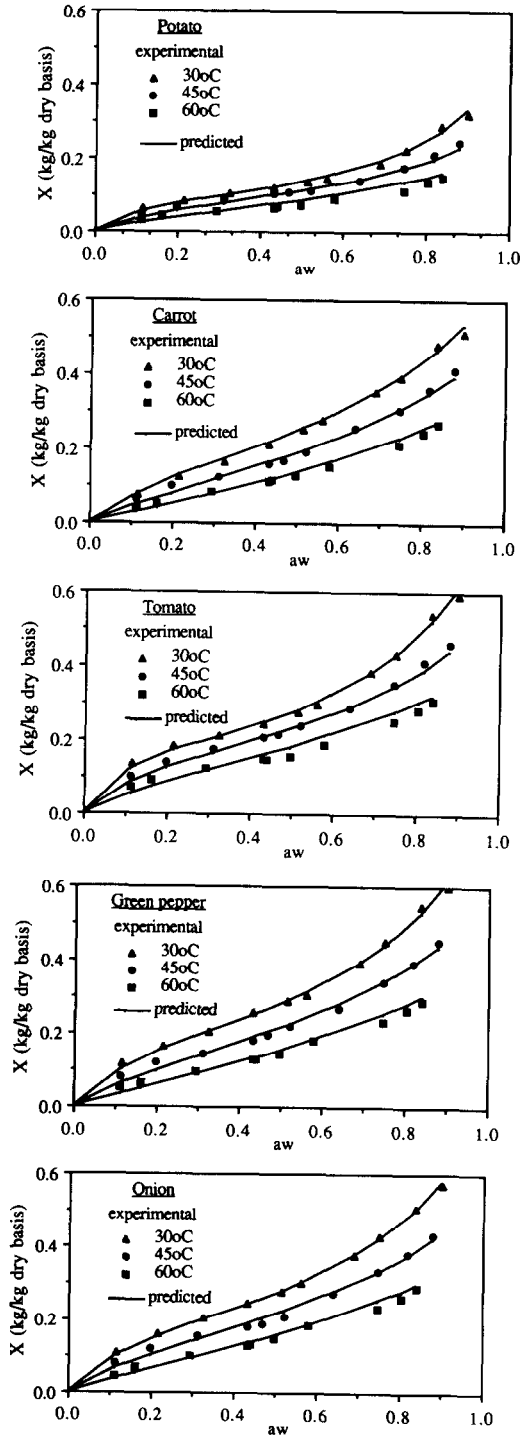


Fig. 2. Equilibrium moisture content and the prediction of the GAB model for vegetables.

sorption of water on the dried material. Parameter ΔH_k is the difference in enthalpy between monolayer and multilayer sorption, which is expected to have a large positive value, due to the straight exothermic interaction of water vapour with primary sorption site of the material. The estimated values for vegetables, which varied between 28.9 and 34.5 kJ/mol, were found to be greater than those for fruits, which varied between 14.3 and 22.2 kJ/mol. Parameter ΔH_c is the difference between the heat of condensation of water and the heat of sorption of a multi-molecular layer. The estimated values were found to be positive for vegetables (5.49 to 6.75 kJ/mol), while in the case of fruits they were found to be negative (-1.30 to -0.154 kJ/mol). The above comparison proves the differences between fruits and vegetables with regard to the sorption characteristics. Furthermore, the desorption isotherms for vegetables do not intersect, while the corresponding ones for the fruits intersect themselves, and the inverse temperature effect was not observed. Intersection of sorption isotherms was observed for products with high sugar content such as fruits (Saravacos *et al.*, 1986; Maroulis *et al.*, 1988), sugar alcohols (Weisser, 1985) and biscuits (Hellen & Gilbert, 1985). For products with low sugars (e.g. apples) the curves do not intersect (Roman *et al.*, 1982). For products with high protein or high starch content, there is also no intersection point with increased temperature (Benado & Rizvi, 1985). On the basis of the above, for foods with very low sugar and moderate high starch and protein content, the expected results should be similar to those presented here.

Isosteric heat of sorption

The isosteric heat of desorption of water at each moisture content was obtained by fitting eqn (13) to the data of equilibrium water activity versus temperature, obtained from desorption isotherms of vegetables. The results are given on Table 5. Equation (14) was then fitted to these data to obtain the dependence of the net isosteric heat of sorption on the equilibrium moisture content.

The results of the nonlinear regression are shown on Table 6. The standard deviation between the experimental and the calculated values is also included.

The experimental points, as well as the predictions of eqn (14) for each vegetable examined, are shown in Fig. 3.

The predictions of this empirical equation for all vegetables are shown in Fig. 4. In the case of potato and tomato, the net isosteric heat of desorption decreased rapidly with material moisture content, while in the case of the other vegetables the net isosteric heat of sorption

TABLE 4
Estimated GAB Parameters for Fruits

Fruit	X_m (% dry basis)	C_0 ($\times 10^3$)	ΔH_c (KJ/mol)	k_0	ΔH_k (KJ/mol)	S_E (% dry basis)	S_R (% dry basis)
Raisins	10.9	5.26	14.3	1.06	-0.154	0.95	1.62
Currants	15.0	1.66	15.6	1.60	-1.299	1.66	1.70
Figs	11.8	0.46	20.2	1.11	-0.361	1.03	1.67
Prunes	11.7	0.51	19.6	1.08	-0.227	2.99	2.02
Apricots	10.5	0.15	22.2	1.30	-0.659	1.36	1.84

TABLE 5
Net Isosteric Heat of Sorption for Vegetables

<i>X</i> (% dry basis)	Potato	Carrot	Tomato	Green pepper	Onion
	q_{st} (KJ/mol)				
6.0	34.8				
8.0	28.0	28.7			
10.0	23.1	24.2		35.3	37.1
12.0	15.9	22.4	42.9	34.3	32.9
14.0	11.6	19.8	36.1	30.7	29.9
16.0	9.5	18.3	32.2	28.4	28.8
18.0	8.3	16.2	28.8	25.2	25.7
20.0	7.3	15.6	23.1	22.9	22.8
22.0		13.7	18.9	19.4	19.0
24.0		12.5	15.4	17.2	16.9
26.0		11.4	13.2	16.4	14.8
28.0		11.0	10.9	15.3	13.6
30.0		10.2	9.4	13.0	12.1
32.0			8.8		
34.0			8.1		
36.0			7.4		

TABLE 6
Estimated Parameters of the Empirical eqn (5) for Vegetables

Vegetable	q_0 (KJ/mol)	X_0 (% dry basis)	S_R (KJ/mol)
Potato	74.6	8.1	1.14
Carrot	40.5	20.6	0.64
Tomato	114.2	12.4	1.02
Green pepper	61.1	19.9	0.92
Onion	65.0	18.3	0.97

decreased smoothly. Furthermore, the net isosteric heat of desorption for green pepper and onion is practically the same.

The results were compared to those of the literature. In the case of potato, Wang and Brennan (1991) reported a value of 8 kcal/mol at 0.02 kg/kg (dry basis) falling to 4 kcal/mol at 0.1 kg/kg (dry basis). Chirife *et*

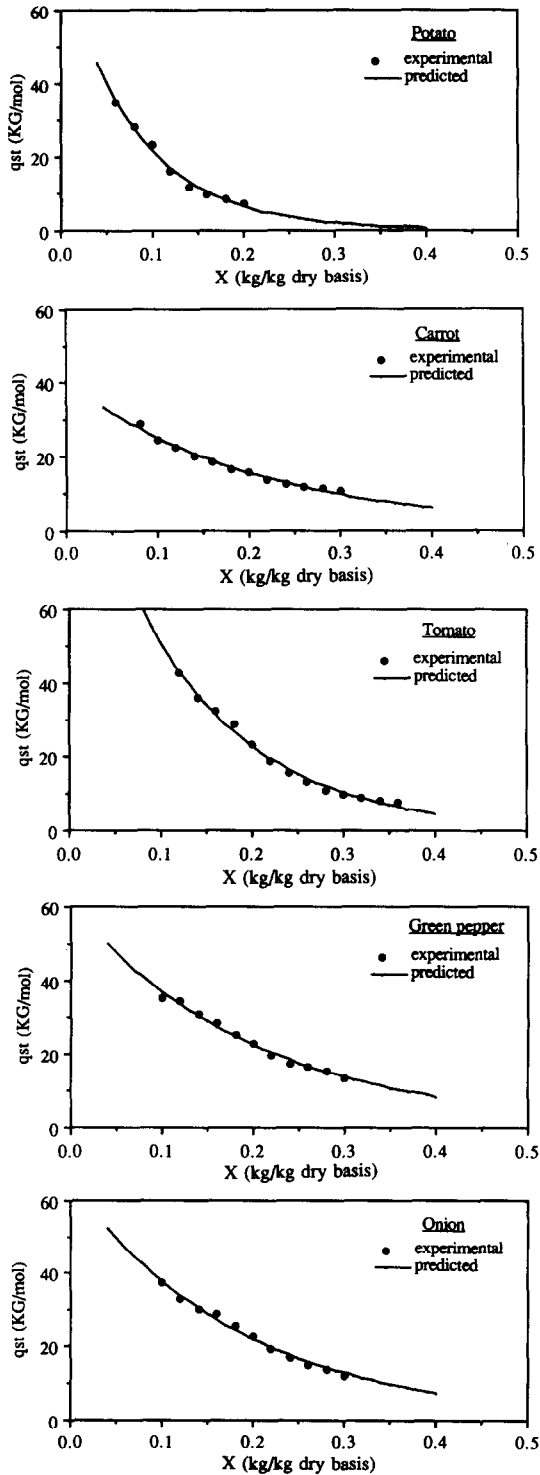


Fig. 3. Net isosteric heat of sorption for vegetables.

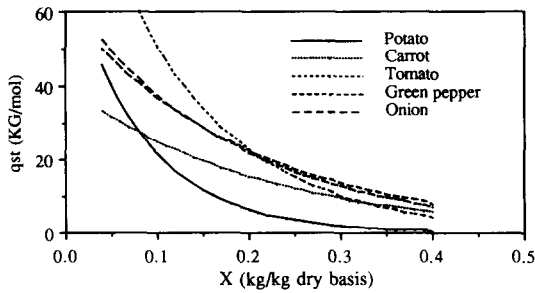


Fig. 4. Comparison of the net isosteric heat of sorption for vegetables.

al. (1977) reported maximum values of net isosteric heat of desorption ranging from 10.8 to 13.8 kcal/mol for the other vegetables.

The net isosteric heat of desorption for vegetables was compared to the one for fruits (Tsami *et al.*, 1990a). In the case of vegetables, the net isosteric heat of desorption remained significant for a wide range of values of material moisture content, while in the case of fruits, it was found negligible over this range of moisture content, and increased dramatically when the material moisture approached zero. Moreover, in the case of fruits the net isosteric heat of sorption was found to have a slightly negative value for large values of material moisture content, a phenomenon that was not observed in the case of vegetables.

CONCLUSIONS

- The standard gravimetric method can be used successfully for the experimental determination of the equilibrium moisture content of vegetables.
- The GAB model proved to be satisfactory for the prediction of the experimental data obtained.
- The net isosteric heat of sorption can be calculated using the Clausius–Clapeyron equation.
- An exponential equation can describe the dependence of the net isosteric heat of sorption on the material moisture content.

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