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Moisture adsorption characteristics of solar-dried mango slices

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Abstract

Moisture adsorption isotherms of solar-dried mango slices (cv. Kitchener) were determined at three temperatures (20°C, 30°C and 40°C) for a water activity (a_w) range of 0.111 to 0.813 using the standard static gravimetric method. The sorption characteristic of solar-dried mango slices followed type III (J-shaped) isotherms, characteristic of high sugar products. An intersection of the curves occurred at a^w=0.75 for the investigated temperatures, which indicates the occurrence of the reverse temperature effect. The experimental equilibrium moisture content data were subsequently fitted to two well-known mathematical sorption models; namely, the BET and GAB models. Non-linear regression analysis was used to evaluate the models' parameters. The validation of the two selected models was done by using statistical parameters: the mean relative percentage deviation (% P) and the coefficient of determination (R^2). The estimated parameters of the GAB model were correlated to temperature. The (*K*) parameter of GAB model was found to increase with temperature, whereas the monolayer moisture content (*Mo*) decreased with increasing temperature. The Arrhenius-type relationships of the GAB parameters were determined. The GAB model was satisfactorily used to predict the equilibrium moisture content (EMC) of solar dried-mango slices.

Introduction

Mango fruit (*Mangifera indica* L.) is one of the most important seasonal fruits of tropical and subtropical countries. It contains high amounts of sugars and considerable amounts of vitamin C and provitamin A. Mango fruit is highly perishable and must be consumed within a few days after harvesting or it must be dried to a lower moisture content for long-term storage. Mangoes are usually sliced before drying to improve the efficiency of the drying process. Due to the considerable potential of solar energy in Sudan, solar drying of mango slices is a promising alternative for the preservation of perishable mango. However, it is necessary to know the moisture sorption isotherms in order to determine the optimum conditions for storage and for selecting the appropriate packing material for dried mango.

Moisture sorption isotherms are useful thermodynamic tools for determining the interaction of water and food substances. They also provide information for assessing food processing operations, such as drying, mixing, packing and storage. Such data can be used for selecting appropriate storage conditions and packing systems that optimise or maximise the retention of aroma, colour, texture, nutrients and biological stability (Labuza *et al*, 1985; Okos *et al.*, 1992 and Rizvi, 1995). Moisture sorption isotherms show the dependence of the water content on the water activity (a_w) of a food at a definite temperature and pressure. Dried foods are microbiologically stable when their activity is reduced below 0.6 (Hayes, 1987).

Numerous mathematical models for the description of the moisture sorption behaviour of foods are available in the literature. Some of these models are based on the theories of the mechanism of sorption, others are purely empirical or semi empirical (Rizvi, 1986). Van den Berg and Bruin (1981) have collected and classified 77 such equations. However, due to the complex composition and structure of food, the mathematical prediction of sorption behaviour is difficult. Furthermore, since irreversible changes can occur at high temperatures, experimental

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measurements at these temperatures are required.

Since the moisture sorption isotherms of food materials represent the integrated hygroscopic properties of various constituents and since the sorption properties may change as a result of chemical and physical interactions induced by heating or other pre-treatment methods, it is difficult to have a unique mathematical model whether theoretical or empirical that describes accurately the sorption isotherm over the whole range of water activities and for various types of foods (Chirife and Iglesias, 1978).

In the past, the well-known Brunauer-Emmett-Teller (BET) sorption isotherm was the model with the greatest application to water sorption isotherms of foods and foodstuffs (Labuza, 1968; Iglesias and Chirife, 1976), although it was known to hold only for a limited range of water activity. Two familiar constants are obtained from the BET model, namely the monolayer moisture content, *Mo*, and the energy constant, C. Despite the theoretical limitations of the BET adsorption analysis, the BET monolayer concept was found to be a reasonable guide with respect to various aspects of interest in dried foods (Karel, 1973; Iglesias and Chirife, 1981). The monolayer moisture content (*Mo*) is the moisture content at which the rate of quality loss is negligible (Labuza, 1984).

In more recent years, among the sorption models, the Guggenheim-Anderson-deBoer (GAB) equation has been applied most successfully to various foods (Timmermann *et al*., 2001). It has a reasonably small numbers of parameters (three) and adequately presents the experimental data in the range of water activity of most practical interest in foods (i.e., 0.05 to 0.90). The GAB model has been recommended by the European COST project on the physical properties of foods (Wolf *et al*., 1985) as being the fundamental equation for the characterisation of the water sorption of food materials. Moreover, the GAB model provides parameters with a physical meaning in terms of sorption isotherms and enables some temperature effects on the isotherms to be described by means of Arrhenius-type equations (Van den Berg, 1984). These parameters may be estimated using either the non-linear regression method or the polynomial regression method.

Both the BET and GAB isotherms are closely related as they follow from the same statistical model (Timmermann, 1989). Therefore, the main objectives of this study were (a) to investigate the moisture sorption isotherms of solar-dried mango slices, (b) to calculate the monolayer moisture content of both the BET and GAB models and (c) to evaluate the applicability of the GAB model for describing the moisture sorption isotherms of solar-dried mango slices.

Materials and Methods

Materials

Fully-ripened Kitchener variety mangoes (*Mangifera indica* L) were washed, manually peeled and transversely cut into 3-mm slices using a stainless steel knife. The mango slices were dried using a direct (cabinet-type) solar dryer to a final moisture content of approximately 5% d.b. in about 16 hours (2 days) in Khartoum (Sudan). The solar-dried samples were then packed in sealed plastic bags, wrapped with aluminium foil and stored in a dark place (a_w<0.5) at room temperature prior to transporting to Germany for the sorption isotherm experiments. The chemical prosperities of fresh mango (cv. Kitchener on a wet basis) were as follows: moisture content 81.4±0.15%; pH: 4.04; total sugar = 12.09% and reducing sugar = 3.37%.

Sorption isotherms

The sorption isotherms of the solar-dried mango slices were determined gravimetrically using the standard static gravimetric method standardised in the European COST project (Wolf *et al*., 1985). The method is based on the use of saturated salt solutions to maintain a fixed relative humidity which corresponds with the tissue's water activity. Seven salts were selected to give different water activities in the range of 0.111-0.813 (Table 1). The saturated salt solutions were used inside glass jars in this study. The water activities of the salt solutions at different temperatures were taken from Greenspan (1977) and Bell and Labuza (2000).

A portion of each saturated salt solution was transferred into separate glass jars, so that a depth of 1.5 cm covered the jar bottom. The salts used were all of reagent grade; however, crystalline salt was always present on the bottom of the jar. The solar-dried samples (2-4 g) were weighed in brass sieves

Table 1. Water activity exhibited by saturated salt solutions at different temperatures

Salt solution	*Water activity, decimal*			*References*
	Temperatures ˚C			
	20	30	40	
Lithium chloride (LiCl)	0.113	0.113	0.111	Greenspan (1977)
Magnesium chloride ($MgCl_2$)	0.331	0.324	0.316	Greenspan (1977)
Potassium carbonate (K_2CO_3)	0.432	0.432	0.4	Greenspan (1977)
Magnesium nitrate ($Mg(NO_3)_2$)	0.544	0.514	0.484	Greenspan(1977)
Sodium nitrite ($Na\ NO_2$)	0.676	0.643	0.614	Bell & Labuza (2000)
Sodium chloride (NaCl)	0.755	0.751	0.747	Greenspan (1977)
Ammonium sulphate $(NH_4)_2SO_4$	0.813	0.8.06	0.799	Greenspan (1977)

with an analytical sensitive balance (Sartorius, Goettingen, Germany) of 0.001 g accuracy. To avoid direct contact with the salt solution, the sieves were placed on tripods in the jars which were then tightly closed and placed in one of three temperature-controlled incubators at the temperatures of 20, 30 and 40˚C. These temperatures were chosen as they reflect the conditions found for storing mango slices in tropical countries. The solar-dried samples were weighed at two-day intervals using the analytical balance. The equilibrium was reached when the sample weight difference between three consecutive readings was equal or less than the balance accuracy of 0.001 (Saravacos *et al*., 1986; Lim *et al*., 1995). Each experiment was performed in duplicate.

The total time required for removal, weighing and putting back in the jars was minimised to less than 1 minute (≈ 30 s) to prevent, as much as possible, atmospheric moisture sorption or desorption during weighing (McMinn and Magee, 1997; Tsami *et al*., 1990). The time required for the solar-dried mango slices to reached equilibrium varied from 4-5 weeks depending on the water activity and the temperature.

The equilibrium moisture content of the samples was determined after each sorption experiment using a precision air-oven method at a temperature of 135˚C for 2 hours until constant weight was achieved, according to the standard method of AOAC (2000). The mean values of two replicates were obtained for each experiment. To establish the moisture sorption isotherms, the equilibrium moisture contents were plotted against water activity at constant temperature.

Development of mathematical model for sorption isotherms

The experimental equilibrium moisture content (EMC) data were processed using Microsoft Excel (2007) software and analysed using the linear trend method on transform linearised equations for the BET parameters; whereas the polynomial regression method was used on the transformed binomial equation for the GAB parameters.

Calculation of the BET parameters

The two-parameter BET model can be expressed as follows (Brunauer, Emmett and Teller, 1938):

$$M = \frac{MoCa_w}{(1-a_w)(1+(C-1)a_w} \quad (1)$$

Where:
M = equilibrium moisture content, %, d.b.
Mo = BET monolayer moisture content, %, d.b.
a_w = water activity, decimal
C = BET constant related to the net heat of sorption
To obtain the two characteristic constants, *Mo* and C, the BET equation (1) was linearised as follows (Timmermann, 2003):

$$\frac{a_w}{(1-a_w)M} = \frac{1}{CMo} + \frac{C-1}{CMo}a_w \quad (2)$$

Which can easily be transformed to a linear equation with the following form

$$\frac{a_w}{(1-a_w)M} = Aa_w + b \quad (3)$$

Where: $A = \frac{C-1}{CMo}$

$b = \frac{1}{CMo}$

The BET parameters were obtained by plotting $a_w / (1-a_w) M$ versus aw which would usually give a linear part at low activities up to 0.55 a_w. From the slope (A) and the intercept (b), the BET parameters were determined using the following relationships:

$$Mo = \frac{1}{(A+b)} \quad (4)$$

$$C = \frac{A+b}{b} \quad (5)$$

Calculation of the GAB parameters

The GAB equation is given by the following equation (Rizvi, 1986)

$$M = \frac{MoCKa_w}{(1-Ka_w)(1-Ka_w+CKa_w)} \quad (6)$$

Where:
M = equilibrium moisture content, %, d.b.
Mo = GAB monolayer moisture content
C = constant related to the monolayer heat of sorption (dimensionless)
K = factor related to the heat of sorption of the multilayer (dimensionless)

This equation has a similar form to BET, but has an extra constant *K*. BET is actually a special case of GAB, where *K* = 1. The GAB parameters were determined following the method of the transformed form of the GAB isotherm (Timmermann *et al*., 2001).

The GAB model can be rearranged to a polynomial equation as follows:

$$\frac{a_w}{M} = \frac{K}{Mo(1/C-1)}a_w^2 + \frac{C-2}{MoC}a_w + \frac{1}{MoCK} \quad (7)$$

After introducing the three parameters $\alpha = \frac{K}{Mo(1/C-1)}, \beta = \frac{C-2}{MoC}$, and $\varepsilon = \frac{1}{MoCk}$, then the GAB equation takes the form:

$$\frac{a_w}{M} = \alpha a_w^{\ 2} + \beta a_w + \varepsilon \quad (8)$$

A polynomial direct nonlinear regression method of aw/M versus aw was carried out using Microsoft Excel (2007) software in order to determine the values of the coefficient of the quadratic term α, the linear term coefficient β and the constant ε. Then the GAB parameters were calculated as follows:

$$K = \frac{f^{1/2} - \beta}{2\varepsilon} \quad (9)$$

$$Mo = \frac{1}{(\beta + 2K\varepsilon)} \quad (10)$$

$$C = 2 + \frac{\beta}{K\varepsilon} \quad (11)$$

Where: $f \equiv \beta^2 - 4\alpha\varepsilon$

The temperature dependency of the GAB parameters was given by the following Arrhenius-type equations:

$$C(T) = Co\exp\left(\frac{\Delta H_c}{RT_{abs}}\right) \quad (12)$$

$$K = Ko\exp\left(\frac{\Delta H_k}{RT_{abs}}\right) \quad (13)$$

$$Mo = Mo'\exp\left(\frac{-E_a}{RT_{abs}}\right) \quad (14)$$

Where:

Co, Ko and Mo′ are adjustable constants for the temperature effect;

ΔH_c = difference in enthalpy between monolayer and multilayer sorption (kJ/kmol)

ΔH_k = difference between the heats of condensation of water and the heat of sorption of the multilayer (kJ/kmol)

R = universal gas constant, 8.314J/mol K

T_{abs} = absolute temperature, K

E_a = activation energy, KJ/mol

For the monolayer moisture content, an Arrhenius–type equation was calculated using a linearised form of Equation (14):

$$\ln(Mo) = \ln(Mo') - E_a/RT_{abs} \quad (15)$$

The coefficient of Equation (15) can be easily obtained by plotting on an ln (*Mo*) versus $1/T_{abs}$ diagram. The E_a and Mo coefficient can subsequently be related to the temperature by applying the regression technique.

E_a was calculated by plotting ln(*Mo*) versus the reciprocal of the absolute temperature. The slope of the curves found by applying the linear regression yields the coefficient E_a/R, while the intercept is equal to ln (*Mo*`). The other GAB parameters' coefficients were determined following the same procedure.

The suitability of model fit was determined in terms of mean relative percentage deviation (P %) value (Boquet *et al.,* 1978; Lomauro *et al.*, 1985), where the model is considered to be suitable if P is < 10% (Lomauro *et al*., 1985; Wang & Brennan, 1991).

$$P(\%) = \frac{100}{N}\sum_{i=1}^{N}\frac{M\exp,i - Mpred,i}{M\exp,i} \quad (16)$$

Where:

$Mexp,_i$ = experimental EMC value

$Mpred,_i$ = predicted EMC value

N = number of experimental observations

also coefficients of determinations (R^2) were determined for both the non-linear and linear regressions.

Results and Discussion

Moisture sorption characteristics of solar-dried mango slices

Due to the occurrence of mould growth at water activities (a_w) >0.755 for the temperatures 30 and 40°C, the EMC data at these water activities were excluded. Sorption isotherms of solar–dried mango slices at the temperatures of 20, 30 and 40ºC in the aw range of 0.111 to 0.813 are shown in Figure 1. The sorption isotherms follow the characteristic shape of high-sugar foods according to the BET classification [i.e. type III (J-shaped], with the EMC increasing sharply at high water activities. These findings are in agreement with those of Pott *et al*. (2005) for the adsorption characteristics of dried Kent variety mango slices at 20°C, Telis-Romero *et al*. (2005) for the adsorption isotherms of dried Haden variety mango pulp at different temperatures (30°C-70°C),and Falade and Aworh (2004) for adsorption isotherms of osmo-oven dried African mango. From the figure it is clear that, at water activity values lower than 0.6-0.75, the EMCs of the mango slices are higher at low temperatures than at high temperatures. These results are in agreement with the general characteristics of food isotherms and physical adsorption which predicts that the quantity of sorbed water at a given water activity increases as temperature decreases. However, for the water activity range (a_w = 0.747-0.755) used in the present study, the EMCs are higher at higher temperatures, as usually occurs in high sugar foods. This resulted in an intersection of the moisture isotherms of the different temperatures at a_w = 0.75 (Figure 1). This intersection point shows that there is a tendency for the reverse temperature effect (the EMC values decrease with increasing temperature at constant water activity) to occur. This is suggested

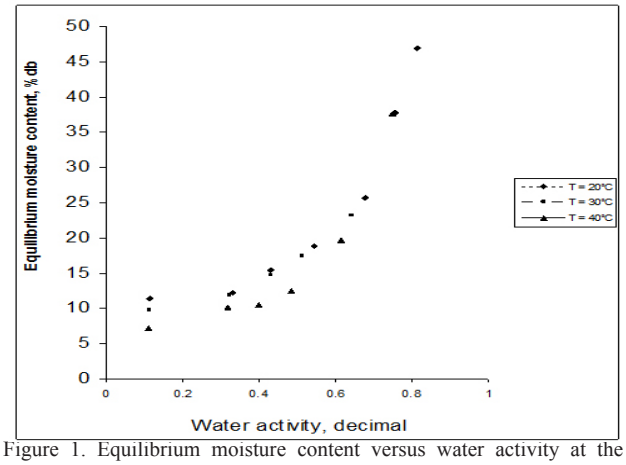

Figure 1. Equilibrium moisture content versus water activity at the different drying temperatures (20, 30 and 40 °C)

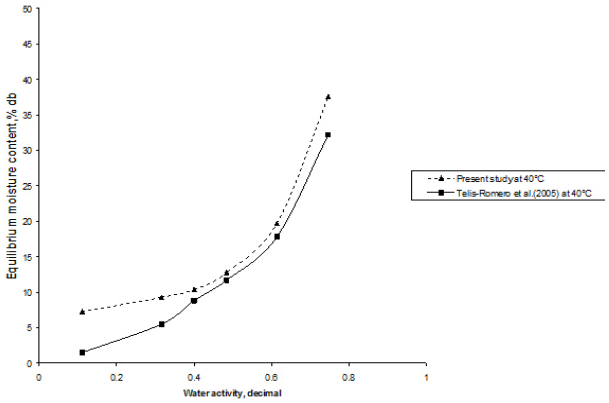

Figure 2. Comparison of sorption isotherms of solar-dried mango slices at 40°C with published data

Table 2. Estimated parameters and P (%) for the BET and GAB models of adsorption isotherms of solar-dried mango slices at different temperatures

Model	Temperature (°C)		
	20	30	40
BET:			
Mo	8.425	8.389	6.41
C	74.187	397.33	130.08
R^2	0.996	0.997	0.993
P (%)	8.13	0.67	4.12
GAB:			
Mo	7.98	7.827	5.84
C	45.23	87.24	60.69
K	1.03	1.05	1.13
R^2	0.99	0.99	0.97
P (%)	*8.17*	*5.11*	5.74

C and K = constants in sorption isotherms models; *Mo* = monolayer moisture content (dry basis), P (%) = mean relative percentage deviation, R^2 = coefficient of determination, BET: Brunauer-Emmett–Teller and GAB: Guggenheim-Anderson-de Boer.

also by the fact that mould grew at 30 and 40°C for water activities of 0.806 and 0.799, respectively, but no mould grew at 20°C even when the water activity was 0.813, which implies that the EMC at 20°C is less than EMCs at 30 and 40°C.

Telis-Romero *et al*. (2005) observed a tendency for this crossing-over to occur at higher water activities in Haden variety mango. Such isotherm crossing points have also been reported for raisins (Saravacos *et al*., 1986), figs, prunes and apricots (Maroulis *et al*., 1988; Tsami *et al*., 1990). The reverse temperature effect is due to the dissolution of sugars or increased solubility of sugars and thus more water was being held at higher temperatures as the result of an increase in the number of adsorption sites upon the breakage of the crystalline structures of sugar (Ayranci *et al*., 1990). Falade and Aworh (2004) reported an intersection of isotherms of osmo-oven dried African mango above 0.8 water activity in their study. The intersection point of isotherms depends on the type of sugar present, the sugar size distribution and the composition of the food (Weisser, 1985). Since this process is known to be endothermic, more sugar was being dissolved and thus more water was being held by the mango slices at the higher temperatures.

The moisture sorption isotherms of the solar-dried mango at 40°C from the present investigation are compared in Figure 2 with the data of Telis-Romero *et al*. (2005) at the same temperature for the Haden variety of mango. There is an excellent agreement between the sorption characteristics of the two different varieties. However, there is variation in EMC at the lower water activities, which may be due to the variety differences or differences in drying methods.

Curve fitting of sorption isotherms of solar-dried mango slices

The estimated parameters of the GAB and BET models are presented in Table 2. The monolayer moisture content (*Mo*) is an important parameter in food deterioration studies. The Mo value decreases with increasing temperature. These results agree well with those of Kumar and Mishra (2006); Labuza (1968); Hossain *et al*. (2001) and Rahman and Labuza (1999).

The decrease in *Mo* values may be due to a reduction in the total number of active sites for water binding as a result of physical and/or chemical changes in the product induced by temperature (Iglesias and Chirife, 1976; Mazza and Le Maguer, 1978). The *Mo* values in this investigation are in agreement with those stated by Van den Berg and Bruin (1984), which indicated that the monolayer moisture content in the food was lower than 10 g/100 g dry matter. As can be seen from Table 2, the GAB monolayer values are smaller than their BET counterparts.

The C values for the BET model are greater than for GAB. However, as can be seen in Table 2, there is no clear correlation between the C parameter and temperature. The values for constant K in the GAB model vary between 1.02 and 1.13 (slightly greater than 1). The K values increase with increasing

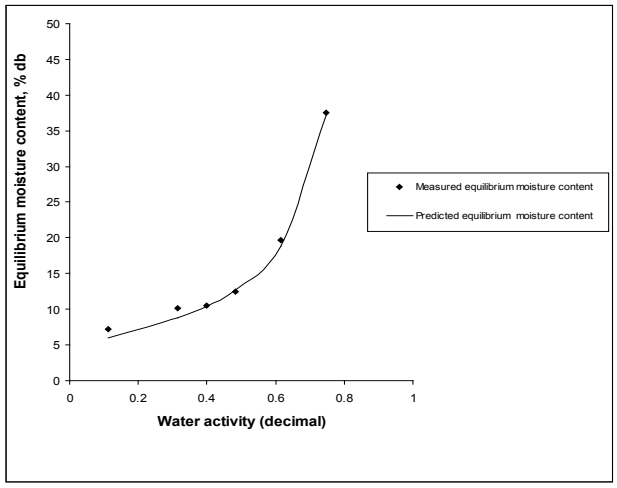

Figure 3. Curve fitting of equilibrium moisture content given by the GAB model to the experimental values at 40°C

Table 3. Temperature-dependent regression equation for GAB model parameters of solar-dried mango slices

GAB Model Parameters	Regression equation	R^2
Mo	$-0.0087T^2+0.4082T+3.384$	1
C	$-0.3339T^2+20.721T-233.84$	1
K	$0.0003T^2-0.0095T+1.11$	1

C and K = constants in sorption isotherms GAB model, Mo = monolayer moisture content (% d.b.)

Table 4. GAB parameter coefficients' values calculated by non-linear regression of LnGAB parameters and reciprocal of absolute temperatures

GAB parameter constants	Value
Mo'	*0.0423 (%, d.b.)*
E_a	12.9kJ/mol
Co	5.401
ΔH_c	5.827kJ/mol
Ko	*3.303*
ΔH_k	-2.8kJ/mol

Mo', Co, Ko, ΔH_c ΔH_k and E_a are fitting constants, which can be obtained using non-linear regression.

temperature. These results are in agreement with Kumar and Mishra (2006).

The experimental data were fitted to the isotherms models. The parameter constants and coefficients of determination R^2 along with mean percentage deviation modulus P (%) are also shown in Table 2. The BET equation is known to hold for water activities of up to about 0.55 (Chirife and Iglesias, 1978). In this low water activity region, the fit of the sorption data to the BET equation was satisfactory. In contrast, the GAB model fitted well throughout the whole range of water activity, giving a low P (%) which varied between 5.11 and 8.17. As this is <10% , it is an indication of good fit according to both Wang & Brennan (1991) and Lomauro *et al.* (1985).

Figure 3 shows the experimental moisture contents and predicted moisture contents given by the GAB model plotted against water activity at 40ºC. It can be seen that the GAB model gives a very close estimate of EMC; such good fits were also found for 20 and 30ºC. Consequently, the GAB model can be recommended for characterising the adsorption behaviour of solar-dried mango slices in the temperature range of 20-40°C and water activity range of 0.111 to 0.813. This is in agreement with a number of authors who used the GAB model on the sorption isotherms and EMC data of various foods [experimental adsorption isotherms at 25°C (Spiess and Wolf 1987); dried fruits (Maroulis *et al*., 1988); mango pulp freeze-dried in the temperature range 15-35°C and aw values from 0.11– 0.89 (Rangel–Marrón *et al*. 2011); guar grain and guar gum splits (Vishwakarma et al., 2011].

The temperature dependence of the GAB model constants (Mo, C and K) and the regression equation for each of these parameters, along with their correlation coefficient (R^2), are presented in Table 3. Temperature dependency of the GAB parameters was correlated with temperature. A polynomial relationship with the highest value of coefficient of determination (R^2 equals 1) was set up for each of the three GAB parameters as shown in Table 3. These equations can, therefore, be used to simulate moisture sorption isotherms of mango slices at any temperature, but experimental comparison should be made in order to generalise the GAB model.

The temperature dependency of the GAB parameters calculated by nonlinear regression is shown in Table 4. The coefficients of the GAB parameters were found to be 0.0423 % d.b. for Mo' and 12.9kJ/mol K for the activation energy. The values ΔH_c = 5.827kJ/mol and ΔH_k= -2.8kJ/mol represent the mean values of the heats of sorption of water on the dried fruit. ΔH_c is the difference in enthalpy between monolayer and multilayer sorption, which is expected to have a large positive value due to the strong exothermic interaction of water vapour with the primary sorption sites of the mango slices. It should be noted that at higher moisture contents ΔH_k may reach a slightly positive value due to endothermic dissolution of fruit sugars (Saravacos *et al*., 1986).

Conclusions

The following conclusions can be drawn from this study:

- The adsorption characteristics of solar-dried mango slices have the typical shape of high-sugar foods according to the BET classification [i.e., Type-III (J-shaped)].
- Intersection of isotherms occurred at aw=0.75. The tendency for a crossing over of the curves at this point

indicates the presence of the reverse temperature effect.
- The GAB model describes the sorption data well over the range of temperatures and water activities studied in this investigation as the GAB model could be satisfactorily used to predict the moisture sorption isotherms of solar-dried mango slices over 20-40ºC and for water activity between 0.111-0.813.
- The sorption data were also adequately expressed by the BET equation in the water activity range of 011-0.5.
- The monolayer moisture content decreased with increasing temperature and was found to be less than 10% (d.b.) for both models.

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