

The prediction of moisture adsorption isotherm for sucrose powder in Côte d'Ivoire

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Abstract:

Moisture sorption isotherms play an essential role in preservation and storage of dehydrated food. To study the behavior of sucrose, its moisture adsorption isotherm was investigated using the static gravimetric method at laboratory temperature ($29 \pm 1^\circ\text{C}$) in a water activity range (a_w) of 0.07-0.97. The results showed that the equilibrium moisture content of sucrose increase substantially above $a_w = 0.6$. The moisture adsorption isotherm was sigmoid in shape, showing a type III BET isotherm. The data obtained were fitted to several models including two-parameter (BET, Hasley, Kuhn and Oswin), three parameter (GAB). A non-linear least square regression analysis was used to evaluate the models constants. The GAB model best fitted the experimental data in the wide range of water activity. The best fitted equation provide a sound basis for future work on the drying and storage of sucrose. The accurate moisture content and the stability profile of sucrose were determined. The results of determination show that sucrose powder was stable below 7.7% RH at 29°C . The content of water adsorbed in the monomolecular layer was calculated (GAB $X_m=0.950$ g H₂O/100 g solids; BET $X_m= 1.072$ g H₂O/100 g solids).

Keywords: Equilibrium moisture content, modelling, sucrose powder

1. Introduction

Carbohydrates play a major role in biological and food systems. Sucrose is possibly the most important sugar for food manufacturing. It is a disaccharide that can be hydrolyzed to glucose (an aldehyde group, aldoses) and fructose (ketone group, ketoses) [1]. Both monosaccharides are also widely used as sweeteners in foodstuff. In general, sugars are hydrophilic to different degrees, depending on their structures which govern their plasticizing and moisturizing properties. The water sorption rate by sugars depends on their purity and homogeneity of crystal structure.

Sucrose is a disaccharide composed of 1 α -D-glucopyranosyl unit and 1 β -D-fructofuranosyl unit joined by a glycosidic linkage [2]. Sucrose can exist in both the crystalline and amorphous states. The crystalline state is an equilibrium, lowest energy and entropy, solid state that exhibits an orderly molecular arrangement of molecules, with a repeating pattern extending in all 3 spatial dimensions [3]. Sucrose is widely used in the food

industry because of the sweet taste and/or functional properties it contributes to food systems. Sucrose is one of the most common ingredients in foods and is important to the structure of many foods. For example, sucrose can either recrystallize or form an amorphous glass after baking, which affects the texture of baked products, such as cookies and biscuits [4].

Moisture sorption isotherms describe the relationship between the equilibrium moisture content and the water activity at constant temperatures and pressures. For food materials these isotherms give information about the sorption mechanism and the interaction of food biopolymers with water [5].

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 theories of the mechanism of sorption, others have been purely empirical, or semi-empirical [6]; [7]. Van den Berg and Bruin [8] have collected and classified 77 such equations. However due to complex composition and structure of foods,

mathematical prediction of sorption behaviour is difficult. Since the moisture sorption isotherms of food materials represent the integrated hygroscopic properties of various constituents and 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 in the whole range of water activity and for various types of foods [9]. A detailed research of the literature showed that moisture sorption isotherms of foods can be described by more than one sorption model [10]. The criteria used to select the most appropriate sorption model were the degree of fit to the experimental data and the simplicity of the model.

The knowledge of the water sorption characteristics is essential in regard to stability and acceptability of food products, dehydration operation modelling, process equipment design, and evaluation of moisture changes during storage or selection of packaging materials [11]; [12]. Food systems typically exhibit Type II and III isotherms according to the BET classification [13]. Experimental water sorption isotherms are based on the determination of three properties: equilibrium moisture content, water activity and temperature (commonly at atmospheric pressure) [14].

In the literature, there are a large number of available empirical and semi-empirical equations to establish mathematical relationships among these three variables, which are usually evaluated by the experimental data fitting [15]. Another possibility is the prediction of water activity, at each moisture content and temperature, from fundamental laws and empirical expressions using prediction algorithms based on chemical composition of foodstuff [16]; [17]. Estimating the hygroscopic properties of sucrose at 29°C could be valuable information in order to generate prediction models which will allow obtaining water adsorption isotherm, in a wide temperature range of many food materials, taking into account their chemical composition [17].

This type of research is considered of great interest, since the existent literature shows an important discrepancy between different experimental water sorption isotherms for certain products. Also, the hygroscopic properties of many food products are not determined and the experiments to obtain the corresponding water adsorption isotherms are time-consuming (usually several weeks). In these cases, the estimation of hygroscopic properties is interesting and the sorption

characteristics of the main components (like sugars, starch, protein, fats, etc) should be well established. Information on adsorption isotherm of sucrose is scarce and limited to specific temperatures [18].

Therefore, the main objectives of this work are to determine the experimental equilibrium moisture content of sucrose as well as to select the appropriate mathematical model for predicting the water adsorption isotherms at 29°C.

2. Material Et Methods

2.1. Material

Sucrose used in the present study was of a commercial grade. A kilogram of well packaged sucrose was purchased at a super market in Abidjan, Côte d'Ivoire. The experiment took place at the technology laboratory of the National Center for Agronomic Research, located in Bingerville, Côte d'Ivoire.

2.2. Methods

2.2.1. Experimental procedure

The methodology used to obtain the equilibrium data was based on the dynamic method with saturated salt solutions. The salts were chosen, to obtain a large range of water activity. The equilibrium moisture contents of coffee were determined by a gravimetric technique, in which the weigh changes were monitored continuously within a dynamic system of thermally stabilized. Although this method requires a long time for the hygroscopic equilibrium to be obtained, it has the advantage of presenting a more restricted domain of moisture content variation [19].

Samples were placed into desiccators with six different saturated salt solutions (KOH, LiCl, MgCl₂, NaCl, KCl and K₂SO₄) at laboratory temperature (29°±1°C) in order to generate controlled humidity environment in a water activity range between 0.07 and 0.97 (Table 1) [20].

Table 1: Water activity values of the saturated salt solution at laboratory temperature (29°C)

Saturated salt solutions	Temperature (29°C)
KOH	0.077
LiCl	0.11
MgCl ₂	0.32
NaCl	0.73
KCl	0.88
K ₂ SO ₄	0.97

Sucrose powder samples ($2 \pm 0.001\text{g}$) were placed in previously weighed aluminium dishes and dried at 45°C in an air-circulated oven over silica gel for 3 days. The samples were subsequently kept in desiccators over sulphuric acid solutions of known relative humidity.

A little quantity of thymol was placed inside the desiccators with high relative humidity ($a_w > 0.7$) to avoid the microbial degradation during storage. Samples were equilibrated for approximately one month, as evidenced by constant values (0.0003) of two consecutive weight readings. After reaching equilibrium, the dry weight was determined after drying in a vacuum oven at 70°C and the equilibrium moisture content was evaluated as an average of three measurements. The total time for removal, weighing and putting back the samples in the desiccators was about 30 s. This minimized the degree of atmospheric moisture sorption during weighing.

2.2.2. Modelling equations

For adjusting of the experimental data from adsorption isotherms of sucrose powder, mathematical models of BET, Kuhn, Hasley, GAB and Oswin were used, represented respectively by the equations in Table 2.

Table 2: Isotherm equations for experimental data fitting

Model	Mathematical expression	a_w range
BET (Brunauer <i>et al.</i> , 1938)	$X_{eq} = \frac{X_m C a_w}{[(1 - a_w)(1 + (C - 1)a_w)]}$	(1) $a_w < 0.50$
GAB (Van den Ben and Bruin, 1981)	$X_{eq} = \frac{X_m C K a_w}{[(1 - K a_w)(1 + C G K a_w - K a_w)]}$	(2) $0.05 < a_w < 0.95$
Hasley (1948)	$X_{eq} = \left(\frac{A}{1 + n a_w}\right)^{\frac{1}{n}}$	(3) $0.05 < a_w < 0.80$
Kuhn (Labuza, Mizrahi and Kasel, 1972)	$X_{eq} = \frac{A}{1 + n a_w + B}$	(4) $a_w < 0.5$

Oswin (1946)	$X_{eq} = A \left[\frac{a_w}{(1 - a_w)} \right]^B$	(5) $0.05 < a_w < 0.90$
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These equations were chosen because they are most widely used to fit experimental sorption data of various food materials. The parameters of the sorption models were estimated from the experimental results using the nonlinear regression analysis (SPSS 9.0 for windows 1998) which minimises the residual sum of squares.

The quality of adjusting different models was evaluated through the best values of the determination coefficient (R^2) and the relative mean deviation (E%). When the MRD value is greater than 10%, the model shows a poor adequacy to describe the experimental behaviour. Also when the coefficient of determination is lower than 0.70, the model shouldn't be used to fit the experimental data [21]. These two parameters were calculated using the following equations:

$$R^2 = \frac{\sum (X_{eq,exp} - X_{eq,pre})^2}{\sum X_{eq,exp}^2 + \sum X_{eq,pre}^2} \quad (1)$$

$$MRD (\%) = \frac{100}{N} \sum_{i=1}^N \frac{|X_{eq,exp} - X_{eq,pre}|}{X_{eq,exp}} \quad (2)$$

Where N is the number of observations, $X_{eq,exp}$ and $X_{eq,pre}$ are the experimental and predicted values of the equilibrium moisture content, respectively.

In the GAB model, X_m is the moisture content corresponding to the formation of a monomolecular layer on the internal surface; G is a constant related to the heat of sorption of the first layer on primary sites and K is a factor correcting properties of the multi-layer molecules with respect to the bulk liquid.

3. Results And Discussion

2.3. Experimental adsorption isotherm

The adsorption isotherm showed an increase of moisture content with the water activity. The equilibrium moisture content of the adsorption isotherms increased slowly between a_w values 0.07 (1.20 g H₂O/100 g solids) and 0.75 (3.5 g H₂O/100 g solids), followed by a steep rise (32.2 g H₂O/100 g solids) (Figure 1). Consequently, the sorption isotherms obtained in this investigation are type III (J shape) isotherms, which are characteristic of products holding small amounts of water at lower a_w values and higher amounts of water at high RH levels [22]. Similar behaviour has been reported previously in different food products i.e. sugars,

apple, raisins, apricot, pineapple, beef [23] and pistachio powder [24] and Madeira cake [23]. A type III isotherm appears when the binding energy for the first layer is lower than the binding energy between water molecules [23]. Experimental data values present the same form as the values reported in earlier works for water adsorption isotherms of fructose at 25°C [25]. These results were also in well harmony with those previously reported for syrup powders [26] and with those found for food materials rich in sugars as sweet potato [27].

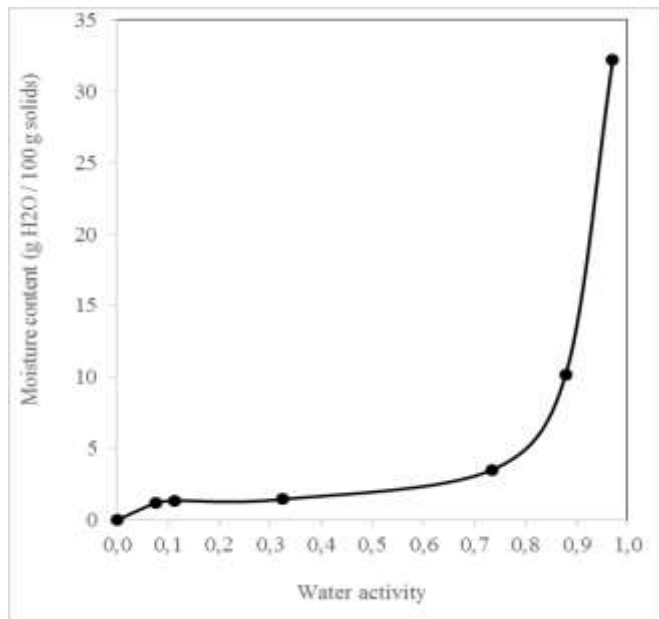


Figure 1: Adsorption isotherms of sucrose powder at 29°C

Foods with high levels of small, soluble molecules and small amounts of polymeric compounds may exhibit a Type III curve [28]. Many candies, including caramel, fudge, and nougat may have sorption isotherms that follow either Type III behavior. Hadjikinova *et al.* [29] found Type III sorption curves for sugar-free hard candies made with sorbitol and isomalt. For pure crystalline ingredients (e.g., sucrose), moisture is only able to interact by hydrogen bonding at the surface of the crystal [14] since the packing arrangement of the crystal lattice excludes foreign molecules such as water. Thus, moisture content remains low and nearly constant until *aw* is high enough to cause dissolution of the crystal surface at the deliquescent point [14]. Above this relative humidity, water is able to dissolve the crystal and the moisture content increases quickly.

2.4. Fitting of sorption models to experimental data

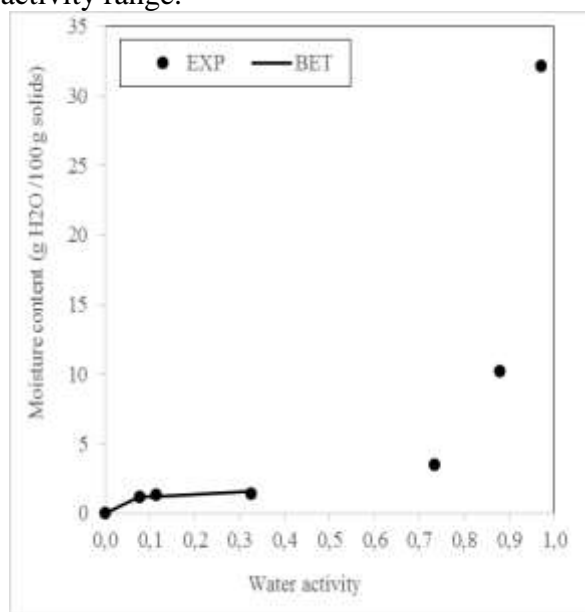
Table 3 contains the parameters obtained by BET, Kuhn, GAB, Oswin and Hasley models for sucrose powder. The parameters of the models applied to the experimental data of adsorption isotherm of sucrose powder (determination coefficients R^2 and the relative average deviations E%) were utilized as evaluation criteria for the representation of isotherms are shown in Table 3.

Table 3 : Estimated values of constants, correlation coefficient (R^2) and the mean relative percentage deviation modulus (MRD) obtained for the models

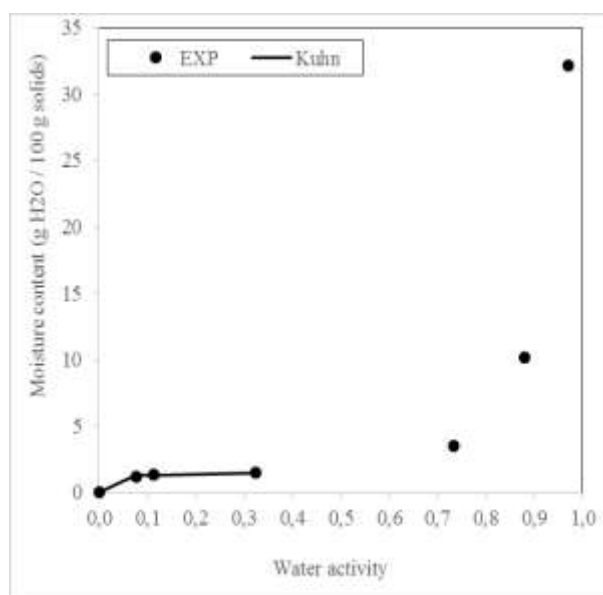
Model	Constants	Values
GAB	Xm	0.950
	C	21142.504
	K	1230.639
	R^2	0.997
	MRD (%)	10.8
BET	Xm	1.072
	C	8052703.825
	R^2	0.859
	MRD (%)	7.7
Kuhn	A	-0.407
	B	1.098
	R^2	0.872
	MRD (%)	3.4
Hasley	A	1.396
	B	1.036
	R^2	0.989
	MRD (%)	29.1
Oswin	A	2.060
	B	0.783
	R^2	0.998
	MRD (%)	33.4

According to the results shown in Table 3, two (BET and Kuhn) of the five models can be considered satisfactory for fitting that adsorption of sucrose powder (Figure 2 a and b), as they exhibited excellent values of R^2 and E%, respectively from 0.859 and 0.872; and from 7.7% and 3.4 %; these values are below the criteria recommended by Aguerre *et al.* [30], where E% less than 10 % indicates a reasonable representation of the models, and by Labuza *et al.* [31], in which the representation of isotherms is considered extremely good for E% less than 5%. These results were also in well harmony with those previously reported for grapes, apricots, apples and potatoes by Kaymak-

Ertekin and Gedik [5] and for fermented cocoa beans (*Theobroma cocoa*) by Akmel *et al.* [32]. It must be said that the Kuhn model was the best for describing the equilibrium moisture data within the water activity range.



(a)



(b)

Figure 2: Adsorption isotherms of sucrose powder predicted by: (a) Brunauer-Emmet-Teller (BET) model and (b) Kuhn model

It should be noted that the BET and Kuhn models are applicable only in the range of water activities from 0 to 0.5. Thus the GAB model ($R^2=0.997$ and $MRD=10.8\%$) was the most applicable model that covers the full range of water activities (Figure 3). This observation is similar to that obtained by other researchers, who studied sorption isotherms of various foodstuffs. So, GAB model is reported by several researchers in forecasting adsorption isotherms of various dehydrated foods,

particularly Ferreira and Pena [33] in obtaining adjustment of this model with R^2 of 0.9996 and 0.9991 for peach-palm powder, Silva *et al.* [34] which found R^2 values higher than 0.99 for yellow mombin powder, and Alam and Singh [35] who obtained the maximum R^2 for aonla flakes. In addition, we can note that the GAB model has been reported as the best fitting model in several products i.e. potato, carrot, tomato, onion and green pepper [36], yellow dent corn [37], several fruits, vegetables and meat products [10] and lemon peel [38].

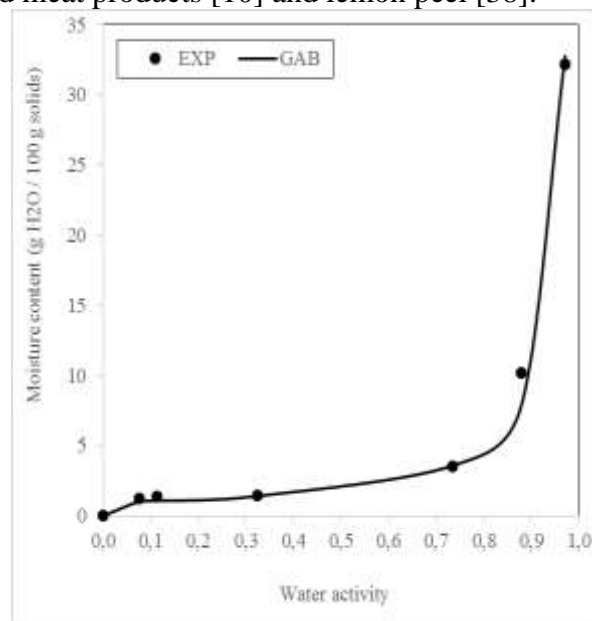


Figure 3: Adsorption isotherm of sucrose powder predicted by Guggenheim-Anderson-de Boer (GAB) model

Hasley model cannot quite accurately describes characteristics of sucrose powder (Figure 4), with the value of MRD (29.1%) definitely higher than 10% (Figure 2). In contrast, the results of Ayranci *et al.* [39], Yanniotis *et al.* [40] for grapes and McLaughlin and Magee (1998) for potatoes showed that this Hasley model described well the sorption curves of these different food products. Kaymak-Ertekin and Sultanoglu [41] concluded that the Hasley equation gave the best fit for peppers. Use of Halsey model is recommended for meat, milk products and vegetables [42]. This model was successfully applied for water sorption data of several nuts and oil seeds Lomauro *et al.* [10]; Iglesias *et al.* [43] and Chirife *et al.* [9] found that, Halsey model useful to explain reasonably well the sorption of dried figs, apricots. Halsey model was proposed an expression for condensation of multilayer's at relatively large distance from the surface.

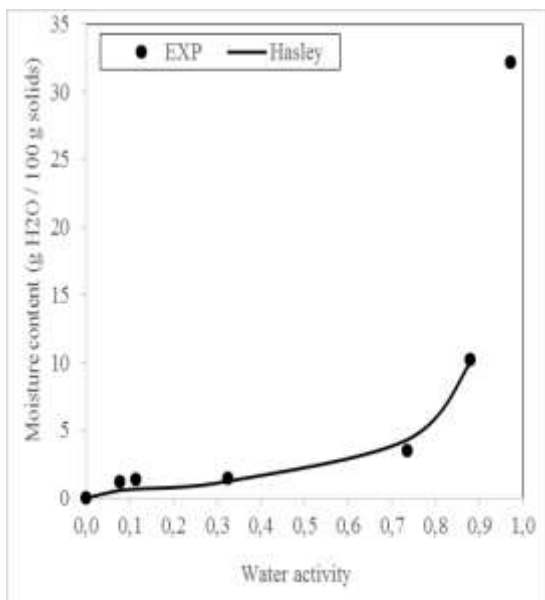


Figure 4: Adsorption isotherm of sucrose powder predicted by Hasley model

In this study, the Oswin model gave the least accurate predictions (Figure 5), giving an average MRD value of 33.4%. Wang and Brennan [44] and Al-Muhtaseb *et al.* [11] also reported that the Hasley model is inadequate for representing the sorption isotherms for various foods such as potato and starch powders. Conversely, several studies have shown that this model showed the best fitting for dehydrated garlic with R^2 ranging from 0.9148 to 0.9488 [45]; Basu *et al.* [46] studying pectin found values of R^2 between 0.82 and 0.97, while E% was less than 0.012. Lomauro *et al.* [10], quoted by Al-Muhtaseb *et al.* [23], reported the Oswin model just fitted 57% of the isotherms described for foods. For grugru palm powder in this study, the Oswin model presented R^2 value of 0.99818 and E% of 0.52. Jain *et al.* [47] stated that the Oswin model can be used to plan and evaluate drying, storage conditions and moisture amount to be removed during dehydration. Sopade *et al.* [48] reported that this model can also be used to predict the extension of hydration or dehydration required. Oswin model was found very suitable to describe sorption isotherms of proteins and starchy foods. Boquet *et al.* [42] and Lomauro *et al.* [10] concluded that this model fitted sorption data for a considerable number of nuts, spices, coffee, etc. This model is based on the mathematical series expansion for sigmoid shaped curves.

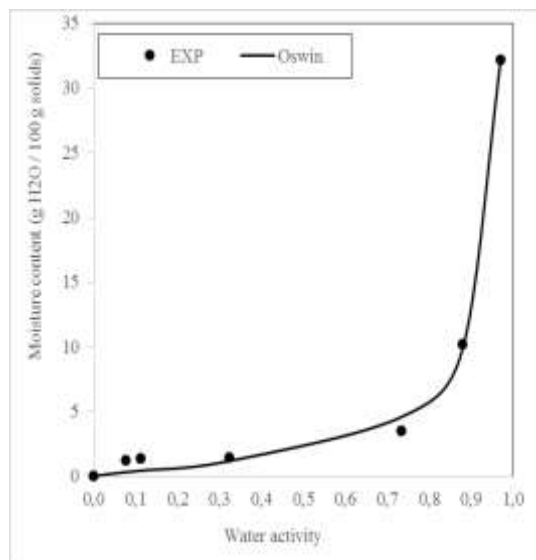


Figure 5: Adsorption isotherm of sucrose powder predicted by Oswin model

As in the BET equation, the monolayer capacity is also represented in one of the three GAB constant. The monolayer moisture content (X_m) obtained by the GAB model (0.950 g H₂O/100 g solids) was lower than that obtained by the BET model (1.072 g H₂O/100 g solids) (Table 3). The monolayer moisture content prediction of BET model is higher than the GAB model. It might be caused the BET model consider there is a multilayer on the monolayer one. This is similar to a study by Wariyah and Supriyadi [49] in rich calcium rice that has moisture content of 0.0721-0.0867 g H₂O/g solid. And also by Liendo-Cardenas *et al.* [50] in cereal products cassava with moisture content of 0.061-0.097 g H₂O/g solid in four different temperatures. In a study by Houssein [51] in pasta dates was getting moisture content of 0.084 g H₂O/g solid using a model GAB. It should be noted that these different water content are higher than those obtained in this study.

The moisture values in the monolayer (X_m) is a critical parameter as it represents the moisture content at which the rate of any associated reaction will be negligible due to the strong binding of water to the surface. An increase in aw equivalent to an increase of X_m by 0.1 units decreases the shelf life of a food product by a factor of 2-3 [52]. It is therefore an important quality parameter with regards to the designing optimal storage conditions for food products [53]. Therefore the X_m values reported in this study represent the optimal moisture content for the storage of sucrose powder.

These moisture values in the monolayer (X_m) parameter also characterize the moisture content under which food is stable, because at lower values of X_m biochemical changes can be observed lipid oxidation [54]. Thus, the values of X_m for sucrose

show low moisture content in the monolayer, hence lipid oxidation can occur during storage. So to prevent and minimize such oxidative processes, packaging should be impervious to air.

The water activity value corresponding to the two moisture values in the monolayer (GAB $X_m = 0.950$ g H₂O/100g solids and BET $X_m = 1.072$ g H₂O/100g solids) was 0.077. We recommend this value for sucrose powder as optimal storage.

As can be seen in Table 3, the estimated value of the BET constant C (related to thermal effects) was higher than those for GAB. The C values are much higher than K indicating that the heat of sorption of the first layer is greater than that of the multilayers. The energy constant C ($C_{GAB} = 21142.504$; $C_{BET} = 8052703.825$) indicates the difference between enthalpy of vaporisation from the monolayer and enthalpy of vaporisation for liquid adsorbent. The value of the C parameter is, according to Lewicki (1997), an indicator of appropriateness for choosing the GAB model to describe empirical data. It has been shown that the GAB model describes well sigmoidal type isotherms when $5.67 < C < \infty$.

The K parameter ($K_{GAB} = 1230.639$) is used for adjusting properties of molecules located in the adsorption monolayer as compared to the liquid phase. The value of the K parameter also indicates the scope of application of the GAB equation, and diversifies monomolecular ($K \leq 0.5$) and multilayer adsorption ($K > 0.5$) [55].

4. Conclusions

The moisture adsorption of sucrose powder was successfully generated by standard static gravimetric method using different saturated salt solutions at the laboratory temperature. The adsorption isotherm resulted in a shape of type III according to BET classification.

The GAB was the best model to predict the adsorption equilibrium moisture content of sucrose powder for a wide range of water activity (0.07-0.97). However, Kuhn and BET equations correlated the data reasonably well in the range of water activity 0.07-0.5. The GAB model may be utilized in predicting the equilibrium moisture content of sucrose powder at 29°C and relative humidity. This study will help in designing packaging systems of an important food product like sucrose so that it can be stored for a longer period for preparations of value added products.

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