The prediction of moisture adsorption isotherm for commercial sodium bicarbonate powder

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Abstract— The quality of preserved sodium bicarbonate powder is largely dependent on its moisture content, moisture migration and moisture uptake of the sodium bicarbonate during storage. Therefore, the water activity level, which corresponds to a range of equilibrium moisture contents, must be determined by, for example, the use of moisture isotherms. Sorption isotherms are therefore of great importance in post-harvest procedures, especially for predicting drying and storage, which help to establish the final moisture content of the product under certain environmental condition. Moisture sorption isotherm of sodium bicarbonate powder was determined at laboratory condition (29 ± 1°C) and relative humidity (7-97%), using the standard static gravimetric method. Adsorption isotherm of sodium bicarbonate was type III following Brunauer's classification. Equilibrium moisture content data were correlated by several equations including two-parameter equations (BET, Kuhn, Hasley, and Oswin), three parameter equation (GAB); and the GAB equation presented the best adjustment for the data over the range of water activities investigated, according to statistical procedures. However, results demonstrated that the BET and Kuhn equations described the sodium bicarbonate adsorption isotherm much better than the GAB equation for the range of water activity ranging from 0.07 to 0.5. The content of water adsorbed in the monomolecular layer was calculated (GAB_{Xm} = 4.702 g H2O/100 g solids; BET_{Xm} = 7.442 g H2O/100 g solids).

Index Terms— Equilibrium moisture content, modelling, sodium bicarbonate, powder.

1 Introduction

For many years, sodium bicarboate has been the primary source of carbonate used by the baking industry. Its stability as powder, pleasant flavor, and good functionality when used in a balanced leavening blend has remained the primary choice for bakers. It is also extremely versatile and is available in many different grades that are suitable for a wide variety applications [1].

Sodium bicarbonate is also an important component in various pharmaceutical solid and liquid dosage forms. Because of its widespread use, the stability of sodium bicarbonate in solid state, both as a raw material and as a formulation component, is of high interest to the scientists [1]. When sodium bicarbonate is stored as a powder, it degrades over time to carbon dioxide and sodium carbonate after sorption of moisture at lower temperature, or degrades directly to carbon dioxide and sodium carbonate without sorption of moisture at elevated temperature [2]. Therefore, it is critical to maintain appropriate temperature and relative humidity during the storage of the raw material and finished product as well as during manufacturing.

Several studies regarding the thermal decomposition of sodium bicarbonate under various conditions have been reported [3]. The rate of sodium bicarbonate decomposition to sodium carbonate is dictated by the moisture sorption kinetics at given temperature and humidity conditions.

The presence os small amounts of moisture in sodium bicarbonate inhibits the flowability of the product and promotes lump formation during storage [4]. The occurence of these undesirable effects has created a need for an accurate and sensitive method for measuring the moisture content of commercial sodium bicarbonate. Moisture content of powder is therefore very important regarding its shelf life, lower the powder moisture, the better its storage stability. The deterioration of baking quality will also be lower which can be credited to retarded respiration and activity of microorganisms at lower moisture content [5].

In climate like Côte d'Ivoire, the shelf life of product a serious problem and due to weather conditions, it is inevitable to explore proper moisture content to over come existing dilemma.

Moisture sorption characteristics provide the necessary information regarding quality, stability and shelf life during packaging and storage of food powders. Hygroscopic nature of sodium biacrbonate causes for caking and follow deterioration by oxidation. The moisture content of the powder as a hygroscopic material exerts a strong influence on its quality and technological properties [6]; [7].

A moisture sorption isotherm explains the relationship between the water activity (aw) and the equilibrium moisture content for a food product at a constant pressure and temperature. The knowledge and understanding of moisture sorption isotherms for food products is of great importance in design and optimization of processing as for instance in drying, for assessing packaging problems, for modeling moisture changes which occur during drying, for predicting shelf life stability, for ingredient mixing predictions etc. [8] ; [9]. Representation of sorption data with best fit sorption model could be used as a tool for achieving these designs.

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 [10]; [11]. Van den Berg and Bruin [12] have collected and classified 77 such equations. However due to complex composition and structure of foods, mathematical prediction of sorption behaviour is difficult [13].

A knowledge of the moisture content-relative humidity relationships of a food products helps in understanding the nature of the physico-chemical and biological deterioration that occurs in the product at various water activities and on the design of suitable packages which can provide protection against moisture interchange [14].

The present study was carried out to extend the shelf life of sodium bicarbonate by determining the proper moisture content suitable for safe storage. The broad objective of this study was to determine the moisture adsorption characteristics of sodium bicarbonate. Specific objectives were to evaluate the moisture adsorption of sodium bicarbonate at laboratory temperature (29 °C), to evaluate the goodness of fit of five sorption models and to obtain the model that best fits the sorption data.

2 MATERIAL AND METHODS

2.1 Material

Sodium bicarbonate powder used in the present study was of a commercial grade. A kilogram of well packaged sodium bicarbonate 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 Experimental procedure

The equilibrium moisture content was determined by standard gravimetric method by exposing the sodium bicarbonate powder to constant relative humidity environment created by saturated solution of a particular salt at laboratory temperature (29±1°C) [15]. Six different salts viz., KOH, LiCl, MgCl2, NaCl, KCl and K2SO4 were used to maintain respective water activity (aw) inside separate vacuum desiccators in the range of 0.077 to 0.97 [16]. Table 1 show the standard value of water activities given for the six salts. After taking the tare weight of weighing dish approximately 2 g of sodium bicarbonate powder weighed for each container. The samples were subsequently kept in dessicators.

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
MgCl2	0.32
NaCl	0.73
KCl	0.88
K_2SO_4	0.97

To prevent mould growth approximately 5 mg potassium sorbet was added to each sample. At predetermined time intervals (12 h), each sample dish was removed and covered with aluminum foil to prevent moisture exchange with the ambient during the transportation. After weighing, the dish was immediately placed back into the desiccator for the next time interval use. The net weight of the sodium bicarbonate powder in each dish was then calculated.

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. Equilibrium was judged to have been attained when difference between the three consecutive weights did not exceed 1 mg [17]. The equilibrium period ranged from 3 to 4 weeks. After reaching equilibrium, the dry weight was determined after drying in a vacuum oven at 50°C for 24 h [18] and the equilibrium moisture content was evaluated.

To measure a true adsorption isotherm, the powder should be dried to zero moisture without changing the properties of the powder prior to isotherm measurement. This can dramatically increase the experimentation time, therefore it is not uncommon to measure a working isotherm. A working isotherm does not require the powder to be completely dry first, but measures the initial moisture content for the powder which is then used to correct the moisture gains/losses for the powders exposed to relative humidities greater than zero. The powders should be left in the controlled relative humidity environments until equilibrium is reached, which is usually 3 weeks [19]. Each set of experiment was repeated thrice and mean values were recorded.

2.3 Modelling equations

For adjusting of the experimental data from adsorption isotherms of sodium bicarbonate, mathematical models of BET, Kuhn, Hasley, GAB and Oswin were used, represented respectively by the equations in Table 2. 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 [20]. These two parameters were calculated using the following equations:

Table 2: Isotherm equations for experimental data fitting

Model	Mathematical expression	aw range
BET (Brunauer *et al.*, 1938)	$X_{eq} = {Xm\,Caw}/{[(1-aw)(1+(C-1)aw)]}$	(1) aw <0.50
GAB (Van den Ben and Bruin, 1981)	$X_{eq} = {XmCKaw}/{[(1-Kaw)(1+CGKaw-Kaw)]}$	(2) 0.05<aw<0.95
Hasley (1948)	$X_{eq} = \left({-A}/{\ln aw}\right)^{\frac{1}{B}}$	(3) 0.05<aw<0.80
Kuhn (Labuza, et *al.*, 1972)	$X_{eq} = {A}/{\ln aw} + B$	(4) aw<0.5
Oswin (1946)	$X_{eq} = A\left[{aw}/{(1-aw)}\right]^{B}$	(5) 0.05<aw<0.90

Variables to measure experimentally: Xeq = equilibrium moisture content (% d.b.); aw = water activity

Parameters to be estimated from the data : A= constant (dimensionless), B = constant (dimensionless), C = GAB or BET model parameter (dimensionless), K = GAB model parameter (dimensionless), Xm = Monolayer moisture content (% d.b.)

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

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

Where N is the number of observations, Xeq, exp and Xeq, pre are the experimental and predicted values of the equilibrium moisture content, respectively.

In the GAB model, Xm 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 multilayer molecules with respect to the bulk liquid.

3 RESULTS AND DISCUSSION

3.1 Adsorption isotherm

The quality of preserved food is largely dependent on its moisture content, moisture migration and/or moisture uptake of the food during storage. Therefore, the water activity level, which corresponds to a range of equilibrium moisture contents, must be determined by, for example, the use of moisture isotherms. Depending on the nature of food powder (crystalline or amorphous), the shape of isotherm is different. Even for such an apparently simple system as sodium bicarbonate powder, numerous factors affect the adsorption of water vapour and, as a consequence, the storage stability [1].

The measure of the hygroscopicity of a product is consequently the magnitude of the increase or decrease in its water content as a function of relative humidity at a constant temperature. Weakly hygroscopie products exhibit no or only a slight change in their water content as a consequence of variations in relative humidity. In strongly hygroscopie products, water content may vary widely [21].

The adsorption isotherm of sodium bicarbonate powder demonstrate an increase in equilbrium moisture content with increasing water activity (Figure 1). The equilibrium moisture content of the adsorption isotherms increased slowly between aw values 0.07 (4.06 g H2O/100 g solids) and 0.75 (21.40 g H2O/100 g solids), followed by a steep rise (157.64 g H2O/100 g solids). Consequently, the adsorption isotherm obtained in this investigation follows the type III BET classification shape, which are characteristic of products holding small amounts of water at lower aw values and higher amounts of water at high RH levels [22]. It should be noted that a type III isotherm appears when the binding energy for the first layer is lower than the binding energy between water molecules [23]. The moisture content increases exponentially above aw equal 0.75 and adjoin an asymptote at a water activity of one.

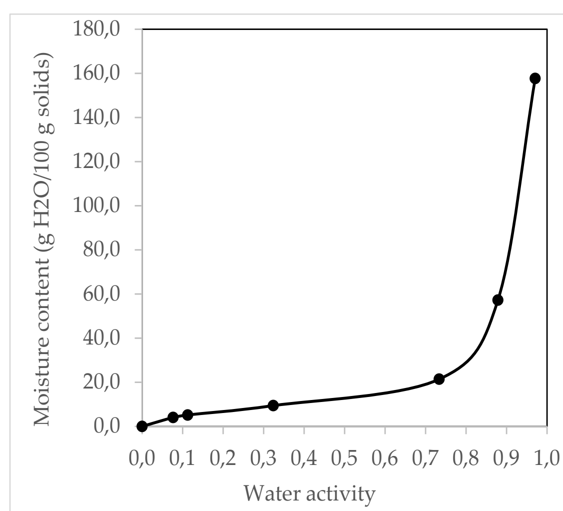

Figure 1: Adsorption isotherm of sodium bicarbonate powder at 29°C

The experimental data in this study are similar to those of sucrose and fructose at 25°C [24]. These results are also in agreement with those of Jouppila and Ross [15] for milk powders. One reason for the exponential increase in moisture content at high water activities is capillary condensation [19]. Capillary condensation is the process where direct condensation can occur, due to surface tension effects, in the capillaries formed at the contact points between adjacent particles. This observation confirm those reported previously in different food products i.e. sugars, apple, raisins, and pistachio powder [25]. These results were also in well harmony with those previously reported for syrop powders [26] and with those found for food materials rich in sugars as sweet potato [27]. Foods with high levels of small, soluble molecules and small amounts of polymeric compounds may exhibit a Type III curve [28].

3.2. Fitting of sorption models to experimental data

The results of nonlinear regression analysis of fitting the GAB, BET, Oswin, Kuhn and Hasley equations to the experimental data are show in Table 3. The coefficient correlation (R2) and the mean relative percentage deviation modulus (MRD) are also given in Table 3. R2 indicates how well the variability has been explained by the given model. The suitability of these five models were evaluated statically by using Mean Relative Determination (MRD) method with the value of < 5 describes that the model was precise, < 10 describes the model was almost precise and > 10 describes the model was imprecise. Lower the values of MRD, better is the goodness of fit of the model [29].

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	4.702
	C	365.499
	K	18.466
	R^2	0.992
	MRD (%)	17.7
BET	Xm	7.442
	C	12.449
	R^2	1
	MRD (%)	0.8
Kuhn	A	-10.448
	B	0.181
	R^2	0.997
	MRD (%)	3.2
Hasley	A	8.439
	B	1.037
	R^2	0.996
	MRD (%)	18.4
Oswin	A	13.302
	B	0.705
	R^2	0.999
	MRD (%)	22.1

In order to confirm BET and Kuhn eqaution’s ability in predicting the equlibrium moisture content data, MRD (%) values corresponding to adsorption isotherm were calculated and the results are given in Table 3. The BET (MRD %= 0.8%) and Kuhn (MRD %= 3.2%) equations gave smaller mean relative percentage deviation modulus (less than 10%) in the activity range 0.07-0.5. These values confirmed that the BET and Kuhn equations give the closest fit to the experimental adsorption isotherm of sodium bicarbonate at 29°C (Figure 2 and 3). Nevertheless, the BET equation gave the best description of the adsorption isotherm. This model is the best for describing the equilibrium moisture data within this range as stated by several researchers [30]; [31]; [32]. The BET equation, which is the most widely used model in food systems, was first proposed by Brunauer, Emmett and Teller [33]. It represents a fundamental milestone in the interpretation of multilayer sorption isotherms, particularly the types II and III [33].

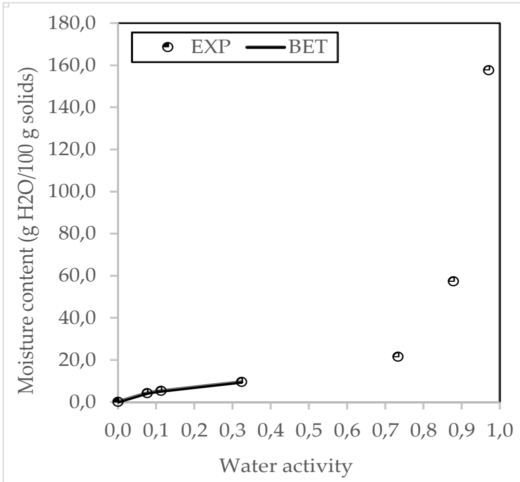

Figure 2: Adsorption isotherm of sodium bicarbonate powder predicted by Brunauer-Emmet-Teller (BET) model

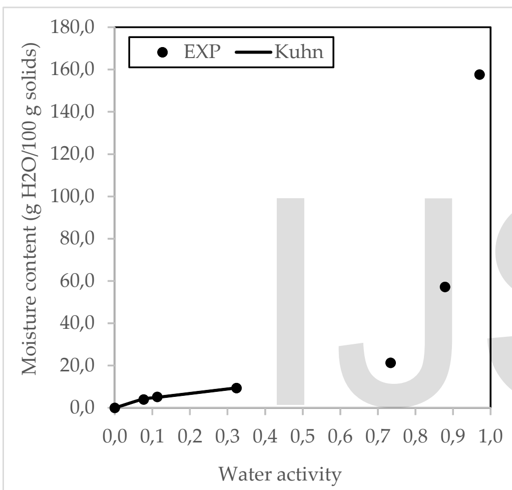

Figure 3: Adsorption isotherm of sodium bicarbonate powder predicted by Kuhn model

It was found that the GAB (MRD% = 17.7%), Hasley (MRD% = 18.4%), and Oswin (MRD% = 22.1%) equation were inadequate to describe the adsorption behavior of sodium bicarbonate powder (Figure 4, 5 and 6). This result does not agree with Lomauro et al. [34], who reported a good fit with GAB, Hasley and Oswin models to food isotherms which exhibited a sigmoid shape.

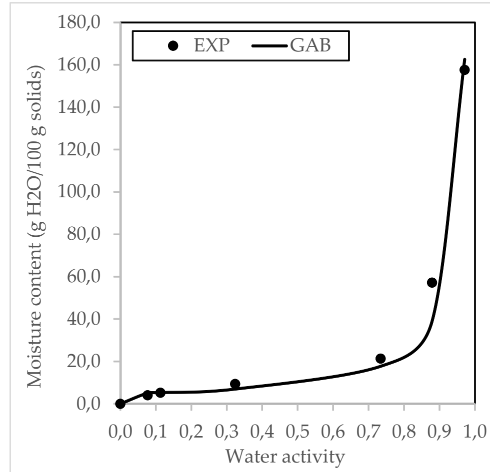

Figure 4: Adsorption isotherm of sodium bicarbonate powder predicted by Guggenheim-Anderson-de Boer (GAB) model

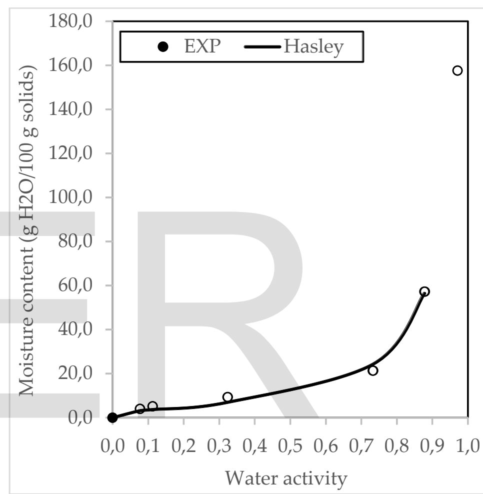

Figure 5: Adsorption isotherm of sodium bicarbonate powder predicted by Hasley model

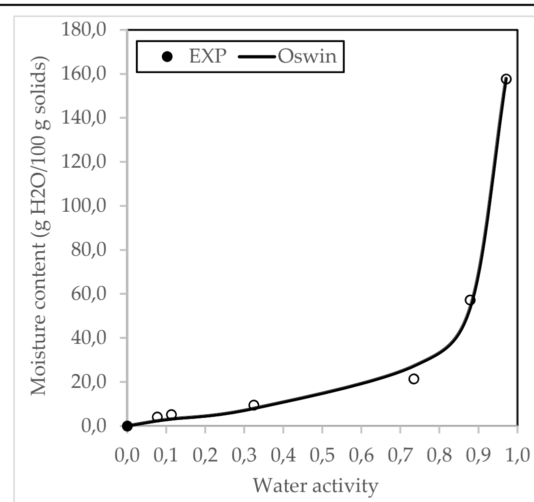

Figure 6: Adsorption isotherm of sodium bicarbonate powder predicted by Oswin model

Nevertheless, of the three equations (GAB, Hasley and Oswin) that cover the entire range of water activity (0.77 - 0.97), the GAB equation is most applicable to the experimental data of the sodium bicarbonate adsorption curve. According to Lomauro et al. [34], the GAB model provides the best fit for more than 50% of fruits, meats and vegetables analyzed. This model has been widely applied to describe equilibrium moisture content in foodstuffs, and was recommended by European Project COST90 [35].

Oswin equation gave the poorest fit to the data (figure 6). Contrary to our results, the Oswin model gives a better fit for most of the food isotherms which exhibited sigmoid shape [13]; [36]. So Ayala [37] reported good fit in cassava flour with the Oswin model. Wang and Brennan [32] previously reported that the sorption isotherms of starch based foods can be reasonably described using Oswin model.

The Hasley equation (MRD% = 18.4%) representing the multilayer adsorption did not give a good fit to the experimental data for sodium bicarbonate powder within a wide water activity range (0.77 - 0.97). Which did not agree with the results of Ayranci et al. [38], Yanniotis et al. [39] for grapes containing high amounts of sugars and McLaughlin and Magee [40] for potatoes. Similarly, Kaymak-Ertekin and Sultanoglu [41] concluded that the Hasley equation gave the best fit for peppers. Also according to Giner and Gely [42], Hasley model permits better representation of sorption isotherms of oilseeds.

The monolayer moisture content (Xm), represents the moisture content of the food material when the entire surface is covered with a unimolecular moisture layer and the optimum moisture content for maximum shelf stability may be determined [43]. As in the BET equation, the monolayer capacity is represented in one of the three GAB constant. The monolayer moisture content (Xm) obtained by the GAB model (4.702 g H2O/100 g solids) was much lower than that obtained by the BET model (7.442 g H2O/100 g solids) (table 3). These values are similar to those reported by other authors; Hossain et al. [43] reported Xm values between 0.041 and 0.05 g per g dm of pineaples. They are also in line with the values reported by Yanniotis [44] for high starchy foods between 20 and 30°C (7.36 kg/100 kg). The availability of active hydrophilic sites on the surface of food is reflected on the amount of monolayer [45]. However, Kiranoudis et al. [46] obtained values between 0.087 and 0.212 g per g of dm of potatoes and carrots; Talla et al. [47] found values between 0.080 and 0.185 g per g of dm of banana, mango and pineapples. These monolayer moisture content are higher than those found in this study. Monolayer capacity is used as an indicator of the availability of polar sites for water vapour, determined not only by the number of components abundant in polar sites, but also their physical status. For most dry foods, the rate of quality loss due the chemical reactions is negligible below the monolayer value [19]. These values are particularly important in storage of the product, since level the water does not act as a solvent, being biologically inert [48].

The energy constant C (C_{GAB} = 365.499, C_{BET}= 12.449) 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 [49], an indicator of appropriateness for choosing the GAB model to describe empirical data.

The K parameter (K_{GAB}= 18.466) 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 [49], and diversifies monomolecular ($K \leq 0.5$) and multilayer adsorption ($K > 0.5$) [50].

4 CONCLUSIONS

The moisture adsorption of sodium bicarbonate 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. Although the BET and Kuhn models showed satisfactory statistical tests, the range of aw in which the models can predict sorption behaviour is incomplete, for this reason, only GAB model present the best fitting performance. For sodium bicarbonate powder, the monolayer moisture content can be used to evaluate the shelf stability and efficient use of energy in the drying process. This study will help in designing packaging systems of an important food product like sodium bicarbonate so that it can be stores for a longer period for preparations of value added products.

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