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Moisture adsorption isotherm of commercial wheat flour in Côte d'Ivoire

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Abstract - *The quality of preserved wheat flour is largely dependent on its moisture content, moisture migration and moisture uptake of the flour 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. The shape of the isotherm curve is dependent on the interactions between the vapour molecules and the solid material which can aid understanding of the mechanism taking place during water sorption. The moisture sorption isotherms of wheat flour was experimentally determined using a standard gravimetric method. Experimental data was compared with seven (BET, GAB, Chung and Pfost, Hasley, Kuhn, Oswin and Freundlich) mathematical models and their goodness of fit evaluated. The isotherms showed that wheat flour exhibited Type II characteristics. BET (R2=0.99 and MRD (%) = 3.4%), Kuhn (R2=0.98 and MRD(%)= 4%) and Hasley (R2=0.92 and MRD (%)= 7.7%) models all adequately represented the results. Critical value of equilibrium moisture content of wheat flour corresponding to the aw equal to 0.6 was 26.2 g water per 100 g dry mater. This value is useful for storage conditions optimisation from point of view microorganism grow and structural changes analyse. The monolayer moisture content (Xm) values for BET (18.146 g water per 100 g dry mater) was higher than those for GAB (1.823 g water per 100 g dry mater).*

***Key Words*:** wheat Flour; adsorption isotherm; matematical model

1. INTRODUCTION

Wheat is the first food crop all over the world for being the basic raw material for the production of Flour used in making bread, and bread is a staple food for more than three quarters of the globe population, so the stability of any country, food security depends on the availability of this article, cultivation, productive and to reach its optimum consumption, as wheat is the raw material for many of the food industry in all its forms, pasta, semolina, bulgur and couscous and other uses according to the habits and patterns of peoples in different countries world.

Wheat occupies the largest share among all grain crops in terms of its presence at the tables food, constitutes in many countries approximately 70% of its food needs, whether it is direct or indirect, and therefore it costs a lot of local and foreign currencies [1]. The amount of the world production of wheat was estimated at 713.2 million tons 2013 [2] comes the result of the increasing consumption bread and nutritional value of the human person, in terms of average global per capita consumption of wheat was about 85 kg/year [3]. And annual increase in global wheat consumption reached to 2% [4].

Globally, wheat is the leading source of vegetable protein in human food, having higher protein content to maize, rice, and other major cereals. Wheat flour is an excellent source of complex carbohydrate. Depending on the flour type, the percentage of the calories from protein ranges from 9 to 15 percent, except the gluten, which has 45% protein content. Wheat flour is a very hygroscopic material like any other flour and its moisture changes with the changes in temperature and humidity of the storage environments. Flour moisture changes can support the acidity alterations caused by enzymatic reactions [5]; [6], [7].

A moisture sorption isotherm has been used to describe the relationship between moisture content and equilibrium relative humidity, and that knowledge is useful for understanding two phenomena in food technology [8]. One is the storage stability of food products. The microbial growth, enzymatic reactions, non-enzymatic browning and lipid oxidation are some of the deteriorative mechanisms that are known to be related to the moisture content [9]. Another is the drying or rehydration of food. The equilibrium moisture content allows us to optimize drying times and energy utilization. In this context, the moisture sorption isotherms of many food products, for example, starchy foods (e. g., corn, potato and rice), high protein foods (e.g., chicken, egg, Milk and cheese), fruits (e.g., banana, apple, apricots and raisins) and vegetables (e.g., green pepper, lentil, tomato, onion, sugar beet root, carrot and celery) have been experimentally determined as reviewed by [10].

A number of models have been proposed in the literature for the dependence of the equilibrium moisture content and the relative humidity. In 1981, van den Berg and Bruin classified the models into 77 types. These models can be further categorized into several groups: kinetic models based on the monolayer sorption theory (e.g., Langmuir model), kinetic models based on the multilayer sorption theory (e.g., BET and GAB models) and empirical and semi-empirical models (e.g., Peleg and Oswin models) [11].

Moisture content of wheat flour is very important regarding its shelf life, lower the flour 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 [12]. The sorption property of wheat flour is a particularly important parameter conditioning the rheological properties of wheat products, because this parameter is a resultant of the structure, composition and biophysical properties of proteins and their interaction with other elements of flour, i.e. with starch, lipids and non-starch polysaccharides [13]. Moisture is also of great importance for the safe storage of cereals and their products regarding microorganisms, particularly certain species of fungi [14]. In climate like Côte d'Ivoire, the shelf life of flour is a serious problem and due to weather conditions, it is inevitable to explore proper moisture content to over come existing dilemma. The present study was carried out to extend the shelf life of flour by determining the proper moisture content suitable for safe storage.

Because of the need to assess the extent of changes in the quality of wheat flour and its properties, under possible storage conditions, purpose of this study was to obtain exprimental equilibrium adsorption isotherm of wheat flour at 29°C and to find out a suitable model describing this adsorption isotherm. In addition, the monolayer moisture content was calculated.

2. MATERIAL AND METHODS

2.1. MATERIAL

The present work was conducted in the Laboraty of Evaluation of Physical Properties and Quality of National Agricultural Research Center in Bingerville, Côte d'Ivoire. Wheat flour which was purchased from a local market in Abidjan (Côte d'Ivoire) was used for the determination of its adsorption iostherm at 29°C.

2.2. METHODS

2.2.1. Determination of adsorption isotherms

The course of wheat flour isotherms was determined by the static desiccator method [15]. The principle of the determination of sorption isotherms is based on the determination of equilibrium moisture between the test sample and the atmosphere of a certain relative humidity, controlled by appropriate saturated salt solutions (table 1). The scope of research included water activity from 0.07 to 0.97. Temperature of the examination was 29°C. The time for establishing equilibrium of the system was 35 days from placing the samples in desiccators.

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

In desiccators with water activity above 0.7 thymol was placed to protect against the development of microorganisms. Individual samples of flour used for the determination of adsorption isotherms in the first phase of the experiment were placed in an amount of about 2 g ± 0.1 mg in dishes with a diameter near 35 mm, so as to completely and evenly cover the surface of the layer. Then the dishes were placed in a desiccator containing P2O5 as the desiccant, at room temperature for 3 weeks in order to minimise the moisture content (~2%) in the tested flours.

After that the masses of the samples were specified and placed in desiccators with appropriate saturated salt solutions. On the basis of the initial weight of the product (determined after 3 weeks of incubation in a desiccator with P2O5) and changes in water content, the equilibrium moisture contents were determined. The desiccators were placed in the laboratory at room temperature and the samples were allowed to equilibrate until there was no discernible weight change (± 0.001 g). 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. In order to observe the experimental errors, the wheat flour moisture reading were measured in triplicates. Equilibrium was reached hen the sample weight difference between two successive measurements was less than the balance accuracy 0.001 g.

2.2.2. Sorption models and statistical analyses

Aiming to mathematically express the relation between the water activity of food and its moisture content, diverse models have been developed suc as nonlinear, linear, regressional models, constituted in their parameters by two, three, four and six partial regression coeff icients, which explain each one of the three zones that the isotherm of sorption of humidity conforms. In many cases, the model that is suitable for certain food product is not suitable for a different one, what is more, the model only exhibits a suitable predictive ability for certain moisture activity ranges. Several mathematical models have been proposed to describe sorption isotherms. Some of them were developed with a theoretical basis to describe adsorption mechanisms [16]; [17]; whereas the others are just empirical or a simplification of more elaborate models. In some ranges of water activity, sorption isotherms can be approximated to linear equations [18].

There are some semi-empirical equations with two or three fitting parameters to describe moisture sorption isotherms. The most common equations that are used for describing sorption in food products are the Langmuir equation, the BET equation, the Oswin model, the Smith model, the Halsey model, the Henderson model, the Iglesias-Chirife equation, the GAB model, and the Peleg model [19].

Experimental data were fitted to seven moisture sorption isotherm models, such as Guggenheim-Anderson-De Boer (GAB), BET, Oswin, Chung and Pfost, Hasley, Kuhn and Freundlich (table 2), where aw represents the equilibrium relative humidity in decimal; Xeq is the equilibrium moisture content in % (db); t is the temperature in °C; A, B, C are sorption isotherm constants specific to each equation. These seven models (table 1) were chosen because they are most widely used to fit experimental sorption data of various food materials.

Table -2: Isotherm equations for experimental data fitting

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

Non-linear regression analysis was used to calculate the respective constants using software like, Microsoft excel 2007 (Microsoft Corp., USA), Systat 8.0 (SPSS, Inc., 1998), and Origin 6.1 (Origin Lab Corporation, Northampton, MA 01060, USA, 2000).

The quality of the fitting of different models was evaluated by calculating the correlation coefficient (R2) and the mean relative percentage deviation modulus (MRD %) between the experimental and predicted equilibrium moisture content [20].

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

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

Where Xeq, exp is the experimental EMC value, Xeq, pre is the predicted EMC value and N is the number of experimental data.

Best fitting aquations were evaluated with the correlation coefficient (R2) and the mean relative percentage deviation modulus (MRD %). The value of R2 close to 1 and MRD below 10% indicates a good fit for practical purposes [21].

3. RESULTS AND DISCUSSION

3.1. Behaviour of adsorption isotherm

The isotherm of wheat flour samples tested was classified as sorption isotherms of type II (Chart 1). According to Limousin et al. [22], sigmoid curves of sorption indicate that the observed phenomenon reflects the formation of polymolecular water layers on the surface of the examined flours. "S"-shaped isotherms, characterized by the occurrence of two points of inflection, are used for the description of the three-degree process of adsorption, in which at least two opposing mechanisms are used [22]. The first section of the isotherm is shaped with the participation of polar groups of hydrophilic components, characterized by a high binding energy. In the case of wheat flour, those components include starch, proteins and pentosans subject to gradual saturation with water molecules. In the second section of the isotherm, formation of water molecules into so-called bunches can be observed. The third section reflects the process of water accumulation in the intermolecular spaces, which leads to partial swelling of matrix macromolecules (mostly proteins) and, consequently, to a loosening of their structure and exposing additional hydrophilic spots [23]; [24]; [25].

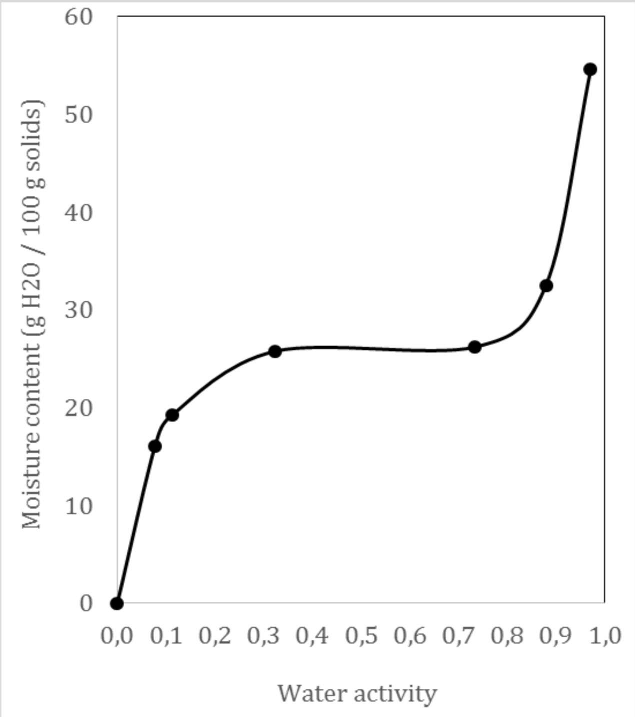

Chart -1: Adsorption isotherms of wheat flour at 29°C

The adsorption of moisture by wheat flour grew in line with increase of aw. The phenomenon of adsorption of moisture from the environnement was conditionned by a considerable amount of protein and starch in the examined flour. The components, being macromolecules rich in polar places (groups –OH or –H), make it possible to adsorb moisture considerably [13].

Above the level of 0.75 aw the effect of adsorption became particularly distinct. One may suppose that above 0.75 aw in wheat flour there occurred the phenomenon capillary condensation [13]. That higher sorption capacity of wheat flour may be due to its lower fat content and higher mass exchange area.

Moisture adsorption by wheat flours grew in the entire range of aw, and no areas of discontinuity were observed, which indicates that the process of water adsorption from the environment was not accompanied by changes leading to an increase in the degree of macromolecule arrangement. Adsorption capacity of flour is determined by arrangement of polar groups in the external part of flour particles, accessibility of those groups for water, determined by the folding of biopolymers forming chains, crystallisation rate of the matrix and the relative force of water-water and water-macroparticle interaction [26].

This increase of moisture adsorption was also due to the lower initial moisture content of wheat flour, which rendered them to absorb moisture from the atmosphere. The results regarding the moisture content during storage supported the earlier findings of Rehman and Shah [27]; Samuels and Modgil [28]; and Kirk and Sawyer [29]. Higher moisture content in wheat flour favored proteolytic activity. These results are in These results are in close agreement with the results obtained by Leelavathi et al. [30] and Upadhyay [31].

Therefore, differentiation of sorption properties of the examined samples can be equated with changes in the conformation of macromolecules (protein and starch). Most probably, flour particles adsorbing water are more easily subject to structural transformations determined by the presence of water particles characterized by higher mobility and activity [32].

3.2. Mathematical modelling of adsorption data

The results of nonlinear regression analysis of fitting the GAB, BET, Oswin, Chung and Pfost, Hasley, Kuhn and Freundlich equations to the experimental data are show in table 3. The coefficient correlation (R^2) and the mean relative percentage deviation modulus (MRD) are also given in table 3. R^2 indicates how well the variability has been explained by the given model and MRD (%) values are indicative of reasonable good fitting when MRD (%) < 10%.

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	1.823
	C	293.042
	K	161.659
	R^2	0.96
	MRD (%)	68.2
BET	Xm	18.146
	C	66.200
	R^2	0.99
	MRD (%)	3.4
Kuhn	A	-18.059
	B	9.887
	R^2	0.98
	MRD (%)	4
Hasley	A	22267990.077
	B	5.453
	R^2	0.92
	MRD (%)	7.7
Oswin	A	25.268
	B	0.199
	R^2	0.96
	MRD (%)	12
Chung and Pfost	A	22.823
	B	0.136
	R^2	0.94
	MRD (%)	12.2
Freundlich	A	41.112
	B	2.469
	R^2	0.80
	MRD (%)	17.7

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 %= 3.4%) and Kuhn (MRD %= 4%) eqautions 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 wheat flour at 29°C (Chart 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 [33]; [34]; [35]. The BET equation, which is the most widely used model in food systems, was first proposed by Brunauer, Emmett and Teller [17]. It represents a fundamental milestone in the interpretation of multilayer sorption isotherms, particularly the types II and III [17].

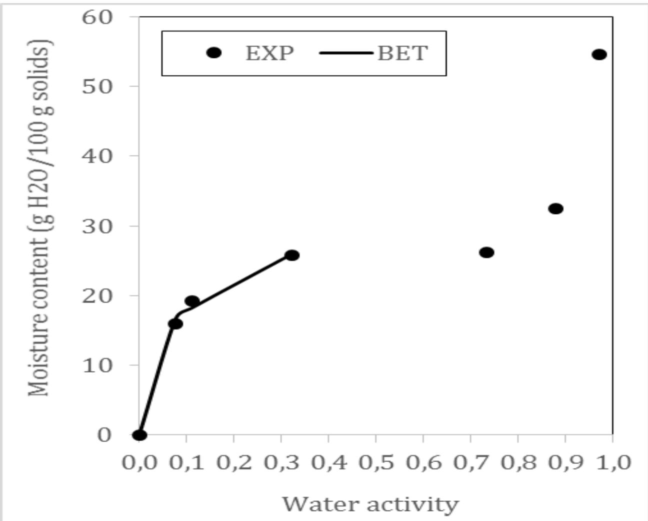

Chart -2: Adsorption isotherms of wheat flour predicted by Brunauer-Emmet-Teller (BET) model

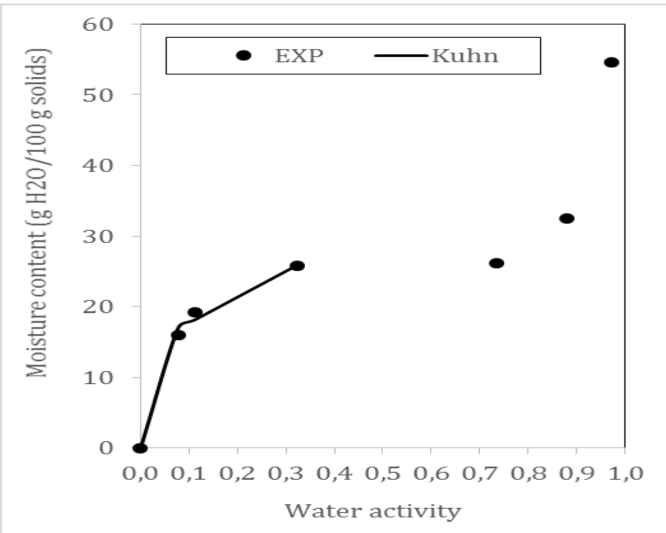

Chart -3: Adsorption isotherms of wheat flour predicted by Kuhn model

It was found that the Oswin (MRD%= 12%), Chung and Pfost (MRD%= 12.2%), Freundlich (MRD%=17.7%) and GAB (MRD%=68.2%) equation were not acceptable for describing the adsorption behaviour of wheat flour (Chart 4, 5, 6 and 7, respectively). GAB equation gave the poorest fit to the data. Contrary to our results, the GAB model gives a better fit for most of the food isotherms which exhibited sigmoid shape [36]; [37].

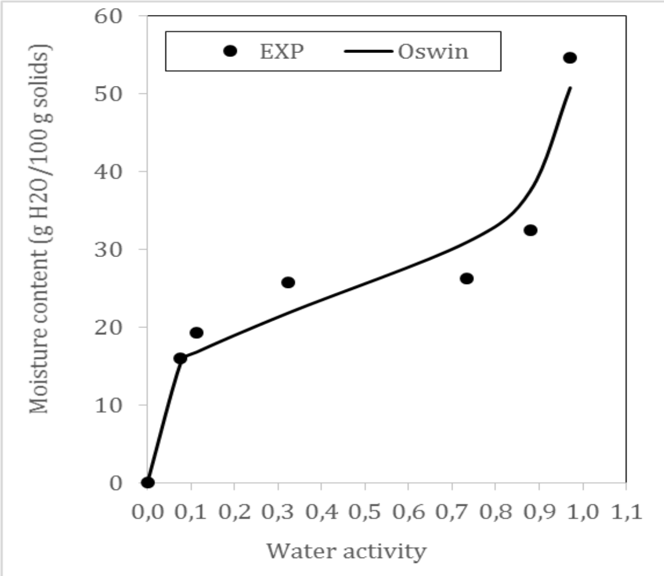

Chart -4: Adsorption isotherms of wheat flour predicted by Oswin model

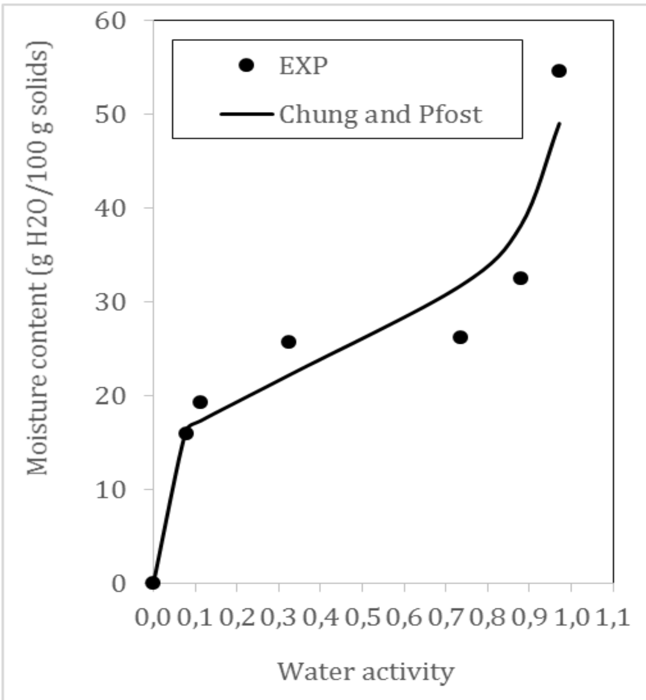

Chart -5: Adsorption isotherms of wheat flour predicted by Chung-Pfost model

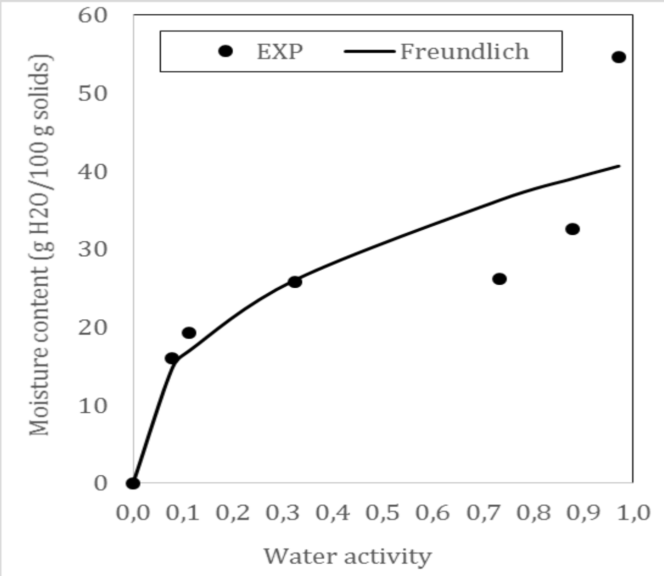

Chart -6: Adsorption isotherms of wheat flour predicted by Freundlich model

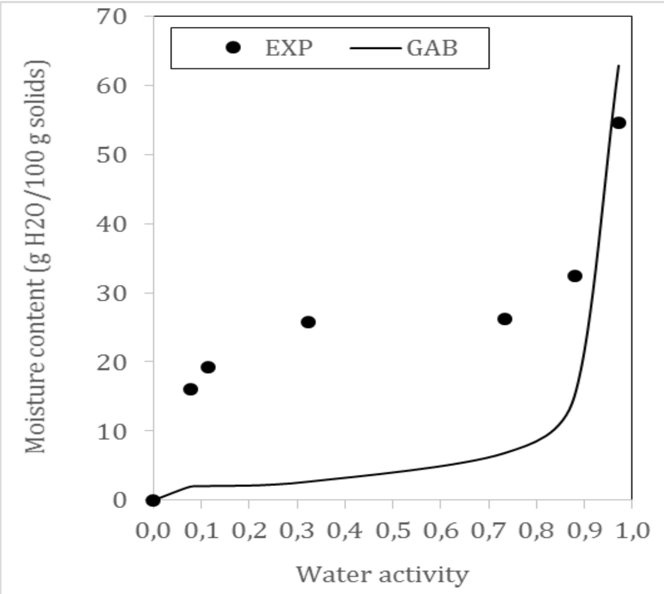

Chart -7: Adsorption isotherms of wheat flour predicted by Guggenheim-Anderson-de Boer (GAB) model

It can be seen from the data on table 3, the Hasley equation (MRD%= 7.7%) representing multilayer adsorption gave the best fit to the experimental data for wheat flour within a wide water activity range (0.07-0.97). There is a good agreement 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. Kaymak-Ertekin and Sultanoglu [41] concluded that the Hasley equation garve the best fit for peppers. Also according to Giner and Gely [42], Hasley model permits better representation of sorption isotherms of oilseeds.

As in the BET equation, the monolayer capacity is also represented in one of the three GAB constant. The monolayer moisture content (Xm) obtained by the GAB model (1.8 g H2O/100 g solids) was much lower than that obtained by the BET model (18.14 g H2O/100 g solids) (table 3). This value is larger than those reported by other authors; Hossain et al., [43] reported Xm values between 0.041 and 0.05 g per g dm of pineaples. Kiranoudis et al., [44] obtained values between 0.087 and 0.212 g per g of dm of potatoes, carrots; Talla et al., [45] found values between 0.080 and 0.185 g per g of dm of banana, mango and pineapples. These values are similar to 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 (in the case of wheat flour, starch and protein above all), but also their physical status.

The starch granules structure's of wheat flour is varied and can additionally be subject to changes as a result of their disaggregation or interactions between them and other flour components, which has been reported in numerous studies [46]; [24]; [47] ; [48] ; [25]. The monolayer value helps to specify the amount of water which is strongly adsorbed and it is considered to be the optimal value at which food is more durable [49].

The results are valid for a complete range of water activity from 0.07 to 0.97 for all the models except for the BET and Khun, which are suitable between 0.07 <aw<0.5. The goodness of fit for the mentioned mathematical models has been reported by other authors studying sorption behaviour of different foods [50]; [51]; [52]. Although the BET and Khun models showed satisfactory statistical tests, the range of aw in which the model can predict sorption behaviour is incomplete, for this reason, only Hasley model present the best fitting performance. Halsey model provides an expression for the condensation of multilayers at a relatively large distance from the surface, assuming that the potential energy of a molecule varies as the inverse nth power of its distance from the surface. This equation is a good representation of adsorption data regarding isotherms type I, II, or III. Moreover, this equation described the sorption behaviour of food products that contain starch [53]; [54].

It should be noted that, the goodness of fit of any sorption model to the experimental data shows only a mathematical quality and not the nature of the sorption process [55].

The energy constant C (CGAB = 2903.042, CBET= 66.200) 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 [56], an indicator of appropriateness for choosing the GAB model to describe empirical data.

The K parameter (KGAB= 161.659) 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 [56], and diversifies monomolecular ($K \leq 0.5$) and multilayer adsorption ($K > 0.5$) [57].

4. CONCLUSIONS

Increase in moisture will provide a suitable environment for microbiological activities which can result in spoilage. It is therefore recommended that wheat flour should be packed in airtight and moisture proof bag especially in areas of high humidity to prevent mold growth and maintain it qualities when stored under room temperature. Adsorption isotherm of wheat flour was characterized by a sigmoid shape (curve typical of the type II classification shape) and continuous course across the entire range of water activity, which indicates a multi-layer process of surface adsorption of water molecules, not accompanied by an increase in the degree of the arrangement of the solid body matrix. Among the sorption models tested, the Hasley equation describes the adsorption data well over the range of water activity studied. The adsorption data were also well expressed by the BET and Kuhn equation in range of water activity 0.07-0.5. For wheat flour, the monolayer moisture content can be used to evaluate the shelf stability and efficient use of energy in the drying process.

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