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COMPARISON AND MODELING OF ADSORPTION ISOTHERMS OF TROPICAL VEGETABLES: PLANTAIN (*MUSA PARADISIACA* AAB VAR. *CORNE* 1), OKRA (*ABELMOSCHUS ESCULENTUS* VAR. *TOMI*) AND BELL PEPPER (*CAPSICUM ANNUUM* VAR. PM17/04A)

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ABSTRACT

Plantain (*Musa Paradisiaca* AAB var. *Corne* 1), okra (*Abelmoschus Esculentus* var. *Tomi*) and bell pepper (*Capsicum Annuum* var. PM17/04A) contribute to food security in developing countries such as Côte d'Ivoire. These foods, which have high moisture content, can be kept only within a few days after harvest. Processing these products by drying and changing into flour is one solution for easy handling and long-term storage. Moisture adsorption isotherm information is clearly needed for processing of plantain, okra and bell pepper flour. However the published literature about adsorption isotherms of these products is limited. The objectives of this work were to determination their adsorption isotherms, and to evaluate GAB sorption isotherm model. The adsorption data were analyzed for determination of monolayer moisture content. The adsorption isotherms had sigmoid sharp profiles (type II). GAB model was the most appropriate for the adsorption isotherm of plantain flour. A higher water content was observed in plantain (33.1 $gH_2O/100g$) compared to okra (28.06 $gH_2O/100g$) and pepper flour (21.14 $gH_2O/100g$). The monolayer moisture content (gH_2O /100 g dry matter) range from 0.7972g for bell pepper to 3.852g for plantain flour. That of okra was 1.296g. These values were optimal in order to ensure safe storage condition.

INTRODUCTION

The storage of fresh plantain, okra and bell pepper in the tropical countries is difficult because of their very perishable character (Talla, 2012). In order to preserve these fruits and vegetables, and make it available to consumers during the whole year, it undergoes specific technological treatments, such as drying. In order to minimize drying cost and to improve the nutritional value, drying methods have to be optimized. One way is to introduce a solar drying system. It is particularly important for countries which have more sun throughout the year (Ekechukwu and Norton, 1999). The consumption of these fruits and vegetables is considered by many authorities as a public health issue and the subject of nutritional recommendations at the global level by WHO (Amiot-Carlin *et al*., 2007). This consumption contributes to food security in developing countries such as Cote d'Ivoire.

The important post-harvest losses of fruits and vegetables in developing countries are mainly due a lack of appropriate handling (Tano, 1997). Reducing the post-harvest losses may be partly due to their processing into flour, allowing their preservation for a long period. Plantain, okra and bell pepper flour are most often encountered on African countries markets, particularly in Côte d'Ivoire. Like most traditional processed flour products, the steps of preservation is done spontaneously and empirical, and the final product is characterized by an unsatisfactory hygienic and organoleptic quality (Yao, 2009). The level of moisture affects the physical and rheological characteristics of food, and their stability (Lewicki, 2004). It is well known that the microbial stability of food is strongly dependent on its water activity (Aw). This water activity which is defined as the ratio of the vapor pressure of water in the food to the vapor pressure of pure water at the same temperature, is used to express the amount of water available in a food system for microbial growth and biochemical reactions. An important aspect of Aw concept is the moisture sorption isotherm which is a graphical representation of the relationship between moisture content of a product and Aw at

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a constant temperature (Nurtama and Lin, 2010). Preservation or storage of dried food products encounters many difficulties in tropical countries. These difficulties are in part due to the poor mastery of storage conditions which generally are empirically. There is a necessity to determine the adsorption isotherms of tropical flour food to maximize their storage and to establish a reliable database for each of them. One important property of food product related to drying is its moisture sorption isotherm, since will determine the degree of drying required to obtain a stable product (Sereno *et al*., 2001). The knowledge and understanding of sorption isotherms is highly important in food processing for the design and optimization of drying equipment, design of packages, predictions of quality, stability, shelf-life and for calculating moisture changes that may occur during storage. Several preservation processes have been developed in order to prolong the shelf-life of food products by lowering the availability of water to micro-organisms and inhibiting some chemical reactions (Siripatrawan and Jantawat, 2006).

Equations for fitting water sorption isotherms in foods are of practical importance in many aspects of food preservation by dehydration, such as for prediction of drying times, the shelf-life of dried product in a specific packaging material, the evaluation of properties of dry mixes (Gonzalo *et al*., 2011). Several different isotherm models have been proposed and compared in the literature. While there are 270 proposed isotherm models, the most commonly used are the GAB and BET models. Since the BET model is only applicable if the Aw is up to 0.5, the GAB model is widely accepted as the most useful for characterizing isotherms across the entire water activity range. Its coefficient also has theoretical and physical meaning such as providing monolayer moisture content (Kane *et al.*, 2008). However, in the literature there is not enough data on the sorption isotherms of most tropical fruits and vegetables. In this study, the adsorption isotherms of plantain, okra and bell pepper flour were determined in order to maintain their quality during storage. Specifically the objectives were to:

- experimentally investigate the adsorption isotherm of plantain, okra and bell pepper flour,
- describe the experimental data using selected mathematical model (Guggenheim, Anderson and de Boer),
- determine the monolayer moisture content of these three products and the parameters of the GAB equation.

MATERIALS AND METHODS

Products flour preparation

Fresh plantains (*Musa Paradisiaca* AAB var. *Corne* 1), okra (*Abelmoschus Esculentus* var. *Tomi*) and pepper (*Capsicum Annuum* var. PM17/04A) used in experiments were purchased from the Abobo market (Abidjan, Côte d'Ivoire). After washing; peeling and slicing (10 mm); products were dried using a hot air dryer (Venticell, Medcenter, Germany) at 45 °C for 3 days (the weight of the sample was measured periodically and drying was stopped when the weight of the sample was being unchanged). The dried products were milled using a lab use grinder to make product flour. Samples used in the experiment were plantain; okra and bell pepper flour 40 mesh.

Measurement of sorption isotherms

For food products, the adsorption isotherm can be measured by means of three different measuring techniques: gravimetric, manometric or hygrometric, according to Iglesias and Chirife (1976). A gravimetric method was used in this work. The method was based on the use of saturated salt solutions to maintain a fixed relative humidity. Eight salts were chosen so as to have a range of relative humidifies of 11 - 97%. The corresponding values of water activities were: LiCl (A_w = 0.11), $MgCl_2$ (A_w = 0.32), K_2CO_3 (A_w = 0.43), Ca $(NO_3)_2$ (A_w = 0.56), NaCl (A_w = 0.75), KCl (A_w = 0.85), $BaCl_2$ (A_w = 0.90) and K_2SO_4 (A_w = 0.97) (Greenspan, 1977). In addition to these salts, distilled water and the desiccant (silica gel) were used for aw of 1 and 0, respectively. The saturated solutions were transferred into desiccators (Pyrex, Newell, France). Ten desiccators were used to serve as a closed chamber for the experiment. The relative humidity is fixed by contact with saturated salts solution whose water vapor pressure at a given temperature is perfectly known. This relative humidity of salts solution and temperature were monitored using a thermo-hygrometer (Haar-Synth. Hygro, Germany). Thymol was placed in the desiccators containing NaCl and $BaCl_2$ in order to prevent the growth of mould (Klewicki *et al*.; 2009).

Triplicate flour samples (0.5 ± 0.0023 g) previously dried in an vacuum oven at 45 °C for 24 hours, were put into small crucibles of aluminum foil and placed in the desiccators which were then tightly closed. The samples were weighed within interval of 24 hours and allowed to equilibrate until there was no discernible weight change, as evidence by constant weight values (± 0.002g). The total time required for removal, weighing and replacing samples in desiccators was 15-25 second. This minimized the degree of atmospheric moisture sorption during weighing. The dry mass was determined gravimetrically. Moisture content of the equilibrated samples was determined by the vacuum oven method (Karmas, 1980). The time required for equilibrium was one or six weeks depending on water activity and sample nature.

Modeling equations

Guggenheim-Anderson-De Boer (GAB) model is considered to be the most versatile and the best one for fitting the sorption data for the majority of food products in a water activity range of 0-0.90 (Mayor *et al*., 2005). Its use is recommended by the European COST 90 project (Yan *et al*., 2008). The major advantages of the GAB model are the following: it has a viable theoretical background since it is a further refinement of Langmuir and BET theories of physical adsorption, it provides a good description of the sorption behaviour of almost every food product, its parameters have a physical meaning in terms of the sorption processes (Al-Muhtaseb *et al*., 2002). The water content sorption isotherms were fitted using the Guggenheim-Anderson-De Boer (GAB) model, as follows:

$$M = \frac{M_0 * C * K * A_w}{(1 - K * A_w)(1 - K * A_w + C * K * A_w)} \quad (1)$$

where Aw represents the equilibrium relative humidity in decimal, M is the equilibrium moisture content (g / 100 g dry matter), M_0 is the monolayer moisture content (g / 100 g dry matter), C (Guggenheim's constant), K (correction factor), are sorption isotherm constants specific to equation. Equation (1) can be transformed into equation (2) (Hailwood and Horrobin, 1946; Abramovič et Klofutar, 2002):

$$M = \frac{A_w}{(\alpha * A_w^2 + \beta * A_w + \sigma)} \quad (2)$$

The isotherm fitting were obtained by extrapolation using the single-hydrate sorption model (Hailwood and Horrobin, 1946). The data were transformed by dividing the water activity with the equilibrium moisture contents (M) earlier. The three constants α, β and σ, were readily determined by a least-square regression of this second degree polynomial. Least-square regression analysis was used to calculate the respective constants using software like, Microsoft excel 2000 (Microsoft Corp., USA). A quadratic curve of second degree polynomial function was obtained from the data points. A graphical illustration of the ratio A_w/M versus A_w, using the parabolic equation was shown and; these three constants (α, β and σ) and the coefficient of determination (R^2) were obtained. From the parameters α, β, σ, the values of K, C and M_0, were calculated through the following relations (Chen et Jayas, 1998):

$$K = \frac{\sqrt{\beta^2 - 4 * \alpha * \sigma} - \beta}{2 * \alpha} \quad (3)$$

$$C = 2 + \frac{\beta}{\alpha * K} \quad (4)$$

$$M_0 = \frac{\beta}{\alpha * K * C} \quad (5)$$

To evaluate the goodness-of-fit for the models (GAB), the coefficient of determination (R^2), Mean Relative Error (MRE) and the Mean Square of Error (MSE) were used. MRE (%) and MSE were calculated as follows (Akanbi *et al.*; 2006):

$$MRE = \frac{100}{N} \sum_{i=1}^{N} \left| \frac{M_{i,e} - M_{i,p}}{M_{i,e}} \right|$$

$$MSE = \sqrt{\frac{\sum_{i=1}^{N} (M_{i,e} - M_{i,p})^2}{d_f}}$$

Where $M_{i,e}$ is the ith experimental equilibrium moisture content (EMC) value, $M_{i,p}$ is the ith predicted EMC value and N is the number of experimental data. "df" is the degree of freedom of the fitting equation. The number of degree of freedom as follows *N-np* where *N* is the number of data points and *np* is the number of parameters. While the value of R^2 close to 1, and that of RMSE close to 0, indicate a better fit, MRE (%) below 10 % to be indicative of a good fit for practical purposes (Lomauro *et al.*, 1985).

Properties of bound water

The physical state of water adsorbed by foods actually determines the actual spoilage. It is therefore essential to generate information related to various aspects of bound water namely its relation to surface area of adsorbent, number of adsorbed monolayer etc. In order to evaluate various parameters, describing the properties of water uptake (Bajpai and Pradeep, 2013). The surface area of adsorbent δ (m^2/g) was determined from the monolayer moisture content, using the following relationship (Mazza and Maguern, 1978).

$\delta = (A_{H2O} N_{Avogadro} M_0)/M_{H2O} = 3530 M_0$

δ = the specific surface of the solid in (m^2/g d. b.); M_0 is the moisture content of the monolayer in (g/100g d. b.); M_{H2O} is the molar mass of water (18 g/mol); $N_{Avogadro}$ is Avogadro's number ($6*10^{23}$ molecules / mol) and A_{H2O} is the water molecule surface area ($1.06 * 10^{-19} m^2$). The Caurie slope (S), can be used to evaluate surface area δ ($m^2 g^{-1}$) using following expression (Cervenka *et al.*, 2008).

δ = 54.54/ S

The number of adsorbed monolayer (N), was obtained by the formula

S = 2/N

of the previous two formulas we deduce:

N = 2δ/54.54

RESULTS AND DISCUSSION

Equilibrium moisture contents

All adsorption isotherms presented demonstrated an increase in the equilibrium water content along with an increase in A_w (figure 1). The typical shape of an isotherm reflects the way in which the water binds the system. Weaker water molecule interactions generate a greater water activity, thus, the product becomes more unstable. Water activity depends on the composition, temperature and physical state of the compounds (Fabra *et al.*, 2009). The adsorption isotherms had a similar shape; one characteristic of material containing considerable quantities of sugars (Moraga *et al.*, 2004). They showed a relatively slow increase in water content for A_w values ≤ 0.32, a faster increase for average values of A_w ≥ 0.32, and a rapid increase for A_w ≥ 0.75.

The isotherm are typically divided into three regions:

- the "monolayer region" (A_w = 0 - 0.32) represents strongly bound water, and the enthalpy of vaporization is considerably higher than the one of pure water. The bound water includes

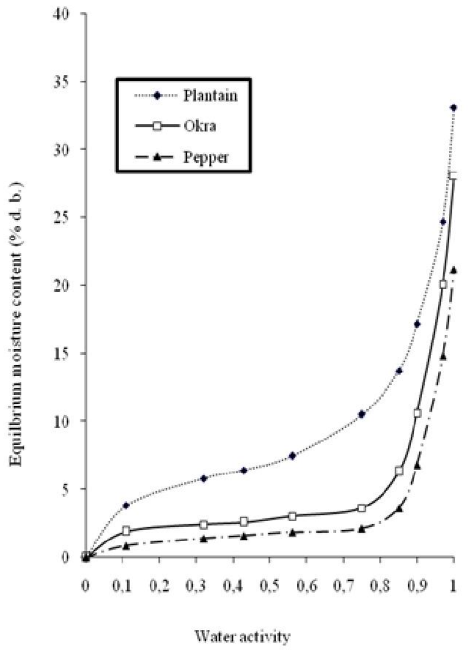

Figure 1. Comparison of experimental curves of the adsorption isotherm of plantain, okra and peppers flour

structural water (H-bonded water) and monolayer water, which is sorbed by the hydrophilic and polar groups of food components (polysaccharides, proteins, etc.). Bound water is unfreezable and it is not available for chemical reactions or as a plasticizer. The relatively slow increase in water content for A_w values ≤ 0.32 is due to the weak attraction between water and constituents of these flours. According to Ferradji *et al.* (2008a), the low water adsorption is explained by the fact that water vapor is adsorbed by the hydroxyl groups of crystalline carbohydrates.

- the "multilayer region" (A_w = 0.32 - 0.75), water molecules bind less firmly than in the first zone, they usually present in small capillaries. The vaporization enthalpy is slightly higher than the one of pure water. This class of constituent water can be looked upon as the continuous transition from to free water. A faster increase for average values of A_w ≥ 0.32 which is due to the high capillarity; typical for most food products (Yué and Tano, 2008).

- the "condensed water region" (A_w = 0.75 to 1), the properties of water in this region are similar to those of the free water that is held in voids, large capillaries, crevices; and the water in this region loosely binds to food materials (Tano *et al.*, 2008). A rapid increase for A_w ≥ 0.75 can be explained partly by the dissolution of carbohydrates and secondly by the passage of carbohydrates from the crystalline form to amorphous form. Anything that increases the water content because this phase transition carbohydrates increases the adsorption sites number (Saravacos *et al*., 1986; Ferradji *et al.*, 2008a).

The experimental adsorption isotherms of banana, okra and bell peppers flour were classified in hygroscopicity order descending following: plantain, okra and bell pepper (Figure 1). The sorption isotherms had a sigmoid sharp (type II) which is common for many hygroscopic products (Kane *et al*., 2008). These results are similar to those of Johnson and Brennan (2000) and Medeiros *et al.* (2006) who worked respectively on the sorption of cocoa powder and chocolate. This sigmoid shape is characteristic of food products containing sugar which absorbs small water content for low water activities and high water content for high water activity (Yué and Tano, 2008). These results also corroborate those of Goula *et al.* (2008) who have worked on the sorption isotherm of tomato powder. At a constant water activity, plantain flour had higher equilibrium moisture content than okra and pepper, indicating its higher hygroscopicity. This can be explained by the fact that plantain is rich in carbohydrate as okra and bell pepper, which promoting high water adsorption. This result is similar to Ferradji *et al.* (2008a) works, which stipulated that products rich in carbohydrates are more hygroscopic than those with low levels of carbohydrates.

Fitting to sorption models

The GAB's model was used to predict the value of the equilibrium moisture content and monolayer moisture content. The sorption relationships detailed in table 1 were fitted to the experimental data for all samples. The moisture content models were compared according to their coefficient of determination (R^2), mean relative error (MRE) and mean square of error (MSE). 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 sorption process. Using these coefficients, the sorption isotherms of plantain, okra and pepper flour were predicted by GAB. The representation of these results is shown in figure 2 to 4 from which it can be noted that the predicted curve by GAB's model and the experimental data had practically the same rate. The predicted curves (Figures 2, 3 and 4) had a sigmoid shape (type II) for all three flour. The mathematical models tested to estimate equilibrium moisture content of plantain; okra and bell pepper flour presented values of determination coefficient (R^2) above 85% (Table 1). The GAB model is applicable to all three flour. This agrees with work of Akanbi *et al.* (2006) who states that the GAB's model is applicable if R^2 ≥ 0.85. It should be noted that only the high value of R^2, is not enough to appreciate the goodness of fit of the model.

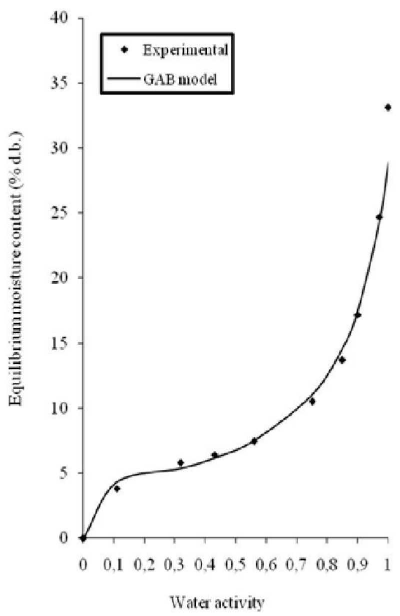

Figure 2. Comparisons of experimental and predicted adsorption isotherms of plantain flour using GAB

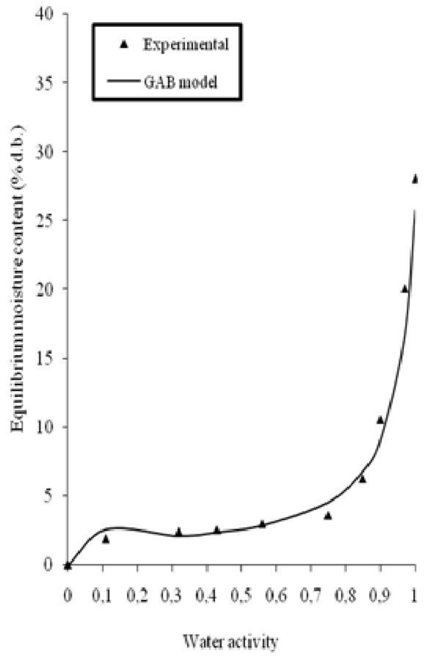

Figure 3. Comparisons of experimental and predicted adsorption isotherms of okra flour using GAB

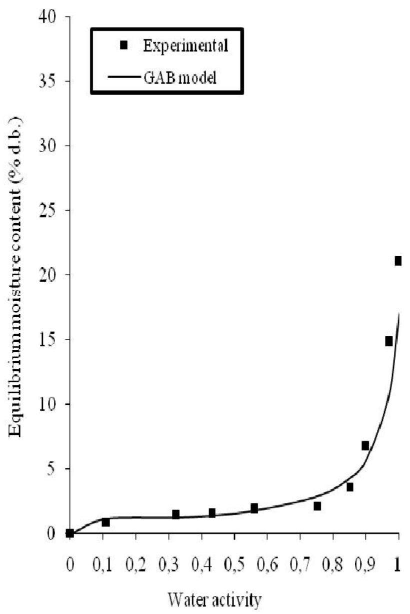

Figure 4. Comparisons of experimental and predicted adsorption isotherms of pepper flour using GAB

There must also be low MRE and MSE values. Low MSE (1.2029, 4.4116 and 8.8548) and also low MRE (%) (1.3873, 2.6242 and 3.764) in ascending order for plantain , okra and pepper respectively shows good fitting experimental curves (Arévalo - Pinedo *et al*., 2004; Talla, 2012). The GAB model was applicable to all three flour (plantain, okra and pepper). But more applicable to the plantain flour (MRE and MSE of plantain flour were lower than those two flour). This assertion is justified by the works of Ferradji *et al.* (2008b) on the adsorption isotherm dates and dried potatoes. It is also consistent with the work of Farahnaky *et al.* (2009) on the modeling of adsorption figs and potatoes. These results are similar to the work of Ferradji *et al.* (2008a) on the adsorption isotherm dates and those of Oluwamukomi (2009) on the modeling of sorption isotherm of *garis.*

Table 1. Estimated GAB model coefficients, R^2, MRE and MSE fitted to the adsorption isotherms of plantain, okra and bell pepper flour

		Plantain	Okra	Pepper
GAB	M_0	3.852	1.2969	0.7972
	δ	13597.56	4578.057	2814.116
	N	498.627	167.878	103.195
	C	2.0786	3.6647	2.3877
	K	0.8666	0.9496	0.9514
	MSE	1.2029	4.4116	8.8548
	MRE	1.3873	2.6242	3.764
	R^2	0.991	0.993	0.990

The mean square of error (MSE) and the mean relative error of the three flours evolve in the same direction. This result is similar to those of Jamali *et al*. (2006) and Farahnaky *et al.* (2009) who worked on the sorption isotherm of leaves of *Citrus reticulata* and the effect of glycerol on the sorption isotherm of figs, respectively. The three flours had not the same monolayer moisture content (M_0). Plantain flour (M_0 = 3.852 $gH_2O*100g^{-1}$) was higher than okra (M_0 = 1.296 $gH_2O*100g^{-1}$) and pepper flour (M_0 = 0.797 $gH_2O*100g^{-1}$) (Table 1). Confirming the hygroscopicity of three flours. Fitting of this model to results is of particular value given the physical significance of the parameters. The monolayer moisture content is the water content to which any available hydrophilic sites are linked to the first monolayer to the water surface of the adsorbent (Ferradji *et al*., 2008b). It represents the maximum water content below which is not available for chemical and biochemical reactions. This parameter is important for the control of product stability during storage (Ferradji and Malek, 2005). It is important to know the monolayer capacity because**,** the corresponding water content of the material is considered optimal from the point of view of food stability; and allows proper storage conditions (A_w) to be selected (Falade *et al*.; 2003). The monolayer moisture content M_0 is recognized as the moisture content according the longest time period with minimum quality loss at given temperature.

It corresponds to the amount of moisture adsorbed by a single layer to the binding sites in the product. The value of monolayer moisture content of a product gives an indication of total number of polar groups binding water and the level of hydration, at which the mobility of small molecules become apparent (Dincer and Esin, 1996). These different values of monolayer moisture content of plantain, okra and pepper flour correspond to A_w = 0.11, 0.08 and 0.11 respectively. These values are lower than A_w = 0.6 which is the minimum for the development of micro-organisms such as yeasts, molds. For most dry product, the rate of quality loss due the chemical reaction is negligible below the monolayer value (Labuza *et al*., 1985). These values are particularly important in storage of the product, since level the water does not act as a solvent, being biologically inert. But oxidation of lipids may be observed in those below $A_w \leq 0.2$ (Mark *et al*., 2004). Lipid oxidation is often the limiting of conservation of certain food dehydrated factor. As rancidity is a major spoilage reactions with low or medium water content can be observed even for A_w = 0 to 0.2 (Pierre and Thomas, 2006). Thus, okra with high lipid levels, susceptible to undergo this type of deterioration for $A_w \leq 0.1$. This result is in agreement with the work of Peter and Thomas (2006) which stated that for $A_w \leq 0.1$, the risk of lipid oxidation are very high. From the foregoing, it would be desirable to keep these flours in A_w between 0.4 and 0.6, where water molecules are less strongly bound. This corresponds to the water content between 6.4 and 7.45% for the plantain; 2.57 and 3% for okra and 1.6 and 1.86% for the pepper. These water contents in all respect, the recommendations of moisture for the conservation of tropical products (≤ 7%) (Lovett, 2004). The constants C and K, which relate to the interaction energies between the water and food, also vary according to the method of regression used. The polynomial regression gave the highest values of C (2.0786, 3.6647 and 2.3877 for plantain, okra and pepper respectively), but the values of K were smaller (0.8666, 0.9496, 0.9514 for plantain, okra and pepper respectively).

Properties of bound water

The specific surface is very important in determining the water binding properties of material particles, including the monolayer moisture content. The surface area of some polysaccharides was determined (Robitzer *et al*., 2011), 200 m^2g^{-1} for kappa Carrageenan, 320 m^2g^{-1} for Agar, etc. It can be seen the values obtained in the present study are significantly higher. It is reported that large surface area of these three foods is due to existence of intrinsic micro porous structures in the material. It appears that okra and pepper does not exhibit porosity largely compared to that of plantain (table 1). Using the mathematical model, one can analyze the contribution of tightly adsorbed water and solution water in the sample. It is clear from Figure 1 that the contribution of solution water is small at low water activities but at higher aw values (> 0.75), the contribution is significant for all the samples.

Conclusion

The adsorption isotherms provide valuable informations about the equilibrium moisture content of plantain, okra and pepper flour. They present a clear idea on the stability of these flour after drying, as well as information on the different kind of water in the product. So, these curves are valuable for storage of plantain, okra and pepper flour. The adsorption isotherms of plantain, okra and pepper flour had been determined by experiment and then described by GAB. The experimental results show that the adsorption isotherms of these three flour taked a form of the sigmoid type (II) and that GAB's model gave a better fit for the three adsorption isotherms of banan, okra and pepper flour. GAB's model was also used to determine the monolayer moisture content of plantain , okra and pepper flour and the values of the constants C and K.

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