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Assessment of adsorption isotherms of three plantain flours (*Musa paradisiaca* L. var. Horn 1, FHIA 21 and PITA 3) and cassava flour (*Manihot esculenta* Crantz var. Bonoua 2)

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ABSTRACT

Adsorption isotherms constitute an important source of information for establishing the stability of food products and their storage conditions. The aim of the work is to help reduce post-harvest losses of plantain and cassava in Côte d'Ivoire by manufacturing new products based on plantain and cassava flours. To achieve this objective, the static gravimetric method was used to determine the various experimental adsorption isotherms for the flours of three plantain varieties (Horn 1, PITA 3, FHIA 21) and cassava flour (Bonoua 2). They were modelled using the GAB equation. Adsorption isotherms were determined at a temperature of 28 ± 1°C in a water activity range (aw) of 0.11 to 0.90. The study showed a decreasing hygroscopicity for the four flours in the following order: Horn 1, PITA 3, FHIA 21 and cassava flour. The adjustment of the experimental values by the GAB model made it possible to estimate the various values of the monolayer. The water content of the monolayer (X_o) of the different flours (X_o (Horn 1) = 14.23 g H_2O / 100 g, X_o (PITA 3) = 12.29 g H_2O / 100 g, X_o (FHIA 21) = 11.40 g H_2O / 100 g and X_o (Bonoua 2) = 9.49 g H_2O / 100 g dry matter) corresponds to relative humidities of 11%, 11%, 10% and 11% respectively. The model fitted the four flours correctly, with correlation coefficients close to 1 and mean relative error of deviation values below 10 %. These values are optimal for ensuring adequate storage conditions for the different flours.

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INTRODUCTION

Agricultural production in most West and Central African countries consists mainly of food crops such as yams, cassava, plantain, taro, fruit and vegetables, etc. These crops have always formed the staple diet of these populations (Gire, 1994; Djimo, 2010). Plantain and cassava are staple products in the diet of people living in lowland areas of humid tropical Africa (Ngalani and Crouzet, 1995). Plantain and cassava contribute to food security. They also represent a substantial source of income for rural and urban populations. These products are highly perishable, leading to huge post-harvest losses, sometimes estimated at over 40% (ANADER, 2013, Brou *et al.*, 2017). Storing plantain and cassava fresh in tropical countries is difficult because of their highly perishable nature (Talla, 2012).

Thus, one of the alternatives for reducing post-harvest losses is to process plantain and cassava into finished products (flour), often called transfo-conservation.

As with the majority of products processed into flour, preservation is one of the major constraints on their biological stability, which must be ensured. Moisture affects the physical, biochemical, microbiological and rheological characteristics of foods, as well as their stability (Lewicki, 2004). However, water activity (aw) has long been considered one of the most important quality factors, particularly for long-term storage. All chemical and microbial deterioration is directly affected by changes in water activity. One of the key factors in controlling the storage conditions of these processed foods is the determination of their adsorption isotherm curves (Kouhila *et al.*, 2004). The sorption isotherm of foods is therefore of great importance in food technology.

It is therefore necessary to contribute to improving the quality and stability of plantain and cassava flours.

The general objective of this study is to determine the adsorption isotherm curves for the flour of three varieties of plantain and cassava in order to preserve their quality during storage. Specifically, the aim is to:

1. experimentally determine the adsorption curves for plantain flour of the Horn 1, PITA 3 and FHIA 21 varieties and for cassava flour (Bonoua 2),
2. to model these adsorption curves using the GAB equation (Guggenheim, Anderson and de Boer).

MATERIALS AND METHODS

Plant material

The plant material consisted of plantain (*Musa paradisiaca* L.) bunches of the Horn 1, FHIA 21 and PITA 3 varieties and cassava (*Manihot esculenta* Crantz) of the Bonoua 2 variety. The bunches of the Horn 1 variety were harvested in Taboitien (Tiassalé, a town about 125 km from Abidjan, latitude 5°53′54″N and longitude 4°49′42″W (Côte d'Ivoire). The bunches of the FHIA 21 and PITA 3 varieties were harvested at Azaguié gare (Azaguié, a town about 45 km from Abidjan, at latitude 5°37'40 ‘N and longitude 4°5'12 ’W (Côte d'Ivoire). Both towns are located in the Agnéby-Tiassa region. The cassava (Bonoua 2) was purchased at the Gonzagville market in the commune of Port-Bouet (Abidjan, Côte d'Ivoire) at latitude 5°14′24″N and longitude 3°53′47″W.

Adsorption isotherm for Horn 1, FHIA 21, PITA 3 plantain flours and Bonoua 2 cassava flour

The adsorption isotherms of the four flours were determined by the standard static gravimetric method recommended by the European COST 90 project (Wolf *et al.*, 1985) at a temperature of 29 ± 1°C. The method is based on the use of saturated salt solutions to maintain the fixed relative humidity corresponding to the activity of the water inside the jars.

The relative humidity of the salt solutions and the temperature were measured using a thermohygrometer (Haar- Synth. Hygro, Germany). Eight salts were selected to give different water activities in the range 0.11 to 0.90. The corresponding water activity values were as follows: LiCl (aw = 0.11), $MgCl_2$ (a_w = 0.32), K_2CO_3 (a_w = 0.43), $Ca(NO_3)_2$ (a_w =

0.56), $SrCl_2$ (a_w =0.69), NaCl (a_w = 0.75), KCl (a_w = 0.85) and $BaCl_2$ (a_w = 0.90) (Greenspan, 1977).

In addition to these salts, desiccant (silica gel) was used for aw = 0. The water activities of the respective saturated solutions (aw = 0.11- 0.90) were based on the values given by Greenspan (1977) and Bell and Labuza (2000). Duplicate samples, each weighing 1±0.0023 g and previously dried for adsorption in an oven at 45°C for 24 h, were weighed into cups using an analytical balance (Sartorius, Goettingen, Germany) accurate to 0.0001 g and placed in jars containing saturated salt solutions prepared according to the recommendations of Labuza (1984) and the COST 90 project (Wolf *et al.*, 1985). The samples were weighed at 48-hour intervals and allowed to equilibrate until there was no further change in weight. The time required for weighing and replacing the samples in the jars was 15 to 25 seconds (Hossain *et al.*, 2001; Kaymak-Ertekin and Gesik, 2004).

The dry mass was determined gravimetrically. The water content of the samples was determined after each adsorption experiment using an electronic moisture meter (METTLER TELEDO, France), at 150°C for 5 to 12 minutes until a constant weight was obtained. The mean values of two replicates were obtained for each experiment. To establish the sorption isotherms, the equilibrium water contents of the product were plotted as a function of water activity at constant temperature.

Modelling the adsorption isotherms of the various samples

The GAB (Guggenheim, Anderson and de Boer) model, which is the most widely used and has a large area of validity, is used in this study. This model is one of the best theoretical models for characterising food sorption curves (Ferradji *et al.*, 2008a). It is used and accepted in food technology by most researchers (Ferradji *et al.*, 2008b). The applicability of this model will be assessed using the following criteria: correlation coefficient (r), relative mean error of deviation (MRE).

The correlation coefficient (R) is one of the first criteria used to assess the smoothing of experimental curves by this model. The model is smoother when r is close to unity (1) (Vega-Galvez and Lemus-Mondaca, 2008). In addition to r, the MRE (%) and is also used to assess the fit of the model.

The GAB model is in the following form (Labuza *et al.*, 1985; Oluwamukomi, 2009):

$$X=(X_o \times C \times K \times a_w)/\{(1-K \times a_w)(1-K \times a_w+C \times K \times a_w)\} \quad (1)$$

X = equilibrium water content

X_o = water content of the monolayer

a_w = water activity

C and K are the constants of the GAB model

The three GAB parameters, namely C, K and X_o derive from the second degree polynomial form (equation 1), which was solved by multilinear regression analysis to obtain graphically the constants α, β and σ.as well as R and MRE (Jouppila and Roos, 1997; Abramovič and Klofutar, 2002):

$$X_{aw}=\alpha a^2_w+\beta a_w+\gamma \quad (2)$$

where : $\alpha = K/Xo(1/C - 1)$;

$\beta = 1/ X_o(1 - 2/C)$;

$\gamma = 1/ X_oKC$

The values of the three constants X_o, C and K are obtained by the following relationships :

$K = f^{1/2} - \beta / 2\gamma$ where $f = \beta^2 - 4\alpha\gamma$

$X_o = 1 / (\beta + 2K\gamma)$

$C = 2 + (\beta / K\gamma)$

To assess the goodness of fit of the model (GAB), the correlation coefficient (R) and the mean relative error (MRE) were used. The correlation coefficient was calculated using the following equation:

$$R = \frac{\sum_{i=1}^{N} (X_{exp} - \overline{X_{exp}})(X_{theo} - \overline{X_{theo}})}{\sqrt{\sum_{i=1}^{N} (X_{exp} - \overline{X_{exp}})^2 (X_{theo} - \overline{X_{theo}})^2}} \quad (3)$$

Where: X_{exp} = experimental value of equilibrium water content

$\overline{X}_{exp}$ = average value of experimental equilibrium water contents

X_{theo} = predicted equilibrium water content value

$\overline{X}_{theo}$ = predicted mean value of equilibrium water content

N = number of experimental observations

The relative mean error of deviation (MRE) is determined according to the following relationship (Kouhila *et al.*, 2004 ; McMinn *et al.*, 2007; Tonon *et al.*, 2009):

$$MRE = \frac{100}{N}\sum_{i=1}^{N}\left|\frac{X_{exp} - X_{theo}}{X_{exp}}\right| \quad (4)$$

Where: X_{exp} = experimental value of equilibrium water content

X_{theo} = value of predicted equilibrium water content

N = number of experimental observations

RESULTS

Experimental adsorption isotherm curves for plantain flour (FHIA 21, PITA 3, Horn 1) and cassava flour (Bonoua 2)

Fig. 1 shows a comparison of the experimental adsorption isotherm curves for Horn 1, FHIA 21, PITA 3 plantain flour and cassava flour (Bonoua 2). The curves are classified in the following decreasing hygroscopic order: Horn 1 flour, PITA 3 flour, FHIA 21 flour and cassava flour. The four curves have a sigmoidal shape and present three zones. They are typical of the type II isotherm. The three zones are as follows:

Zone I (aw = 0 to 0.32), shows a slight increase in water content of 6.98 g/100g dry matter (DM); 5.2 g/100g DM, 4.42 g/100g DM and 4.14 g/100g DM for a variation in water activity (aw) equal to 0.32 for plantain flour Horn 1, FHIA 21, PITA 3 and cassava flour respectively (Fig. 1).

Zone II (aw = 0.32 to 0.75) shows an average increase in water content of 24.38 g/100g DM; 20.92 g/100g DM, 24.38 g/100g DM and 18.5 g/100g DM respectively for plantain flour Horn 1, PITA 3, FHIA 21 and cassava flour for a variation in aw equal to 0.43 (Fig. 1).

Zone III (aw = 0.75 - 0.9), shows an exponential increase in water content of 37.18 g/100g DM; 41.48 g/100g DM, 39.98 g/100g DM, and 33.92 g/100g DM respectively for plantain flour Horn 1, PITA 3, FHIA 21 and cassava flour for a variation in aw equal to 0.15 (Fig. 1).

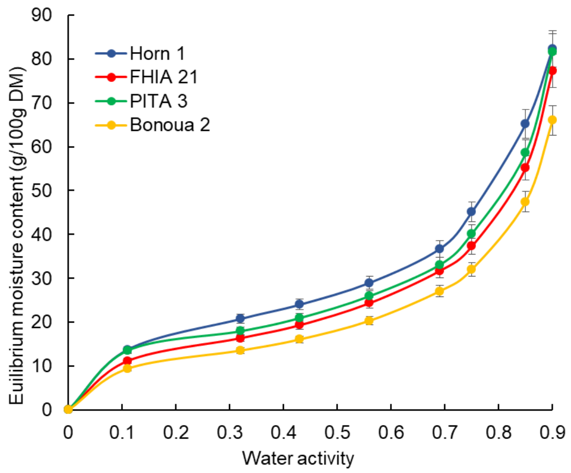

Fig. 1. Comparison of experimental adsorption isotherm curves for Horn 1, FHIA 21 and PITA 3 plantain flours and Bonoua 2 cassava flour

However, at relative humidities (RH) of 75% or less, the flours adsorb a moderate amount of water, i.e. 45.12, 40.18, 37.3 and 32.06 g/100 g of dry matter respectively for plantain flour Horn 1, PITA 3 FHIA 21 and cassava flour. Similarly, at RH between 75% and 85%, the products adsorb significant quantities of water, i.e. 65.24, 58.56, 55.22 and 47.46 g of dry matter respectively for Horn 1, PITA 3 FHIA 21 plantain flour and cassava flour.

On the other hand, at RH above 90%, these four products adsorb a large quantity of water, i.e. 82.3; 81.66; 77.28; 65.98 g of dry matter for Horn 1 plantain flour, PITA 3 FHIA 21 and cassava flour respectively (Fig. 1).

The four products are classified in the following descending hygroscopic order: plantain flour Horn 1, PITA 3, FHIA 21 and Bonoua 2 cassava flour.

Modelling of experimental adsorption isotherm curves for plantain flour (Horn 1, FHIA 21, PITA 3) and cassava flour (Bonoua 2)

The GAB (Guggenheim, Anderson and de Boer) model was used to adjust the experimental water content of the four products, enabling comparisons to be made between the experimental curves and those predicted.

The different theoretical water contents were used to construct the theoretical adsorption isotherm curves for the four products studied. Comparison of the experimental and theoretical adsorption isotherm curves shows that, in general, they are all superimposable, or they coincide for each (Figs 2-5).

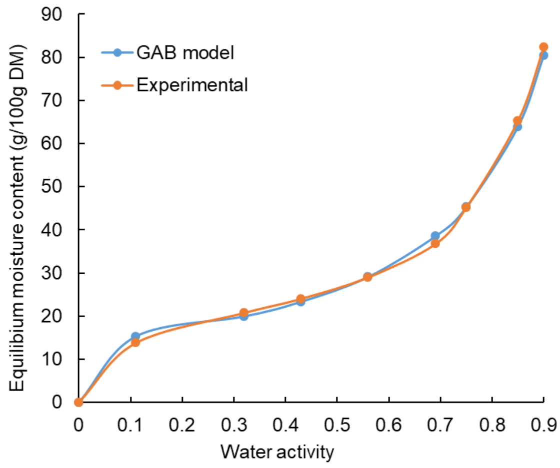

Fig. 2. Comparison of experimental and theoretical adsorption isotherm curves for Horn 1 plantain flour

Fig. 2 shows the experimental and theoretical adsorption isotherm curves for plantain flour (Horn 1). It can be seen that over the whole range of water activities, the theoretical and experimental curves are superimposable, or they coincide. However, at aw between 0 - 0.32 and 0.65 - 0.75, the equation is overestimated because the theoretical curve is above the experimental curve.

The experimental and theoretical adsorption isotherm curves for PITA 3 plantain flour are shown in Fig. 3. The experimental and theoretical adsorption isotherm curves for PITA 3 flour are almost superimposable over the whole range of water activities. However, at aw between 0.65 and 0.75, the equation is overestimated because the theoretical curve is slightly above the experimental curve. But at aw above 0.85, the experimental curve is above the theoretical curve, so the equation is underestimated (Fig. 3).

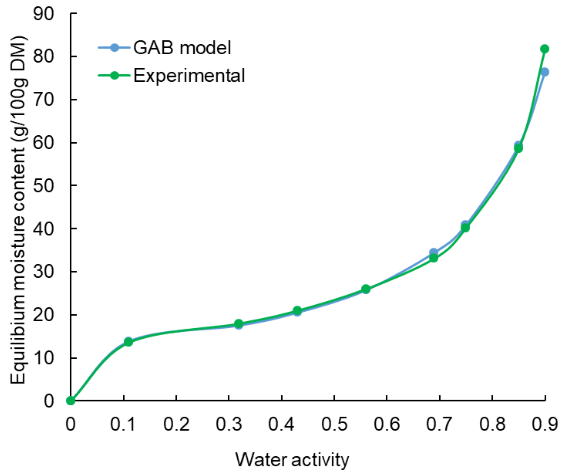

Fig. 3. Comparison of experimental and theoretical adsorption isotherm curves for PITA 3 plantain flour

Regarding Fig. 4, which presents the experimental and theoretical adsorption isotherm curves for FHIA 21 plantain flour, shows that the experimental curves are practically identical over the entire range of water activities. However, there is a slight difference in water activities between 0.0 and 0.32 and between 0.75 and 0.85, where the model is overestimated because the theoretical curve is slightly above the experimental curve.

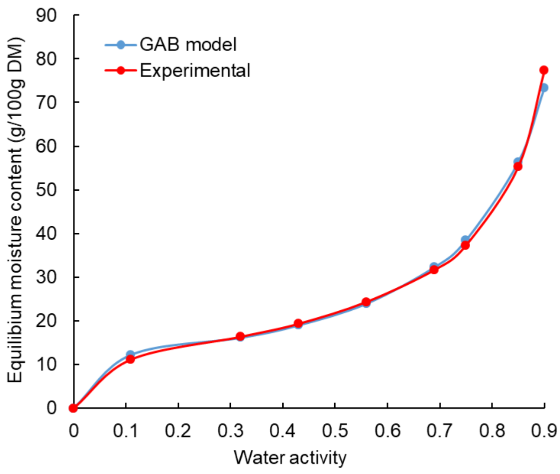

Fig. 4. Comparison of experimental and theoretical adsorption isotherm curves for FHIA 21 plantain flour

The experimental and theoretical adsorption isotherm curves for cassava flour (Bonoua 2) are superimposable over the full range of water activities, or are almost identical (Fig. 5).

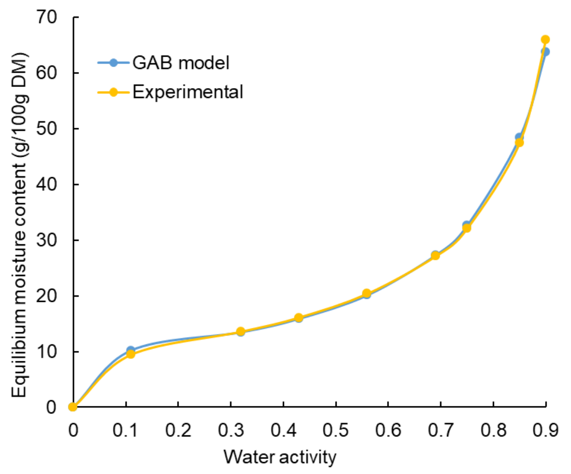

Fig. 5. Comparison of experimental and theoretical adsorption isotherm curves for Bonoua 2 cassava flour

The criteria for assessing the applicability of the GAB model, such as the correlation coefficient (r) and the relative mean error of deviation (RME (%)) of the four flours, calculated are shown in Table 1. Analysis of these criteria shows that the correlation coefficient for plantain flour, Horn 1 is r = 0.999, for FHIA 21 (r = 0.998), for PITA 3 (r = 0.997) and for cassava flour is r = 0.999. The relative mean error of plantain flour PITA 3 (MRE (%) = 0.1403), that of plantain flour Horn 1 (MRE (%) = 0.0454), that of plantain flour FHIA 21 (MRE (%) = 0.0443) and that of cassava flour (Bonoua 2) (MRE (%) = 0.0158) and evolve respectively in this decreasing order of classification.

With regard to the GAB parameters (X_0, K and C) shown in Table 1, we note that the four products do not have the same values for monolayer water content X_0. The monolayer water content represents the maximum amount of water below which water is not available for chemical and biochemical reactions.

In fact, the highest monolayer water content (X_0) of the four flours is that of Horne 1 flour (X_0 = 14.235 g/100g dry matter) followed by PITA 3 flour (X_0 = 12.28 g/100g dry matter) then FHIA 21 flour (X_0 = 11.408 g/100g dry matter) and finally cassava flour (X_0 = 9.49 g/100g dry matter). These water contents in the monolayer of the four flours, namely Horn 1, PITA 3, FHIA 21 and cassava, correspond to water activities of 0.11, 0.10, 0.11 and 0.11 respectively. This parameter is important for controlling product stability during storage.

Table 1. Estimated parameters and constants of the GAB model for the four flours (Horn 1, PITA 3, FHIA 21 and Bonoua 2)

Model		Horn 1	PITA 3	FHIA 21	Bonoua 2
G A B	X_0	14,2351	12,2829	11,4089	9,4925
	C	256,0216	873,1251	233,5088	222,7406
	K	0,9146	0,9324	0,9384	0,9459
	MRE(%)	0,0454	0,1403	0,0443	0,0158
	R	0,9992	0,9978	0,9983	0,9992

The values of the C parameters of the GAB equation in Table 1 make it possible to characterise the sorption isotherms. Horn 1 flour (C = 256.0216), PITA 3 flour (C = 873.1251), FHIA 21 flour (C = 233.5088) and cassava flour (C = 222.7406) are type II adsorption isotherms. All four flours have K values significantly less than or equal to 1. The values for the four flours are 0.9146, 0.9324, 0.9384 and 0.9459 for Horn 1, PITA 3, FHIA 21 and cassava respectively (Table 1).

DISCUSSION

Adsorption characteristics of the four flours

All adsorption isotherms demonstrated an increase in the equilibrium moisture content along with an increase in water activity. The adsorption isotherms of formulated flours of the three varieties plantain and cassava flour follow the characteristic shape of high-sugar foods according to the BET classification with the equilibrium moisture content (EMC) increasing sharply at the high water activities

(Moraga *et al.*, 2004). The adsorption isotherm curves obtained in this study are sigmoidal in shape and are type II according to the classification of Brunauer *et al.* (1938). This result is similar to those of Medeiros *et al.* (2006) on cocoa and chocolate powders and Brou *et al.* (2018) who worked on plantain flours. The sigmoidal shapes of the four flours can be explained by parameter C of the GAB model, which has values greater than 10. According to Medeiros *et al.* (2006), when C ≤ 10, the sorption isotherm is type III, and when C ≥ 10, the sorption isotherm is type II. The sigmoid curves obtained characterise agro-food products rich in sugars (Maskan and Gögüs, 1997). This characteristic is similar to that observed by many other authors on starches (Chungcharoen and Lund, 1987). The isotherm are typically divided into three zones:

The "monolayer zone" (aw= 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 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 aw 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 zone" (aw = 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 aw ≥ 0.32 which is due to the high capillarity; typical for most food products (Yué and Tano, 2008).

The "condensed water zone" (aw= 0.75 to 0.90), 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 aw ≥ 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 four flours (Horn 1, FHIA 21, PITA 3 and cassava) were ranked in descending order of hygroscopicity as follows: Horn 1 flour, PITA 3 flour, FHIA 21 flour and cassava flour. The sorption isotherms showed a pronounced sigmoid curve (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 characteristics of the moisture sorption isotherm of plantain (Musa, AAB) and on the sorption isotherm of cocoa powder and chocolate. This sigmoid shape is characteristic of food products containing sugar, which absorb a low water content for low water activity and a high water content for high water activity (Yué and Tano, 2008; Brou *et al.*, 2014). These results also corroborate those of Goula *et al.* (2008), who studied the sorption isotherm of tomato powder.

Modelling of the GAB model to the adsorption isotherms data of four flours of Horn 1, FHIA 21, PITA 3 and Bonoua 2

The GAB's model was used to predict the value of the equilibrium moisture content and monolayer moisture content. The sorption relationships were fitted to the experimental data for all samples. The moisture content models were compared according to their coefficient of correlation (R) and mean relative error (MRE). 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 Horn 1, FHIA 21, PITA 3 (plantain) and Bonoua 2 (cassava) flours were predicted by GAB. The representation of these results can be noted that the predicted curve by GAB's model and the experimental data had practically the same rate. The predicted curves had a sigmoid shape (type II) for all four flours. The mathematical models tested to estimate equilibrium moisture content of plantain; and cassava flours presented values of correlation coefficient (R) above 85%. 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 ≥ 0.85. It should be noted that only the high value of R, is not enough to appreciate the goodness of fit of the model. There must also be low MRE values. The adjustment of the GAB model is acceptable when the mean relative error (MRE) is less than 10%. The various mean relative errors of the four flours are less than 10%. In fact, all four mean relative error (MRE) values for the different flours are less than 10%. These results are similar to those of Johnson and Brennan (2000) and Farahnaky *et al.* (2009). These findings were also reported by McMinn *et al.* (2007) and Oluwamukomi (2009) regarding the modelling of the adsorption isotherm of oat flour and oat flakes in the United Kingdom and the modelling of gari in Nigeria, respectively, using the GAB equation.

The GAB model can be used to predict the equilibrium water content, the water content of the monolayer, and the enthalpies of the monolayer and multilayer bonds. The GAB model gives physical meaning to the parameter values. Thus, the water content (X_o) of the monolayer represents the amount of adsorbed water for which all available hydrophilic sites are bound to the first monolayer of water on the surface of the absorbent (Ferradji *et al.*, 2008b). In other words, it represents the maximum amount of water below which water is not available for chemical and biochemical reactions. This parameter is important for controlling product stability during storage (Ferradji and Malek, 2005). The different monolayer values determined for Horn 1, PITA 3, FHIA 21 and cassava (Bonoua 2) flours correspond to aw values of 0.11, 0.10, 0.11 and 0.11 respectively. These values are below 0.6, which is the minimum threshold for the development of microorganisms such as yeasts, moulds and bacteria (Marc *et al.*, 2004). Lipid oxidation reactions and enzymatic reactions could be observed.

CONCLUSION

The moisture adsorption isotherms of the four flours (Horn 1, PITA 3, FHIA 21 and Bonoua 2) at temperature 29±1°C under 0.11 to 0.90 water activity (aw) range were determined with standard gravimetric methods using various saturated salt solutions. The adsorption isotherms provide valuable informations about the equilibrium moisture content of Horn 1, PITA 3, FHIA 21 and cassava 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 Horn 1, FHIA 21, PITA 3 and Bonoua 2 flour. The adsorption isotherms of Horn 1, FHIA 21, PITA 3 and Bonoua 2 flour had been determined by experiment and then described by GAB. The experimental results show that the adsorption isotherms of these four flours taked a form of the sigmoid type (II) and that GAB's model gave a better fit for the four flour adsorption isotherms. Among the four flours, the Horn 1 variety of plantain is the most hygroscopic, followed by the PITA 3 variety, then the FHIA 21 variety, and finally cassava (Bonoua 2). GAB's model was also used to determine the monolayer moisture content of the four flour and the values of the constants C and K. From the results obtained, we can conclude that the sorption isotherms of the four flours follow the general shape of the type II sorption curve and that the sorption isotherms are adequately described by the G.A.B. model.

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