

Adsorption isotherms of three composites flours of plantain (*Musa spp* var. *Horn 1* (AAB), *FHIA 21* (AAAB) and *PITA 3* (AAAB)) and cassava (*Manihot esculenta* var. *Bonoua 2*)

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Abstract

Adsorption isotherms constitute a source of significant information to establish the stability of the foodstuffs and their storage conditions. The storage of flours requires the knowledge of their hygroscopicity. Adsorption isotherms of composite flours of plantain of the local variety *Horn 1* (FC) (*Musa* AAB) and the two plantains hybrids varieties *FHIA 21* (FF) (*Musa* AAAB) and *PITA 3* (FP) (*Musa* AAAB) and cassava (var. *Bonoua 2*), were determined using the standard static gravimetric method. The adsorption isotherms were determined at the temperatures of $28 \pm 1^\circ\text{C}$ under 0.11 to 0.90 water activity (a_w) range. The adsorption isotherms of three composites flours were found to be type II (S-shaped), characteristic of high sugar products. The model of GAB adjusts well the data expérimentales for the isotherm of adsorption of three studied composite flours. A higher water content was observed in *Horn 1* composite flour (FC ($74.16 \pm 0.04\text{g H}_2\text{O} / 100\text{g}$)) compared to *PITA 3* composite flour (FP ($68.38 \pm 0.01\text{g H}_2\text{O} / 100\text{g}$)) and *FHIA 21* composite flour (FF ($66.37 \pm 0.12\text{g H}_2\text{O} / 100\text{g}$)) of dry matter. The GAB monolayer moisture content (X_0) (g $\text{H}_2\text{O} / 100\text{g}$ dry matter) range from 6.086 g $\text{H}_2\text{O} / 100\text{g}$ for *FHIA 21* flour to 7.375g $\text{H}_2\text{O} / 100\text{g}$ for *Horn 1* composite flour. That of *PITA 3* composite flour was 6.443g $\text{H}_2\text{O} / 100\text{g}$. These values were optimal in order to ensure safe storage condition.

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Keywords

Plantain
Composite flour
Adsorption isotherm
GAB model
Water activity
Storage.

Introduction

The banana (plantain) represents one of the most significant cultures in the world (Odah *et al.*, 2013). Plantain constitutes a basic starch food in the population diet of the areas low altitude of wet tropical Africa (Ngalani and Crouzet, 1995). Plantain represents also a substantial source of incomes for many rural and urban populations. The annual production of plantain in Côte d'Ivoire is estimated at 1.5 million tons, which classifies it in the 3rd place of the food products behind the yam and rice (Ducroquet, 2002; FAO, 2009). The storage of fresh plantain in the tropical countries is difficult because of their very perishable character (Talla, 2012). However, storage of plantain in a fresh state causes problems limiting its availability and utilisation period. Thus, for the storage of food product over a long period, its transformation into flour proves to be necessary to reduce the post-harvest losses. In order to preserve these fruits, and make it available to consumers during the whole year, it undergoes specific technological treatments, such as drying. Drying may be an interesting method in order to

prevent fresh fruit deterioration (Elamin and Dieter, 2013). The consumption of plantain contributes to food security in developing countries such as Côte d'Ivoire. The important post-harvest losses of fruit such as plantain 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.

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 (a_w). 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 (Brou *et al.*, 2014). Water activity (a_w) has long been considered as one of the most important quality factors, especially for long-term storage. All chemical and microbial deteriorations are directly affected by changing

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water activity. Therefore, the determination of the relationship between water activity and moisture content is significant. Moisture adsorption isotherms describe the relationship between the equilibrium moisture content (EMC) and the water activity at constant temperature and pressure. For food material, these isotherms give information about the sorption mechanism and the interaction of food biopolymers with water. The moisture adsorption isotherms are extremely important in modelling the drying process, in the design and optimisation of drying equipment, in predicting shelf-life stability, in calculating moisture change which may occur during storage and in selecting appropriate packaging material (Gal, 1987). 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). Several researchers have determined water sorption isotherms for various products and temperatures and at selected ranges of water activity (Labuza, 1984; Maroulis *et al.*, 1988; McLaughlin and Magee, 1998; Gabas *et al.*, 1999). The water sorption isotherm of foods is therefore of great importance.

Empirical and semi-empirical equations have been proposed to correlate the equilibrium moisture content (EMC) with the water activity of food products. These models or equations were chosen because of their suitability for high carbohydrate foods, application over a wide range of water activities, simplicity, and ease of evaluation (Ajisegiri *et al.*, 2007; Oluwamukomi, 2008). GAB (Guggenheim-Anderson-De Boer) model has been applied most successfully to describe food isotherms up to a_w of 0.9 (Iglesias and Chirife, 1976; Van de Berg and Bruin, 1981; Labuza *et al.*, 1985) and this has also been recommended by the European Project Group COST 90 on the Physical Properties of Foods as the fundamental equation for the characterization of water sorption of food materials (Wolf *et al.*, 1985).

Therefore the objectives of this study were: (1) to investigate the moisture adsorption isotherms of three composite flours and (2) to evaluate the applicability of the GAB adsorption model for predicting the equilibrium moisture content (EMC) of the three composite flours.

Material and Methods

Material

Material used in this study is consisted of plantain formulated flours FC, FF and FP of varieties *Horn 1*, *FHIA 21* and *PITA 3* respectively, used

for the preparation of foutou. The bunches of the variety *Horn 1* were harvested in Tiassalé of latitude 5°53'54"North and longitude 4°49'42"West (Côte d'Ivoire). The bunches of the varieties *FHIA 21* and *PITA 3* were harvested in Azaguié of latitude 5°37'40"North and longitude 4°5'12"West (Côte d'Ivoire). The two towns are located in Agnéby-Tiassa area. Cassava (variety *Bonoua 2*) was purchased from the Port-bouet market (Abidjan, Côte d'Ivoire). These three formulated flours are made up each one of a mixture 60% of ripe plantain and 40% of cassava. The three composite flours were produced at the laboratory of the Institut of Ivorian Tropical Technology (I2T) Abidjan, Côte d'Ivoire.

Adsorption isotherm of the formulated flours of FC, FF and FP

The adsorption isotherms of the three plantain flours were determined by the standard static gravimetric method standardised in the European COST project (Wolf *et al.*, 1985) at temperatures of $28 \pm 1^\circ\text{C}$. The method is based on the use of saturated salt solutions to maintain a fixed relative humidity which corresponds with the tissue's water activity. 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. This relative humidity of salts solution and temperature were monitored using a thermohygrometer (Haar-Synth. Hygro, Germany). Eight salts were selected to give different water activities in the range of 0.11-0.90. The corresponding values of water activities were: Lithium chloride (LiCl) ($a_w = 0.11$), Magnesium chloride (MgCl_2) ($a_w = 0.32$), Potassium carbonate (K_2CO_3) ($a_w = 0.43$), Calcium nitrate ($\text{Ca}(\text{NO}_3)_2$) ($a_w = 0.56$), Selenium chloride (SrCl) ($a_w = 0.69$), Sodium chloride (NaCl) ($a_w = 0.75$), Potassium chloride (KCl) ($a_w = 0.85$), and Baryum chloride (BaCl_2) ($a_w = 0.90$) (Greenspan, 1977). In addition to these salts, the desiccant (silica gel) was used for a_w of 0. The water activities of the respective saturated solutions ($a_w = 0.11 - 0.90$) were based on the values given by Greenspan (1977) and Bell and Labuza (2000). Duplicate samples, 1 ± 0.0023 g each previously dried in an vacuum oven at 45°C for 24 hours, for adsorption were weighed into glass bottles with an analytical sensitive balance (Sartorius, Goettingen, Germany) of 0.0001g accuracy and placed in 2 little anaerobic jar containing saturated salt solutions prepared as recommended by Labuza (1984) and the COST 90 project (Wolf *et al.*, 1985). Two replications of the same experiment were carried out. The samples were weighed within interval of 48 hours and allowed to equilibrate until there was no

discernible weight change, as evidence by constant weight values (± 0.002 g). The total time required for removal, weighing and replacing samples anaerobic jar was 15-25 second.

The dry mass was determined gravimetrically. The EMC of the samples was determined after each adsorption experiment using an electronic humidimeter (METTLER TELED0, France), at 150°C for 5 to 12 mins until constant weight was reached. The mean values of two replicates were obtained for each experiment. To establish the moisture sorption isotherms, the EMCs were plotted against water activity at constant temperature.

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 0.0-0.90 (Mayor *et al.*, 2005). This model is one of the best for the characterization of the curves of food adsorption (Ferradji *et al.*, 2008a).

The equilibrium moisture content (EMC =X) data were processed using Microsoft Excel (2007) software and analysed using the polynomial regression method on transformed binomial equations for the GAB parameters. The GAB equation can be expressed as follows (Van de Berg, 1984 ; Labuza *et al.*, 1985):

$$X = \frac{X_0 \times C \times K \times a_w}{(1-K \times a_w)(1 - K \times a_w + C \times K \times a_w)} \tag{1}$$

Where: X = equilibrium moisture content, %, d.b.
 X_0 = GAB monolayer moisture content
 C = constant related to the monolayer heat of sorption
 K = factor related to the heat of sorption of the multilayer

The three parameters of GAB values of c, k and X_0 were derived from the second order polynomial form (equation 1) which was solved by multi-linear regression analysis to obtain, R and MRE (Jouppila and Roos, 1997; Abramovič and Klofutar, 2002):

$$\frac{a_w}{X} = \alpha a_w^2 + \beta a_w + \gamma \tag{2}$$

Where; $\alpha = K/X_0(1/C - 1)$
 $\beta = 1/ X_0(1 - 2/c)$
 $\gamma = 1/ X_0KC$

The values of parameters α , β , and γ were obtained

at the temperature through the following relations:

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

$$X_0 = 1 / (\beta + 2K\gamma)$$

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

To evaluate the goodness-of-fit for the models (GAB), the coefficient of correlation (R) and Mean Relative Error (MRE) were used. The coefficient of correlation was calculated using the following equation:

$$R = \frac{\sum_{i=1}^N (X_{exp} - \bar{X}_{exp})(X_{theo} - \bar{X}_{theo})}{\sqrt{\sum_{i=1}^N (X_{exp} - \bar{X}_{exp})^2 (X_{theo} - \bar{X}_{theo})^2}} \tag{3}$$

Where : X_{exp} = experimental EMC value
 \bar{X}_{exp} = experimental EMC mean value
 X_{theo} = predicted EMC value
 \bar{X}_{theo} = predicted EMC mean value
 N = number of experimental observations

The suitability of model fit was determined in terms of mean relative percentage deviation (MRE (%)) value (Boquet *et al.*, 1978; Lomauro *et al.*, 1985). A model is considered to be suitable if MRE < 10% (Lomarou *et al.*, 1985; Wang and Brennan, 1991). The mean relative error percentage deviation was calculated using the following equation:

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

X_{exp} = experimental EMC value
 X_{theo} = predicted EMC value
 N = number of experimental observations

Results and Discussion

Sorption characteristics of three composites flours

The adsorption isotherms obtained from three formulated flours containing plantain and cassava at 28±1°C are given in Figure 1. All adsorption isotherms demonstrated an increase in the equilibrium water content along with an increase in a_w (Figure 1). The adsorption isotherms of formulated flours of the three varieties plantain follow the characteristic shape of high-sugar foods according to the BET classification [namely, type II (S-shaped)] with the EMC increasing sharply at the high water activities (Moraga *et al.*, 2004). These findings are in agreement with Pott *et al.* (2005) for the adsorption characteristics of dried Kent variety mango slices at 20°C, Telis-

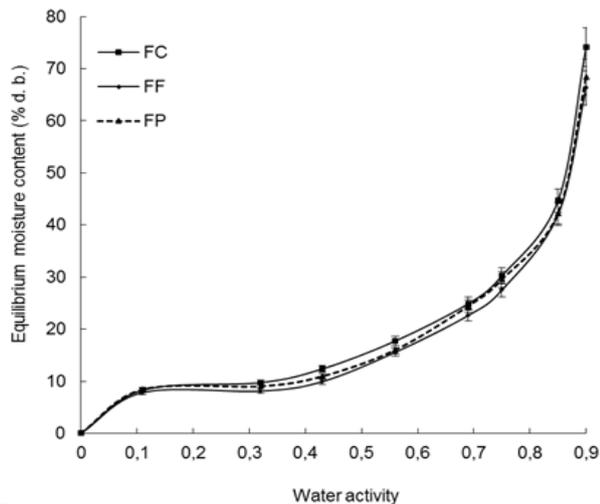


Figure 1. Comparison of experimental curves of the adsorption isotherms of the three composites flours of plantain (var. *Horn 1*, *FHIA 21* and *PITA 3*) and cassava (var. *Bonoua 2*)

Romero *et al.* (2005) for the adsorption isotherms of Haden variety mango pulp at different temperatures (30-70°C), and Falade and Aworh (2004) for the adsorption isotherms of osmo-oven dried African mango. 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 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 $a_w \geq 0.32$ 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

Table 1. Estimated GAB model coefficients, R and MRE fitted to the adsorption isotherms of three composites flours of plantain (var. *Horn 1*, *FHIA 21* and *PITA 3*) and cassava (var. *Bonoua 2*)

Model	FC	FF	FP	
GAB	X_0	7,375	6,086	6,443
	C	90,335	402,463	1490,212
	K	1,000	1,020	1,011
	E (%)	0,227	0,208	0,662
	R	0,997	0,994	0,995

rapid increase for $a_w \geq 0.75$ 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 three formulated flours containing plantain (FC, FF and FP) were classified in hygroscopicity order descending following: *Horn 1* flour (FC), *PITA 3* flour (FP) and *FHIA 21* flour (FF) (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 moisture sorption isotherm characteristics of plantain (*Musa*, AAB) and the sorption isotherm 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; Brou *et al.*, 2014). These results also corroborate those of Goula *et al.* (2008) who have worked on the sorption isotherm of tomato powder.

The characteristic shape of moisture isotherms depends upon the variety and total amount of hygroscopic materials present in the particular heterogeneous mixture of hydrophilic substances (Falade *et al.*, 2003). At low water activities, water can be adsorbed only onto the surface-OH sites of crystalline sugars. Wiesser (1985) reported that at low water activities there was a local dissolution of sugar, a swelling of the biopolymers and the appearance of new active sites. At high water activities, dissolution of sugar occurs and crystalline sugar is converted into amorphous sugar. The amount of water to be absorbed

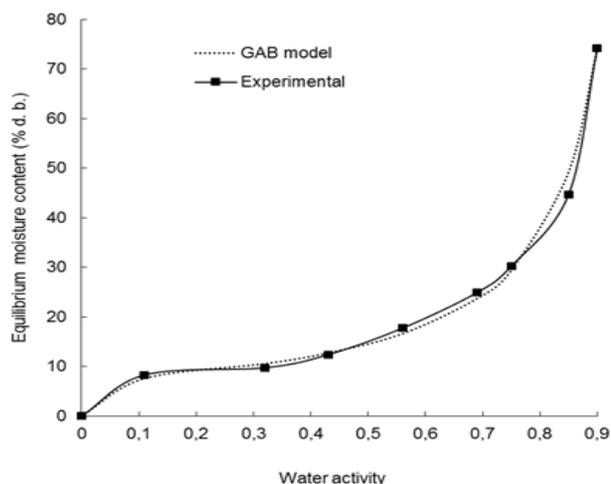


Figure 2. Comparisons of experimental and predicted adsorption isotherms of the composite flour of plantain of the variety *Horn 1* and cassava variety *Bonoua 2* using GAB model

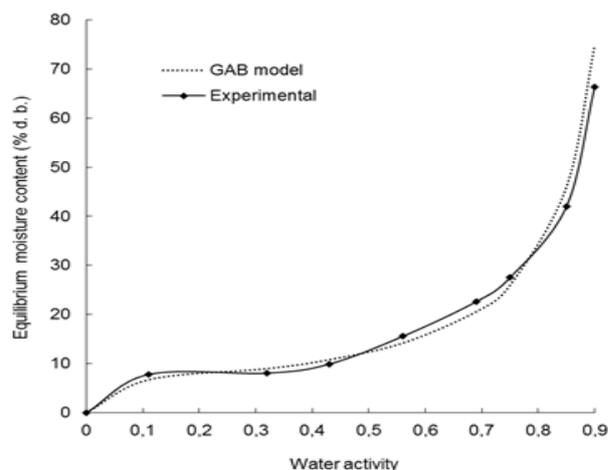


Figure 3. Comparisons of experimental and predicted adsorption isotherms of the composite flour of plantain of the variety *FHIA 21* and cassava variety *Bonoua 2* using GAB model

increases greatly after this transition because of the increase in the number of adsorption sites arising upon the breakage of the sugars' crystalline structure (Ayranci *et al.*, 1990).

Fitting of the GAB model to the adsorption isotherms data of three formulated flours of Horn 1 (FC), FHIA 21 (FF) and PITA 3 (FP)

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 correlation (R) and mean relative error (MRE).

Using these coefficients, the adsorption isotherms of formulated flours FC, FF and FP 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) have a sigmoid shape (type II) for all three formulated flours containing plantain. The mathematical models tested to estimate equilibrium moisture content (EMC) of *Horn 1* flour; *FHIA 21* flour and *PITA 3* flour presented values of determination correlation (R) above 85% (Table 1). The GAB model is applicable to all three flours. This agrees with the work of Akanbi *et al.* (2006) who states that the GAB's model is applicable if $R \geq 0.85$. 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. Low MRE (%) (0.227, 0.208 and 0.662) in ascending order for FP, FC and FF respectively shows good fitting

experimental curves (Arévalo - Pinedo *et al.*, 2004; Talla, 2012). The GAB model was applicable to all three flours (FC, FF and FP). The GAB model fitted well over the whole range of water activity, giving a low MRE (%) which varied between 0.208 and 0.662. This result is <10% and so it is an indication of good fit according to both Wang and Brennan (1991) and Lomauro *et al.* (1985). This assertion is justified by the works of Ferradji *et al.* (2008b) adsorption isotherm of dates and dried potatoes. It is also consistent with the work of Farahnaky *et al.* (2009) modeling of adsorption figs and potatoes. These results are similar to the work of Ferradji *et al.* (2008a) on the adsorption isotherm of dates and those of Oluwamukomi (2009) and Brou *et al.* (2014) on the modeling of sorption isotherm of garis and plantain, okra and pepper flour respectively of GAB model. But more applicable to the plantain composite flour (MRE of *PITA 3* composite flour were higher than those two composite flours (*Horn 1* and *FHIA 21*)).

The three formulated flours had not the same monolayer moisture content (X_0). *Horn 1* composite flour ($X_0 = 7.375 \text{ g H}_2\text{O} \cdot 100 \text{ g}^{-1}$) was higher than *FHIA 21* composite flour ($X_0 = 6.086 \text{ g H}_2\text{O} \cdot 100 \text{ g}^{-1}$) and *PITA 3* composite flour ($X_0 = 6.443 \text{ g H}_2\text{O} \cdot 100 \text{ g}^{-1}$) (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 adsorbent water surface (Ferradji *et al.*, 2008b). It represents the maximum water content below which is not available for chemical and biochemical reactions.

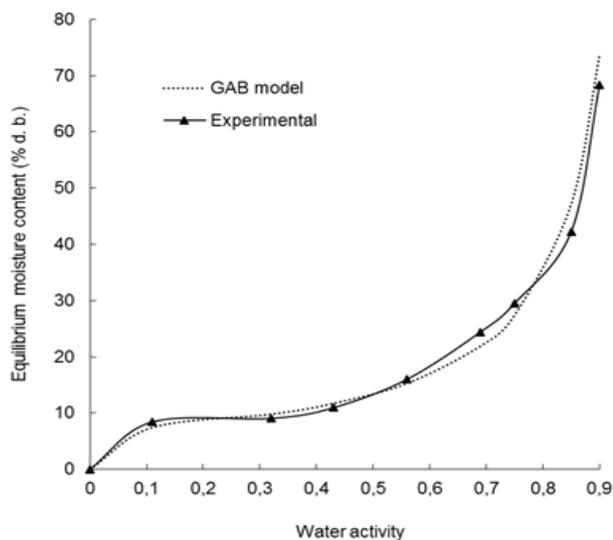


Figure 4. Comparisons of experimental and predicted adsorption isotherms of the composite flour of the variety *PITA 3* and cassava variety *Bonoua 2* using GAB model

This parameter is important for the control of product stability during storage (Ferradji and Malek, 2005). The monolayer moisture content X_0 is recognized as the moisture content according the longest time period with minimum quality loss at given temperature.

The constants C and K, which relate to the interaction energies between the the highest values of C (90.335, 402.463 and 1490.212 for FC, FF and FP respectively), but the e water and food, also vary according to the method of regression used. The polynomial regression gav values of K were smaller (1.000, 1.020, 1.011 for FC, FF and FP respectively). The constants C shows also the characteristics of the isotherm. If $C < 10$, the isotherm is of type III. But, if $C > 10$, the isotherm is type II according Medeiros *et al.* (2006).

Conclusion

The moisture adsorption isotherms of the three plantain formulated flours (FC, FF and FP) at temperature $28 \pm 1^\circ\text{C}$ under 0.11 to 0.90 water activity (a_w) range were determined with standard gravimetric methods using various saturated salt solutions. The adsorption isotherms provide valuable informations about the equilibrium moisture content of FC, FF and FP 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* and *PITA 3* flour. The adsorption isotherms of *Horn 1*, *FHIA 21* and *PITA 3* flour had been determined by experiment and then described by GAB. The

experimental results show that the adsorption isotherms of these three flours took a form of the sigmoid type (II) and that GAB's model gave a better fit for the three adsorption isotherms of *Horn 1*, *FHIA 21* and *PITA 3* flour. GAB's model was also used to determine the monolayer moisture content of *Horn 1*, *FHIA 21* and *PITA 3* flour and the values of the constants C and K.

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