



## Determination of the adsorption isotherms of two flour formulations based on plantain (*Musa x paradisiaca*) and cassava (*Manihot esculenta*) intended for the preparation of *Foutou*

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### Abstract

This study aims to experimentally determine the adsorption isotherms of two flour formulations consisting of plantain and cassava, intended for the preparation of foutou, a traditional African dish. The bananas and cassava used come from the Agneby-Tiassa region and the Abidjan district. The collected data obtained after analyses were subjected to statistical processing which included nonlinear regression analyzes. The results revealed that the determined adsorption isotherms are type II characterized by a sigmoidal shape. In addition, the GAB model (for 25 and 30°C) and the Peleg model (for 40°C) showed good agreement with the experimental data. Thus, these models make it possible to predict the hygroscopic behavior of the product during storage at these different temperatures.

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## Introduction

Plantain (*Musa spp.*) is a widely consumed product in Central and West Africa. It makes an essential contribution to food security, job creation, income diversification in rural and urban areas, gross domestic product (GDP) and, in so doing, to the fight against poverty. In Ivory Coast, plantains play an important role not only in feeding populations but also in cropping systems (Thiémiélé *et al.*, 2017).

Ivory Coast, located in West Africa, is self-sufficient in yams, cassava and plantains. In Ivory Coast, annual plantain production was 1,600,000 tonnes in 2012 (FAO, 2012). It is the most consumed food crop with 53.4% in urban areas and 46.6% in rural areas (BNETD, 1998). These fruits are most often used for the preparation of certain traditional dishes such as *foutou*, *aloco*, *claclo*, *apiti*, etc. and other food products including among others protein flour. Many studies undertaken over the past twenty years have focused on the transformation of plantains into baking flour, pastry flour and drinks (juice, beer and wine). In addition, formulated feeds in the form of flour or pulp are also made from plantains to expand the field of use of this commodity (Yao *et al.*, 2019).

Despite the importance of this food in diets, producers face problems of post-harvest losses related to the moisture content of the product. To reduce losses, extend shelf life and add value to the product, conventional drying is adopted. It is an operation widely used both in the agri-food industry and on an artisanal basis by farmers. This process stabilizes the product by lowering the water activity.

Furthermore, to optimize the storage conditions of a product so as to ensure its physicochemical and microbiological stability, the determination of sorption isotherms is a necessity. In addition, the study of sorption isotherms is a privileged means of understanding the distribution of the intensity of water bonds, as well as its functional availability in poorly or moderately hydrated agrifood products. These isotherms are curves which give precise information on the hygroscopic balance of a product

because they allow knowing its domain of stability after drying by determining the final water content.

During the last two decades, a significant number of works have focused on the study of sorption isotherms of food products, the influence of temperature on isotherms and the study of mathematical models describing sorption isotherms (Koko *et al.*, 2018).

However, very little work has been devoted to determining the isotherms of sorption of plantain flour and cassava. And yet, this is an essential step in optimizing the storage and drying conditions of these food products.

In order to contribute to the reduction of post-harvest losses of plantain in Ivory Coast by the development of new products, we have proposed in this work to study the adsorption isotherms of flour formulations based on plantain (*musa x paradisiaca*) of the *Corne1* and *FHIA 21* varieties and cassava roots of the *Bonoua* variety.

## Material and methods

### Plant material

The plant material of the present study consists of flour formulations based on two varieties of plantain (*corne1* and *FHIA 21*) composed of green (stage 1 or 2) and ripe (stage 4 or 5) fingers and cassava roots of the *bonoua* variety (Fig.1).



**Fig. 1.** *foutou* flour made from plantain and cassava. Scale: 1 / 3cm

### Methods

#### Sampling

Plantains and cassava were collected in the localities of Agboville, Tiassalé and Abidjan (Gonzagueville).

Banana bunches with known maturity periods were harvested randomly from a one hectare (1 Ha) plot assigned to each variety. The number of bananas of the *Corne1* variety was 10 bunches and that of the *FHIA 21* variety 7 bunches. That of cassava was 25 tubers. These bananas and cassava roots have been transformed into flour.

For each sample of *foutou* flour, the required proportions of plantain and cassava cassettes are weighed. The various weighed chips are then mixed and then crushed with a hammer mill. The mixture of crushed chips is ground with a miller (forplex, France) containing a 50 µm sieve to obtain the flour.

#### *Determination and modeling of absorption isothermal curves of different composite flours for conservation*

The FC2 (60% ripe banana + 40% cassava) and FH2 (60% ripe banana + 40% cassava) flour formulations respectively for the *Corne1* and *FHIA 21* varieties, the most appreciated by the panelists for each cultivar resulting from the sensory analyzes been used for the determination of the experimental adsorption isotherm curves. These absorption curves were modeled by the GAB equation (Guggenheim, Anderson and de Boer). All the measurements were repeated twice and the experiment was performed once.

#### *Preparation of saturated saline solutions*

Saturated saline solutions are prepared by adding different salts to 100 mL of distilled water with stirring using a magnetic stirrer (Selecta, Spain) with a bar magnet until supersaturation. Each solution is placed inside a tightly closed jar for 48 hours to fix the expected relative humidity before placing the samples (Garcia-Pérez *et al.*, 2008). The different saturated saline solutions set the respective relative humidities from 0.11 to 0.90. So, LiCl ( $a_w = 0.11$ ), MgCl<sub>2</sub> ( $a_w = 0.32$ ), K<sub>2</sub>CO<sub>3</sub> ( $a_w = 0.43$ ), Ca(NO<sub>3</sub>)<sub>2</sub> ( $a_w = 0.56$ ), SrCl ( $a_w = 0.69$ ), NaCl ( $a_w = 0.75$ ), KCl ( $a_w = 0.85$ ), BaCl<sub>2</sub> ( $a_w = 0.90$ ) (Greenspan, 1977). The verification of these relative humidities (RH) is made with the thermohygrometer which indicates the relative humidity and the temperature inside the jar. For the water activity of 0 desiccants (silica gels) are

used. The jars are exposed to the ambient temperature of  $28.2 \pm 1.3^\circ\text{C}$ .

#### *Determination of experimental absorption isothermal curves*

The method used for determining the adsorption curves is the static gravimetric method or the microclimate method (Lang *et al.*, 1981). This method was recommended by the European COST 90 project group using desiccators or jars (Wolf *et al.*, 1985). It consists of determining the kinetics of water uptake and determining the maximum adsorption capacity of each sample as a function of the different equilibrium relative humidities (RH). Therefore, the torque (HR, X) constitutes a point of the sorption isotherm. The graphic representation of the sorption isotherm curve is made up on the abscissa of the different  $a_w$  or RH equilibrium of saturated saline solutions and on the ordinate the water content (X) in grams (g) of water per 100 g of dry matter.

#### *Modeling of absorption isotherms of different samples*

The most widely used model of GAB (Guggenheim, Anderson and de Boer) with a large area of validity is used in this study. This model is one of the best theoretical models for the characterization of food sorption curves. It is used and accepted in food technology by most researchers (Ferradji *et al.*, 2008 a & b). This model is used in this study because it has a wide range of validity where the relative humidity is between 10 to 95% (Tonon *et al.*, 2009). The GAB equation looks like this (Labuza *et al.*, 1985 and Oluwamukomi, 2009) :

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

X = equilibrium water content

X<sub>o</sub> = water content of the monolayer

$a_w$  = water activity

C and K are the constants of the GAB model.

#### *Smoothing assessment criteria*

The applicability of this model was assessed by the criteria of the correlation coefficient (R), the mean square error (MSE) and the relative mean error of deviation (RMED).

The correlation coefficient (R) and the mean squared error (MSE) are the two criteria which make it possible to assess the smoothing of the experimental water contents by the GAB model. The correlation coefficient (R) is one of the first criteria to assess the smoothness of the model of the experimental and predicted curves. It is calculated to give an indication of the quality of the smoothing (fit) of the model. The closer the correlation coefficient (R) is to unity (1), the better the smoothing quality of the model (Vega-Galvez *et al.*, 2008).

In addition to R, the mean squared error (MSE) which is an absolute value, is used because it gives a clear idea of the mean divergence between the predicted and measured water contents. It is calculated as follows (McMinn *et al.*, 2007) :

$$EQM = \sum_{i=1}^n (X_{exp} - X_{théo})^2 * \frac{1}{N} \quad (2)$$

With :  $X_{exp}$  = experimental water content

$X_{théo}$  = theoretical water content

N = number of experimental water contents

In general, the higher values of the correlation coefficients (R) associated with the lower values of the mean squared errors (MSEs) show that the model is able to smooth the experimental sorption data (Vega-Galvez *et al.*, 2008).

The relative mean error of deviation RMED (%) is the criterion which allows to assess the applicability of this model. It is an absolute value that is used to assess the average divergence between the predicted and measured water contents. It is calculated by the following relationship (Kouhila *et al.*, 2004 ; McMinn *et al.*, 2007 ; Tonon *et al.*, 2009):

$$E(\%) = \sum_{i=1}^n \frac{(X_{exp} - X_{théo})}{X_{exp}} \times \frac{100}{N} \quad (3)$$

This criterion has been recommended by several researchers such as Maroulis *et al.* (1988) to assess the applicability of the GAB model. This relative mean deviation E (%) is widely adopted in the literature, with a value of less than 10% as a good indicator for adjusting experimental water contents (Vega-Galvez *et al.*, 2008).

In general, the lower the values of E (%), i.e. <10%, the better the model is able to fit the experimental data (Maroulis *et al.* 1988, Vega-Galvez *et al.*, 2008).

#### Statistical analysis

All measurements were performed in duplicate for the adsorption isotherm. Statistical analysis of the results was carried out using Statistica 7.1 software. One-way analysis of variance (ANOVA) and Duncan's test were performed to compare the variables analyzed on the different composite flours of banana *foutou* with incorporation of cassava. The differences were considered significant for values of  $P \leq 0.05$ .

For the modeling of the adsorption isotherm curves, statistical analysis was performed by means of MSE (Mean Squared Error), RMED (%) (Relative Mean Error of Deviation) and R (Correlation Coefficient) which allow to appreciate the correlation between the experimental data and those calculated.

## Results and discussion

### Results

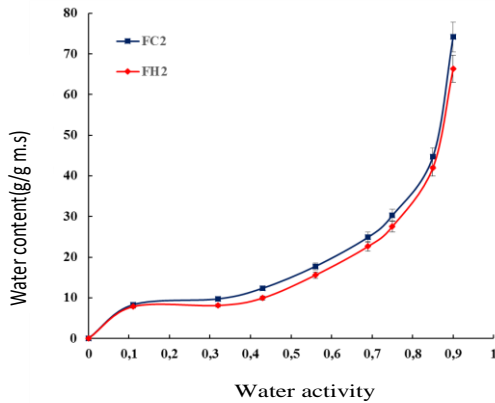
#### Experimental and theoretical adsorption isotherm curves of the two formulated *foutou* flours

#### Experimental adsorption isotherm curves of two formulated *foutou* flours

Fig. 2 illustrates the comparison of the experimental adsorption isotherm curves of flour formulations composed of plantain FC2 (60% ripe banana + 40% cassava) and FH2 (60% ripe banana + 40% cassava) of the cultivars *Cornel1*, *FHIA 21* respectively. The two curves have a sigmoidal shape and present three characteristic zones. They are typical to the type II isotherm as with most agri-food products. Fig.2 shows an increase in the water content of different flours as a function of the water activity at room temperature. The two absorption isothermal curves show three areas which are as follows (Fig. 2).

Zone I ( $a_w = 0$  to 0.32), has a slight increase in water content of 8.74% and 9.05% for a variation of  $a_w$  equal to 0.32 for the flour of FC2 and FH2 respectively. Then zone II ( $a_w = 0.32$  to 0.75) which shows an average increase in the water content of 20.51% and 22.42% respectively for FC2 and FH2 for

a variation of  $a_w$  equal to 0.43. Finally, zone III ( $a_w = 0.75$  to  $0.90$ ), exhibits an exponential increase in the water content of 43.91% and 33.77% for a variation of  $a_w$  equal to 0.15 (Fig. 2).



**Fig. 2.** Comparison of the experimental curves of the adsorption isotherm of formulated flours from FC2 and FH2 *foutou*.

However, at relative humidities less than or equal to 56%, FC2 flour weakly adsorbs water ( $17.73 \pm 0.01$  g / 100g of dry matter), as does FH2 ( $15.57 \pm 0.2$ g / 100g of dry matter respectively). Likewise, at RH of between 56% and 75%, the two composite flours produced adsorb moderately water, i.e.  $30.25 \pm 0.05$  and  $27.53 \pm 0.00$ g of dry matter for FC2 and FH2 respectively (Fig. 2).

On the other hand, at RH greater than 75%, they adsorb a large quantity of water, i.e.  $74.16 \pm 0.04$  g / 100 g of dry matter for FC2 and  $66.37 \pm 0.12$  g/100 g of dry matter for FH2 (Fig. 2).

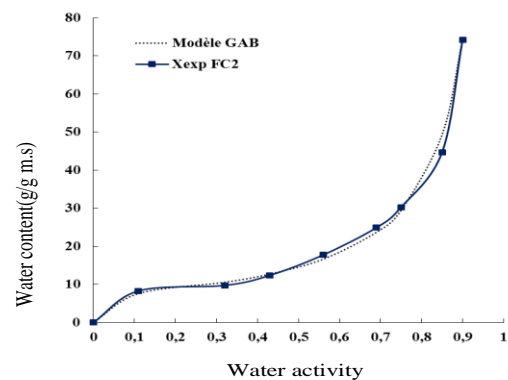
The *foutou* flour formulations of the two plantain varieties all exhibit isothermal adsorption curves with high hygroscopicity. However, the two formulations are classified in the following descending hygroscopic order : FC2 followed by FH2.

*Modeling of experimental absorption isothermal curves for flours composed of FC2 and FH2 foutou*

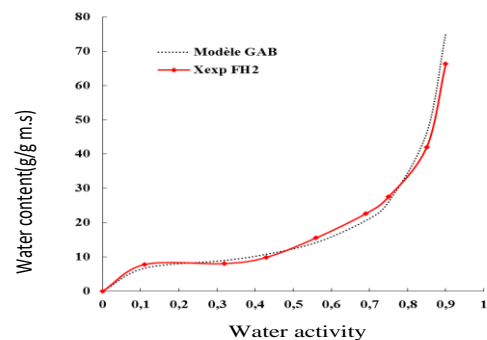
The adjustment of the experimental water contents of the two formulated flours carried out by the GAB model (Guggenheim, Anderson and de Boer) makes it possible to make comparisons between the

experimental curves and those predicted. The different calculated water contents made it possible to construct the predicted absorption isotherm curves of the two products studied. These curves have a sigmoidal shape and are type II. These curves are illustrated in Fig. 3 and 4.

Fig. 3 illustrates the experimental and theoretical adsorption isotherm curves of the FC2 formulation. It can be seen that over the entire range of water activities, the theoretical curve and the experimental curve are superimposable, that is to say they are the same. On the other hand, Fig. 4 shows the curve of the experimental and theoretical adsorption isotherm of the FH2 flour formulation. We observe between the water activity from 0.11 to 0.56  $a_w$ , the theoretical and experimental curves are almost confused. At  $a_w$  between 0.56 to 0.75  $a_w$ , the theoretical curve is below the experimental curve and from 0.75  $a_w$  the theoretical curve is above the experimental curve.



**Fig. 3.** Comparison of the experimental and theoretical adsorption isotherm curves of the FC2 formulation.



**Fig. 4.** Comparison of experimental and theoretical adsorption isotherm curves of the FH2 formulation.

The criteria for assessing the applicability of the calculated GAB model are shown in Table I. These criteria include the correlation coefficient (R), the mean square error (MSE) and the relative mean error of deviation (RMED) The analysis of these assessment criteria presented in Table I shows that the correlation coefficient of flour FC2 is  $R = 0.997$  and that of FH2 is  $R = 0.994$ . Both formulations exhibit higher correlation coefficients and are greater than 85%. The FC2 formulation has a higher mean square error (MSE = 2.161 compared to the MSE of FH2 which is 1.399).

The mean deviation error of the FC2 flour is RMED (%) = 0.227 and that of the FH2 formulation (RMED (%)) = 0.208.

The parameters of GAB are shown in Table I. The results show that the two products do not have the same values of the water content of the  $X_0$  monolayer. Thus, the water content of the monolayer of the FC2 formulation ( $X_0 = 7.375$  g of water/100 g of dry matter) is higher than that of the FH2 flour ( $X_0 = 6.086$  g of water/100 g of dry matter). The values of the parameters C of the GAB equation in Table I are used to characterize the sorption isotherms. When  $C \leq 10$ , the sorption isotherm is type III while if  $C \geq 10$ , the sorption isotherm is type II (Medeiros *et al.*, 2006). Thus, FC2 ( $C = 90.335$ ) and FH2 ( $C = 402.463$ ) flours have type II adsorption isotherms.

The two formulated flours of foutou have values of K substantially greater than or equal to 1. Thus, the values of the two formulations of flours are 1.000 and 1.020 respectively for FC2 and FH2 (Table I).

**Table I.** Parameters and estimated constants of the GAB model for FC2 and FH2 flours.

Model		FC2	FH2
GAB	$X_0$	7.375	6.086
	C	90.335	402.463
	K	1.000	1.020
	RMED (%)	0.227	0.208
	MSE	2.161	1.399
	R	0.997	0.994

## Discussion

The FC2 and FH2 flours adsorption curves during this study have a sigmoidal shape according to the classification by Brunauer *et al.* (1938). The isothermal adsorption curves for FC2 and FH2 flours are type II, characteristic of agrifood products. These results are similar to those of Johnson & Brennan (2000) and Medeiros *et al.* (2006) who worked on plantain, cocoa and chocolate powders respectively. These same observations have been reported by Brou *et al.* (2014) on plantain flour of the *Cornei* variety, pepper (*Capsicum annuum var. PM17/04A*) and okra (*Abelmoschus esculentus var. TOMI*).

The C parameter of the GAB model of the two compound flours is greater than 10 ( $C \geq 10$ ), meaning that the adsorption isotherm is type II (Medeiros *et al.*, 2006). This typical sigmoidal shape is characteristic of agro-food products containing sugar which absorbs a small amount of water at low water activities and a large amount of water at high water activities (Roca *et al.*, 2008; Yué & Tano, 2008). These results are consistent with those of Goula *et al.* (2008) who worked on tomato powder. The two flours composed of foutou studied are all hygroscopic. This can be explained by the fact that the two flours all have a high level of carbohydrate and which would promote the great adsorption of water vapor. This result is similar to that of Ferradji *et al.* (2008a) which stated that high carbohydrate products are more hygroscopic than those with low carbohydrate content.

The applicability of the GAB model is judged by criteria. Thus the correlation coefficients (R) of the two formulated flours are high while their relative mean errors of deviation (RMED) and their mean square errors (MSE) are low. This observation is similar to that reported by Arévalo-Pinedo *et al.* (2004) and Oluwamukomi, (2009). This result is in agreement with those of Farahnaky *et al.* (2009) on the modeling of adsorption isotherms of figs and Al-Muhtaseb *et al.* (2004) on the modeling of potatoes by the GAB equation. The correlation coefficient of FC2 flour is 0.997 while that of FH2 flour is 0.994. The GAB model is applicable to the two flours FC2 and FH2 because it remains close to 1.



This observation is in agreement with the work of Akanbi *et al.* (2006) who said that the GAB model is applicable if the correlation coefficient  $R \geq 85\%$ .

The fit of the GAB model is acceptable when the relative mean error (RMED (%)) is less than 10%. The various relative mean errors of the two *foutou* flours are less than 10%. These results are similar to those of Johnson & Brennan (2000) and Farahnaky *et al.* (2009). These observations have also been reported by McMinn *et al.* (2007) and Oluwamukomi (2009) respectively on the subject on the modeling of the adsorption of oatmeal and oatmeal cookies in the United Kingdom and the modeling of gari flour in Nigeria by the GAB equation<sup>3</sup>. The GAB model can be used to predict the value of the equilibrium water content, the water content of the monolayer, and the binding enthalpies of the monolayer and multilayer. It has been demonstrated by the work of Ferradji *et al.* (2008a) on the adsorption isotherm of dates as well as the work of Oluwamukomi (2009) on the modeling of gari flour. The GAB model gives physical meaning to the values of the parameters. Thus, the water content ( $X_0$ ) of the monolayer represents the quantity of adsorbed water for which all the available hydrophilic sites are linked to the first monolayer of water on the surface of the absorbent (Ferradji *et al.*, 2008b). It represents the maximum amount of water below which water is not available for chemical and biochemical reactions. This parameter is important for controlling the stability of products during storage (Ferradji & Malek, 2005). These values of the water content of the monolayer of the products indicate that the chemical alteration reactions are weak and the stability of the two products is satisfactory during storage (Karel, 1975).

### Conclusion

Determining and modeling adsorption isotherms is an essential step in any food drying and storage process. The absorption curves of the plantain *foutou* flour (FC2 and FH2) of the two varieties were determined experimentally at  $28 \pm 1$  ° C with the static gravimetric method. These curves of the two flours have a sigmoidal shape and are type II, proving a high hygroscopicity like most food products.

The theoretical curves obtained coincide well with the curves of the experimental results. The GAB model makes it possible to correctly predict the hygroscopic behavior of these. The isothermal absorption curves of the two flours are correctly smoothed by the GAB model and they follow the general shape of the absorption curves of food products. It can therefore be said that the two flour formulations have good stability during storage.

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