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Full Length Research Paper

Adsorption isotherm modeling of soy-melonenriched and un-enriched 'gari' using GAB equation

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Fresh samples of soy-melon enriched and un-enriched (control) "gari" (a fermented, dewatered and toasted granule from cassava) widely consumed all over West Africa and in Brazil, were prepared and used to determine their sorption isotherms. The sorption isotherms were determined within a range of water activities of (0.20 - 0.92) at three different temperatures of 20, 30 and 40°C using the static gravimetric method. Results showed that the water sorption was temperature dependent as typical of food systems. The higher the temperature, the lower the equilibrium moisture content (EMC) at constant water activity (a w). EMC for soy-melon "gari" ranged between 0.022 and 0.320 kgkg⁻¹(db) within the temperature range of 20 and 40°C; while those of un-enriched (control) "gari" ranged between 0.054 and 0.335 kgkg⁻¹(db). The GAB monolayer moisture values (Mo) were 0.044, 0.032 and 0.023 kgkg⁻¹(db) at 20, 30, and 40°C respectively for soy-melon "gari"; while for the control "gari" samples, they were 0.080, 0.059, and 0.050 kgkg⁻¹ (db). The 'Mean relative percent modulus' (E%) values were 9.45, 7.37, and 9.61% at 20, 30 and 40°C respectively for soy-melon "gari" samples; while for the control samples they were 3.76, 2.48, and 2.04%. The RSS values ranged from 0.0002 to 0.0008 for soy-melon gari while they ranged from 0.0001 to 0.0004 for the un-enriched gari at temperature between 20 and 40°C. All the E% values were below 10%. These showed that there was perfect temperature between the experimental moisture sorption and the predicted GAB sorption. However, the values for un-enriched (control) "gari" samples were consistently lower than those of the enriched gari showing that GAB model could be used to predict the moisture equilibrium of control "gari" samples better than those of soy-melon "gari" at the range of water activities used in this study.

Key words: Soy-melon "gari", supplementation, EMC, sorption isotherm.

INTRODUCTION

"Gari" is a toasted granule from grated, fermented and dewatered cassava meal which is widely consumed all

Nomenclature: , , , , GAB parameters in Equation (8); aw, water activity (decimal); c, k, GAB constants; db, dry basis; df, degree of freedom of regression model; E%, mean relative percentage deviation (%); ERH%, equilibrium relative humidity (decimal); Mcal, calculated equilibrium moisture content (kg kg dry solid); Mexp, experimental equilibrium moisture content (kg kg dry solid); Mo, monolayer moisture content (kg kg dry solid); No, the number of experimental points; r², coefficient of fit (decimal); RSS, Residual sum square (decimal) and SEE, standard error of the estimate (decimal).

over West Africa and in Brazil. It is processed by pressing the juice out of the peeled and grated cassava roots which has been allowed a natural lactic acid fermentation of 2 - 3 days to take place (Adeyemi and Balogh, 1985). The fermented mash is then toasted in an open aluminum pan over open fire until the starch gelatinizes to 65% of its native form and the moisture content falls to less than 12% dry basis (Chuzel and Zakhia, 1991; Zakhia et al., 1985). The problem of poor keeping quality of many dehydrated foods in the tropics is related to their moisture uptake during merchandising because of poor packaging materials and the moisture levels at which they were prepared (Igbeka et al., 1975; Onayemi and Oluwamukomi, 1987; Mazza, 1986). The shelf life of packaged food ma-

terials has been shown to be influenced by the temperature, relative humidity and moisture content and thus the water activity (aw) of the material (Iglesias and Chirife, 1976). The relationship between water activity and moisture content of a food-stuff is important in predicting quality stability during storage and the selection of appropriate packaging materials for retail purposes (Igbeka, 1982). The moisture levels at which some dehydrated foods have good storage stability have been found to agree closely with the moisture levels calculated from the sorption isotherm at different ambient temperature and RH% (Igbeka and Nwachukwu, 1983; Mazza, 1986). Some studies have been carried out on the sorption isotherm of Cassava products such as "gari" (Chuzel and Zakhia, 1991), "lafun" (Onayemi and Oluwamukomi, 1987), Instant cassava flour (Oluwamukomi, 1999), "fufu" (Sanni et al., 1997), "lafun" and Tapioca (Kuye and Sanni, 2002).

However there is dearth of information on sorption isotherm of many African food products and specifically protein enriched cassava products. A knowledge and understanding of such information on the optimum moisture levels, which should be arrived at during manufacture and for selection of good packaging material is of great importance in order to avoid their quality deterioration during storage. The water sorption isotherm of foods is therefore of great importance. Many empirical relationships describing the sorption characteristics of food such as Hasley (1948), Henderson (1952), Chung Pfost (1967), Oswin (1946), B.E.T (1938), Langmuir (1940), Smith (1947), Caurie (1970): and the three-parameter GAB model (Wolf et al., 1985; van de Berg, 1984). These models 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 aw of 0.9 (Van de Berg and Bruin, 1981, Iglesias and Chirife, 1976; 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).

The objective of this study therefore is to subject soymelon protein enriched and un-enriched (control) "gari" to sorption isotherm study in order to determine if GAB sorption model could be used to describe the sorption behavior of the two samples.

MATERIALS AND METHODS

Samples of soy-melon "gari" were produced according to the methods of Oluwamukomi (2006) and Banjoh Ikenebomeh (1996). The Cassava tubers were peeled manually with a sharp knife, washed and grated in a locally fabricated mechanical grater driven by a powered system (7 hp) (Agunbiade, 2001). The cassava mash Obtained was allowed to ferment for 72 h after which it was pressed

in a mechanical press (Addis Engineering Nigeria Ltd., Nigeria) to dewater the mash (Agunbiade, 2001; Adeyemi and Balogh, 1984, 1985). The dewatered wet meal was pulverized and enriched with full fat soy-melon supplement at 15% enrichment level and taking into consideration the water content of the mash of 65% (Akingbala et al., 1993). It was then toasted in a wide iron pan (garifier) being heated over wood fire (Adeyemi and Balogh, 1984, 1985). The toasted and cooled soy-melon "gari" samples were then packaged in HDPE film and kept under refrigerated storage until ready for further analysis.

A static gravimetric method was used for the experiment (Greenspan, 1977; Wolf et al., 1985). Duplicate samples, 3 ± 0.001 g each, of soy-melon and control "gari" were placed in Petri dishes inside 6 desicators containing saturated salt solutions (KC2H3O2, MCl2, K2CO3, MNO3, NaCl, KCl) providing constant relative humidity environments ranging from 23 - 92% (Rockland, 1960; Labuza et al., 1985) . The sample had been pre- dried in a desiccator with P2 O5 at room temperature for 15 days. At high relative humidity (aw > 0.7), toluene (1.5 ml) was placed in the desiccators to prevent microbial growth (Labuza, 1984; Kumar and Mishra, 2004). The desiccators were kept in temperature controlled cabinets at constant tempera-tures of 20, 30 and 40°C. The samples were weighed at intervals of 3 days using a digital balance (Model Mettler PE1600, Mettler In-struments Corporation, Greifensee, Zurich, Switzerland) with an ac-curacy of 0.1 mg until constant weighs were obtained after three consecutives recordings, when the samples were assumed to be at equilibrium (± 0.001 g). The bone dry mass was determined by the oven-drying method for 8 - 10 h at 105 -110°C (AOAC, 1990). The time to reach equilibrium ranged from 15 to 30 day depending on the water activity in each desiccator with those at higher water acti-vities reaching equilibrium faster than those at lower water activi-ties. The equilibrium moisture contents were calculated from which the moisture sorption isotherms were determined. The data for the water adsorption were fitted to the GAB equation to describe their moisture adsorption using the GAB equation (Equation 1) (Van de Berg and Bruin, 1981).

Theory of GAB equation

= 1/ Mokc

The GAB equation can be expressed as follows (Van de Berg, 1984; Labuza et al., 1985):

$$M_e = \frac{ckM_o a_w}{(1 - ka_w)(1 - ka_w + cka_w)}$$
 (1)

The three parameters of GAB values of c, k and M_{\odot} were derived from the second order polynomial form (equation 2) which was solved by multi-linear regression analysis to obtain , , , r^2 and RSS (Jouppila and Roos, 1997; Abramovi and Klofutar, 2002; Xion, 2002):

$$\frac{a}{w} = \alpha a_w^2 + \beta a_w + \gamma$$

$$M_e$$
Where; = k/M₀(1/c - 1)
= 1/ M₀(1 - 2/c)
(2)

The values of parameters , , and were obtained for each temperature through the following relations:

$$\frac{M}{\sigma} = \frac{1}{\sqrt{\left(\beta^2 - 4\alpha\gamma\right)}} \tag{3}$$

Table 1. Equilibrium moisture contents (Me_{cal}) (kgkg⁻¹ db) for soy-melon enriched and un-enriched (control) "gari" stored at 20. 30 and 40°C.

	Soy-n	nelon enriched	"gari"	Un-enriched (Control) "gari"				
	20ëC	30ëC	40ëC	20ëC	30ëC	40ëC		
0.23	0.040±0.002	0.023±0.001	0.022±0.003	0.084±0.003	0.066±0.001	0.054±0.002		
0.35	0.061±0.001	0.035±0.002	0.032±0.004	0.107±0.002	0.073±0.001	0.066±0.003		
0.45	0.081±0.002	0.045±0.003	0.040±0.002	0.130±0.004	0.096±0.003	0.086±0.002		
0.61	0.084±0.002	0.052±0.004	0.041±0.002	0.153±0.003	0.109±0.003	0.100±0.003		
0.75	0.120±0.001	0.105±0.006	0.080±0.004	0.190±0.002	0.148±0.004	0.141±0.003		
0.92	0.320±0.003	0.170±.005	0.150±0.004	0.335±0.005	0.231±0.004	0.216±0.004		

Table 2. Experimental (Meexp) and GAB equilibrium moisture contents(Mecal) (kgkg⁻¹ db) for soy-melon "gari" stored at 20, 30, and 40°C.

Temperature of storage	20°C		3	0°C	40°C		
Water activity (aw)	Me _(exp)	Me _(cal)	Me _(exp)	Me _(cal)	Me _(exp)	Me(cal)	
0.23	0.040±0.002	0.0431±0.003	0.023±0.001	0.0239±0.002	0.022±0.003	0.0239±0.001	
0.35	0.061±0.001	0.0554±0.002	0.035±0.002	0.0342±0.001	0.032±0.004	0.0301±0.002	
0.45	0.081±0.002	0.0770±0.001	0.045±0.003	0.0436±0.002	0.040±0.002	0.0362±0.003	
0.61	0.084±0.002	0.0930±0.002	0.052±0.004	0.0633±0.003	0.041±0.002	0.0501±0.002	
0.75	0.120±0.001	0.1036±0.003	0.105±0.006	0.0915±0.004	0.080±0.004	0.0723±0.005	
0.92	0.320±0.003	0.3457±0.002	0.170±.005	0.1700±0.002	0.150±0.004	0.1412±0.003	

$$c = \frac{2\sqrt{(\beta_2 - 4\alpha\gamma)}}{-\beta + \sqrt{(\beta - 4\alpha\gamma)}}$$
(4)

$$k = \frac{-\beta + \sqrt{(\beta_2 - 4\alpha\gamma)}}{2\alpha\gamma} \tag{5}$$

 M_{\odot} is the monolayer moisture content, c is a constant related to thermal effects and k is the constant related to the properties of multilayer water molecules with respect to the bulk liquid. These GAB constants M_{\odot} , c, and k values were fitted into the GAB equation (1) resulting in predicted GAB equilibrium moisture content $(M_{\rm e})_{\rm (cal)}$ values obtained over the range of water activities (0.23 - 0.92) used for this study. The GAB equilibrium moisture contents $(M_{\rm e(cal)})$ and the experimental equilibrium moisture contents $(M_{\rm e(exp)})$ were compared to determine its fitness to describe the sorption isotherms of the samples.

The quality of fitness of the GAB model were evaluated by calculating the "Mean relative percent deviation modulus" (%E), the standard error of estimate (SEE) (Lomauro et al., 1985; Chen and Morey, 1989) and Coefficient of determination (r²) (Akanbi et al., 2006) and the Residual sum of squares (RSS) (Sun, 1999) by using equations (6, 7 and 8) respectively. M - M

$$\frac{100}{M} = \frac{M - M}{M}$$
% $E = N$ exp exp (6)

$$SEE = \sqrt{\frac{\left(M_{\rm exp} - M_{cal}\right)^2}{df}}$$
 (7)

$$_{2}$$
 RSS $(M_{cal} - M_{ave})^{2}$

$$r = TSS = \left(M_{\text{exp}} - M_{\text{ave}}\right)^2 \tag{8}$$

A "Mean relative percent deviation modulus (%E) of less than 10% is an indication that the GAB model is well suited for describing adsorption isotherm of the sample (Aguerre et al., 1989; Lomauro et al., 1985; Ajibola, 1986b).

RESULTS AND DISCUSSION

The results of the determinations of the equilibrium moisture content at the three temperatures of 20, 30 and 40°C are as shown in Table 1.

Fitting of GAB sorption model to the experimental sorption data

The results of the fitting of the GAB equations to the experimental data in Table 1 are shown in Table 2 while the GAB model isotherm curves are shown in Figures 1 and 2. The coefficients, , , and of the regression equation of the GAB model obtained from the isotherm plots (Figures 1 and 2) are shown in Table 3; while the GAB constants M_0 , c, and k values obtained are also shown in the Table 3. The calculated equilibrium moisture content, $Me_{(cal)}$ compared and fitted to the experimental sorption data, $Me_{(exp)}$ to determine if GAB model can be used to describe the sorption behavior soy-melon "gari" over

Table 3. GAB constants (k, c and M₀), E%, SEE, r² and RSS values for soy-melon enriched and control (un-enriched) "gari" stored at 20, 30, and 40°C.

Sample	Temperature of storage	k	С	M₀ (kgkg ⁻¹ db)	SEE	E%	r ²	RSS
Soy-melon enriched "gari"	20 [°] C	0.909	13.09	0.044	0.012	9.45	0.968	0.0002
	30°C	0.900	5.76	0.032	0.009	7.37	0.942	0.0003
	40°C	0.916	14.57	0.023	0.006	9.61	0.907	0.0008
	20 [°] C	0.804	44.06	0.080	0.009	3.76	0.936	0.0001
Un-enriched (Control) "gari"	30°C	0.811	38.64	0.059	0.004	2.48	0.964	0.0002
	40°C	0.816	13.93	0.050	0.003	2.04	0.983	0.0004

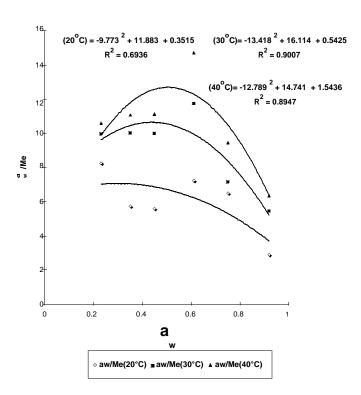


Figure 1. GAB Model Isotherm curve for Soy-Melon "gari" stored at 20, 30 and 40 °C.

the entire range of water activity are shown in Figures 3 and 4 for both soy-melon enriched and un-enriched (control) "gari".

GAB monolayer moisture content (M_o): The GAB monolayer values (M_o) for soy-melon enriched "gari" were 0.044, 0.032 and 0.023 kgkg⁻¹ (db) at 20, 30, and 40°C respectively while for the control "gari" samples, they were 0.080, 0.059, and 0.050 kgkg⁻¹ (db) at 20, 30 and 40°C respectively. The GAB monolayer moisture values (M_o) obtained were temperature dependent (Wang and Brennan, 1991; Lomauro et al., 1985; Schar and Ruëgg, 1985; McLaughlin and Magee, 1998) . As the temperature increased from 20 to 40°C, the GAB monolayer moisture content (M_o) decreased from 0.044 to 0.023 kgkg⁻¹ (db). For the control "gari", M_o decreased from 0.080 to 0.050

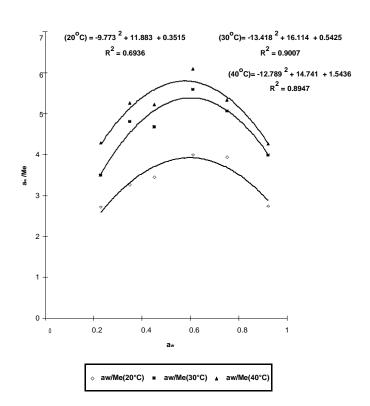


Figure 2. GAB Model Isotherm curve for Control gari at 20, 30 and 40°C.

kgkg⁻¹ (db) at the same temperatures. A decrease in GAB monolayer moisture content (Mo) with increase in temperature was an indication that the absorbed molecules gained kinetic energy making the attractive forces to be loosened and this allowed some water molecules to break away from their sorption sites thus decreasing the equilibrium moisture values (Arevalo-Pinedo et al., 2004; Labuza et al., 1985). The monolayer moisture contents for the soy- melon "gari" ranging from 0.023 - 0.044 kgkg⁻¹ (db) at the three temperatures were lower than those of the control "gari" ranging from 0.057 - 0.060 kgkg⁻¹ (db). These values were also lower than other similar un-en-riched cassava products such as "fufu" (0.043 - 0.049), tapioca (0.049 - 0.058), unenriched "gari" (0.057 - 0.060) (Kuye and Sanni, 2002; Sanni et al., 1997; Chuzel and Zakhia, 1991 respectively). The monolayer moisture con-

Table 4. Comparison of the GAB sorption parameters for un-enriched (control), soy- melon enriched "gari" with other past findings on similar cassava products ("lafun", "gari", "fufu", and tapioca) at similar temperatures of storage.

Estimated GAB Parameters	Un-enriched (Control) "gari"			Soy-melon "gari"			Un-enriched "gari"*		
	20°C	30°C	40°C	20°C	30°C	40°C	15°C	25°C	35°C
	-9.90	-13.41	-12.78	-38.45	-19.54	-5.98	-13.3	-15.1	-14.5
	11.802	16.114	14.741	39.14	16.457	2.056	16.0	17.0	15.7
	0.407	0.542	1.543	2.855	6.77	6.920	40.2	45.4	94.4
r²	0.936	0.964	0.983	0.96	0.940	0.910	0.989	0.996	0.988
SEE	0.009	0.004	0.003	0.012	0.009	0.006	ND	ND	ND
E%	3.759	2.4833	2.04	9.936	7.377	9.611	4.8	3.4	4.6
k	0.804	0.811	0.816	0.900	0.909	0.916	0.824	0.869	0.877
С	44.061	38.645	13.93	13.090	5.765	14.569	50.4	45.1	20.9
Mo(kgkg ⁻¹ db)	0.080	0.059	0.050	0.044	0.032	0.023	0.060	0.056	0.057
GAB constants	"Fufu"**			"Lafun"**			Tapioca**		
	25°C	32°C	45°C	25°C	32°C	45°C	25°C	32°C	45°C
k	0.6924	0.715	0.661	0.686	0.710	0.665	0.6201	0.528	0.5509
С	126	41.11	18.17	99.539	43.256	19.786	27.2	9.203	7.762
_M₀(kgkg ⁻¹ db)	0.049	0.045	0.043	0.049	0.045	0.043	0.058	0.064	0.049

^{*}Chuzel and Zakhia (1991).

^{**}Kuye and Sanni (1997, 2002).

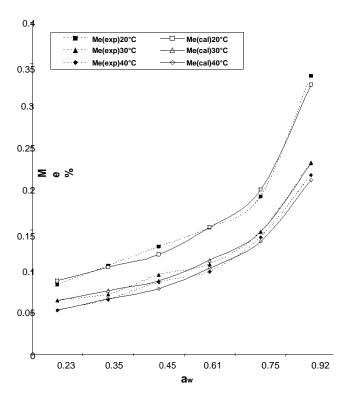


Figure 3. Experimental and GAB fitted data for Control "gari" at 20, 30 and 40° C.

contents, M_o were less than 0.1 kgkg⁻¹ (db) in all the products which was the maximum value reported for food

materials (Labuza et al, 1985; Van den Berg, 1985, Kaymak-Ertekin and Sultanoglu, 2001). These values agree with literature data for other starchy products (Chuzel and

Zakhia, 1991, for "gari", Kuye and Sanni, 2002, for "laun", Sanni et al., 1997) for "fufu" and tapioca) (Table 3).

GAB constants (k and c): The GAB parameters Mo, c and k, were shown to be temperature dependent (Table 3). Increase in the temperature resulted in either a decrease or increase in the M_{o} , c and k values (Diosady et al., 1996). For soy-melon enriched "gari" samples, with increase in temperature from 20 to 40°C, Mo decreased, c and k increased. For the un-enriched (control) "gari", as temperature increased, the GAB constant (k) increased from 0.804 to 0.816, while c decreased from 44.06 to 13.93. Increase in (k) was an indication that at higher temperature, the multilayer molecules became more entropic while a decrease of c was an indication of more enthalpy and a gain in kinetic energy resulting in the loss of more moisture at higher temperature (Diosady et al., 1996). The range of values of k less than unity (1.0) was in agreement with the GAB model's assumption that the multilayer has properties between those of the monolayer and bulk liquid. When k = 1, the multilayer has bulk liquid properties. This is consistent with findings made for similar products by Chuzel and Zakhia (1991) for un-enriched "gari"; Sanni et al. (1997) for "fufu" and tapioca Kuye and Sanni (2002) for "lafun" (Table 4); Wang and Brennan (1991), Lomauro et al. (1985) and Schar and Ruëgg (1985).

However, for the soy-melon "gari", there was an initial

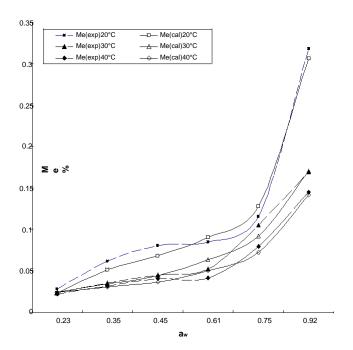


Figure 4. Experimental and GAB fitted data for Soy-melon "gari" stored at 20, 30, and 40°C.

decrease in k and c values from 0.909 and 13.090 to 0.900 and 5.765 respectively as the temperature was increased to 30°C but they later increased to 0.916 and 14.569 when the temperature was further increased to 40°C. This is was a slight deviation from the general trend which was supposed to be temperature dependent, and for k to increase while c decreases. This unusual behavior is similar to that of canola meal where Diosady et al; 1996) observed that there was a decrease in both k and c as the temperature was increased. The common behavior of both canola meal and soy-melon "gari" might have been due to their similar properties of being an oily product and their differences in composition, preparation and granulation from other non-oily starchy products (Diosady et al., 1996).

When the experimental and fitted isotherms were compared, best fits were obtained at a_w up to 0.90. The "Mean relative percent deviation modulus (%E) values which are indication of goodness of fit of the GAB data with those of the experimental values were lower than than 10%. Generally, a good fit of isotherm is assumed when the E% value is less than 10% (McLaughlin and Magee, 1998). The E% values were 9.45, 7.377, and 9.61% at 20, 30 and 40°C respectively for soy- melon "ga-ri" samples; while for the control samples they were 3.76, 2.48, and 2.04% at the same temperatures. The RSS va-lues ranged from 0.0002 to 0.0008 for soy-melon gari while it ranged from 0.0001 to 0.0004 for the un- enriched gari at temperature between 20 and 40°C (Table 3). These showed that there was perfect fitness between the experimental moisture sorption data and the predicted

GAB sorption for both the un-enriched (control) "gari" and soy-melon "gari" samples. Hence, GAB model is an appropriate and satisfactory model that could be used to predict the moisture sorption properties of both soy-melon enriched and control "gari" at all the water activities up to 0.92 used in this study. This is consistent with find-ings made for similar products by Chuzel and Zakhia (1991) for un-enriched "gari"; Sanni et al. (1997) for "fufu" and tapioca Kuye and Sanni (2002) for "lafun" (Table 4); Schar and Ruëgg (1985) for wheat flour; Caden (1988) for several food fibers, De Jong et al. (1996) for wheat gluten; and Diosady et al. (1996) for canola meal. Van den Berg (1984), McMinn and Magee (1999) and Timmermann et al. (2001) also reported that the GAB model adequately represented the sorption isotherms of potato, wheat starch and starchy materials respectively. In the range of water activity $0.35 < a_w < 0.9$; Al-Muhtaseb et al. (2004) reported GAB model adequately represented the sorption isotherms of potato, highly amylopectin and highly amylose starch.

However, when the values of both the enriched and unenriched gari were compared, the values for the un-enriched (control) "gari" were consistently lower than those of soy-melon enriched "gari". This shows that there was a better perfect fitness for the un-enriched "gari" than sovmelon enriched "gari". This must have been due to the effect of the oil content in the soy and melon flours which reduced the sorption capacity of the soy-melon enriched "gari" (Oluwamukomi et al., 2008). This suggests that control "gari" has more polar sites on its surface than the soy-melon "gari", and thus, the energy of binding between the water molecules and the surface is higher (Tsami, 1991). This further explains the ability of control "gari" to absorb more moisture than soy-melon "gari" at those water activity levels. Labuza et al. (1985) also confirmed this in dehydrated corn and starch meal. Corn meal had a higher sorption capacity than fish meal which was attributed probably because corn meal is predominantly carbohydrate and has more polar groups which contributes to the increase in the sorption capacity in agricultural products (Iglesias and Chirife, 1982; Fasina et al., 1999).

Conclusion

When the experimental and fitted isotherms were compared, best fits were obtained at $a_{\rm W}$ up to 0.90. All the values were below 10% showing that there was perfect fitness between the experimental moisture sorption and the predicted GAB sorption. Hence, GAB model could be used to predict the moisture equilibrium of both soy-melon enriched and un-enriched (control) "gari" samples at the range of water activities used in this study. However, the values for the un-enriched (control) "gari" were consistently lower than those of soy-melon enriched "gari" showing that there was a better perfect fitness for the un-enriched (control) "gari" than soy-melon enriched "gari".

This must have been due to the effect of the oil content in the soy flour which reduced the sorption capacity of the soy-melon enriched "gari".

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