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THE STUDY OF SORPTION ISOTHERMS FOR VARIED TEMPERATURES OF BITTER KOLA (*GARCINA KOLA*)

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Abstract

Sorption isotherms of bitter kola (Garcina kola) were determined experimentally using the static gravimetric method at 30 ^{0}C, 40 ^{0}C, 50 ^{0}C and 60 ^{0}C within the range of 0.113 – 0.970 water activity. The equilibrium moisture content increased with increasing water activity at a given temperature and decreases with increase in temperature. The experimentally obtained data were used to evaluate the suitability of BET, GAB and Oswin models. The models' parameters were computed using an iterative nonlinear regression technique. The experimental data were also fitted to an empirical model using multiple regression analysis and the results were compared. Monolayer moisture content was determined from BET and GAB models, the values were observed to be consistent with other food materials. The sorption isotherm curves obtained were sigmoidal (typical type II isotherm). The results demonstrated that the three models adequately predicted the equilibrium moisture content of bitter kola. On the basis of fit, all the models were suitable for describing the observed data. The best result on the basis of monolayer moisture content was observed at 30 ^{0}C and 40 ^{0}C. This reveals that at low temperatures, the shelf life of bitter kola is extended.

Keywords: *Bitter kola, Sorption isotherms, Equilibrium moisture content, Monolayer moisture content*

Introduction

The food sorption isotherm describes the thermodynamic relationship between water activity and the equilibrium of moisture content at constant temperature and pressure. The moisture sorption isotherms are extremely important in modeling the drying process, in design and optimization of drying equipment, in predicting shelf-life stability, in calculating moisture changes which may occur during storage and in selecting appropriate packaging material (Ricardo, *et al*., 2011). Also, the knowledge of the sorption data is essentially useful to predict microbiological, enzymatic and chemical stability of food materials (Oyerinde and Lawal, 2015). Experimental determination and modeling of sorption isotherms of food materials has attracted numerous researches because their values are used in industrial purposes. Sorption isotherms are usually classified according to their shape in five different types. The sorption isotherms for most foods are nonlinear and generally with sigmoid shape (Ricardo, *et al*., 2011).

Bitter kola which is also known as 'Garcinia kola' is also a specie of flowering plant in the Clusiaceae or Guttiferae family. It is found in Benin, Cameroon, Democratic Republic of the Congo, Ivory Coast, Gabon, Ghana, Liberia, Nigeria, Senegal and Sierra Leone. Its natural habitat is subtropical or tropical moist lowland forests (Teodoro and Palmeira, 2016). The fruit, seeds, nuts, stem and bark of the plant have been used for centuries in folk medicine to treat ailments from coughs to fever, for dental care in the form of chewing-sticks (Adu-Tutu *et al*. 1979). According to a report from the Center for International Forestry Research, Bitter kola trade is still important to the indigenous communities and villages in Nigeria. It is highly used for its medicinal purposes because of its anti-viral, anti-inflammatory, nanti-diabetic, bronchio-dilator and anti hepotoxic attributes. Fruit extracts from it have proven effective at stopping Ebola virus replication in laboratory test (Iwu and Okunji, 2002; Wootton, 2002; Okoye and Okereke, 2014).

It has been utilized in folklore remedies by the Igbo people of the Southern Nigerian to treat ailments associated with poisoning, liver disorders, hepatitis, diarrhoea, laryngitis, bronchitis and gonorrhea (Adesina, *et al*., 1995; Iwu, 1993). The plant has been described as a "wonder plant" because; every part of it has one or more medicinal value (Adegboye, *et al*, 2008). Some of these uses depend on principles of adsorption. Extensive research findings on nutritional and phytochemical compositions of bitter kola have been reported (Adegboye, *et al*., 2008; Afolabi, *et al*, 2006; Adesuyi, *et al*. 2012; Odebunmi, *et al*. 2009). Traditionally, these nuts were chewed as a masticatory substance, to stimulate the flow of saliva (Leakey, 2001) but are now widely consumed as snack in West and Central Africa. The kernels of the nuts are widely traded and eaten as a stimulant (Leakey, 2001; Omode *et al*. 1995). Bitter kola is also rich in caffeine and threo-bromine and is also believed to be an aphrodisiac. Unlike other kola nuts however, bitter kola is believed to clean the digestive system, without side effects such as abdominal problems, even when a lot of nuts are eaten (Onochie and Stanfield, 1960). In folk medicine, bitter kola is dried, grounded and mixed with honey to make a traditional cough mixture.

The objectives of this study are to determine the experimental sorption isotherm of bitter kola at 30 ^{0}C, 40 ^{0}C, 50 ^{0}C and 60 ^{0}C and to evaluate the best sorption isotherm model to fit experimentally. This is expected to provide information for those involved in their processing and application in food and drug formulation.

Materials and methods

Source and preparation of the material

Bitter kola (*Garcina kola*) was purchased in commercial quantity from the Botany Department of UNIUYO. They were washed, peeled and cut manually with a steel knife, sliced and dried using a hot air oven at 105 ^{0}C until constant weight. The dried samples were grinded into powder of size 2 mm and stored in an air tight container.

Experimental procedure

Determination of the Adsorption Isotherms

The adsorption isotherms were determined using a temperature controlled cabinet (Hot Pack, US); with an accuracy of ±1 ^{0}C at the selected temperatures of 30 ^{0}C – 60 ^{0}C using the static gravimetric method, according to the COST 90 project. This method consists of applying saturated saline solutions to maintain a given constant value of a_w of the *garcina kola* samples, when equilibrium is achieved between the atmosphere and the samples.

Nine saturated saline solutions which include; Lithium Chloride (LiCl), Sodium Chloride (NaCl), Magnesium Chloride ($MgCl_2$), Sodium Bromide (NaBr), Strontium Chloride ($SrCl_2$), Potassium Chloride (KCI), Potassium Carbonate(K_2CO_3), Potassium Iodide (KI) and Pottasium Sulphate (K_2SO_4) were used, which were enclosed in hermetic containers and had a_w values that varied between 0.113 and 0.970. 3 g of *garcina kola* were introduced into each hermetic container with the corresponding saline solution. Thymol was placed into the containers with saline solutions that had a_w values higher than 0.65 to prevent microbial growth. The samples were periodically weighed every three (3) days until a constant weight was achieved, to ensure equilibrium between the samples and the saline solutions. The duration of the periodical weighing of the samples was less than 1 min to avoid any effect on the results. The EMC of the samples was determined using the oven method by AOAC. The adsorption experiments for each temperature were performed in triplicate (Park, *et al.*, 2002).

The water activity values of saturated salt solutions at different experimental temperatures were taken from data reported by Kiranoudis, *et al*., (1993), Julius K (1998) and Lewis G (1976) as given in Table 1 below.

Table 1: Water activity values of saturated salt solutions at different temperatures

Saturated salt solutions	Temperatures (^{0}C)			
	30	40	50	60
Lithium Chloride (LiCl)	0.113	0.112	0.111	0.111
Magnesium Chloride($MgCl_2$)	0.324	0.316	0.305	0.293
Potassium Carbonate (K_2CO_3)	0.432	0.432	0.432	0.432
Sodium Bromide (NaBr)	0.560	0.532	0.509	0.497
Potassium Iodide (KI)	0.679	0.661	0.645	0.631
Strontium Chloride ($SrCl_2$)	0.691	0.673	0.657	0.643
Sodium Chloride (NaCl)	0.751	0.747	0.744	0.745

Pottasium Chloride (KCl)	0.836	0.823	0.812	0.803
Potassium Sulphate (K_2SO_4)	0.970	0.964	0.958	0.952

Mathematical Description of Moisture Sorption Isotherms

Although several mathematical models exist to describe water sorption isotherms of food materials (Labuza, 1968 and Iglesias *et al.*, 1975), no one equation gives accurate results throughout the whole range of water activities, and for all types of foods (Iglesias and Chirife, 1976). The ones used for this study are discussed below.

The Brunauer-Emmett-Teller (BET) Equation

The Brunauer, Emmett and Teller (BET) sorption equation, formulated in 1938, represents a fundamental milestone in the interpretation of multilayer sorption isotherms, particularly Type II and III (Timmermann, 1989); it provides an estimation of the monolayer value of moisture adsorbed on the surface. The monolayer moisture content of many foods has been reported to correspond with the physical and chemical stability of dehydrated foods. However, in almost all cases the so-called BET plots are only linear over the lower relative pressure region (a_w) of the sorbate ($0.05<a_w<0.35$).

The BET equation is generally expressed in the form as shown by Equation 1 below.

$$\frac{M}{M_0} = \frac{Ca_w}{(1-a_w)(1-a_w+Ca_w)} \tag{1}$$

Where M is the moisture content (kg/kg dry solid), M_o is monolayer moisture content (kg/kg dry solid), a_w is the water activity, and C is a constant related to the net heat of sorption. The estimation of the constants is based on linearization of Equation 1.

Guggenheim-Anderson-de Boer (GAB) Equation

The three parameters GAB equation, derived independently by Guggenheim (1966), Anderson (1946) and De Boer (1953), is a semi-theoretical, multi-molecular, localized, homogeneous adsorption model. It has been suggested to be the most versatile sorption model available in the literature and has been adopted by a group of West European food researchers (Van de Berg and Bruin, 1981 and Bizot, 1983). It can be written as Equation 2 as shown below.

$$M = \frac{M_0\ G\ Ka_w}{(1-Ka_w)\ (1-Ka_w+GKa_w)} \tag{2}$$

where M is the moisture content (kg/kg dry solid), M_o is the monolayer moisture content; G and K are constants related to the energies of interaction between the first and further molecules at the individual sorption sites. Theoretically they are related to the sorption enthalpies (Van den Berg *et al.*, 1981) as shown in Equations 3 and 4 below.

$$G = G_0\ exp\left[\frac{H_m - H_n}{RT}\right] \tag{3}$$

$$K = k_0\ exp\left[\frac{H_1 - H_n}{RT}\right] \tag{4}$$

Where G_0 and k_0 are entropic accommodation factors; H_m, H_n and H_1 are the molar sorption enthalpies of the monolayer, multi-layers and bulk liquid, respectively (KJ/mol). The GAB model represents a refined extension of the BET theory, postulating that the state of the sorbate molecules in the second and higher layers is equal, but different from that in the liquid-like state. This assumption introduces an additional degree of freedom (an additional constant, K) by which the GAB model gains its greater versatility. Incorporation of the parameter K, however, assumes that multilayer molecules have interactions with the sorbent that range in energy levels somewhere between those of the monolayer molecules and the bulk liquid. If K is less than unity, lower sorption than that demanded by the BET model is predicted; this allows the GAB isotherm to be successful up to high water activities (i.e. $a_w \approx 0.9$). In the special case where K=1, the GAB equation reduces to the BET equation (if K>1, the sorption isotherm will become infinite at a value of a_w less than unity, which is physically unsound) (Chirife *et al.*, 1992).

Oswin Equation

Oswin (1946) developed an empirical model which is a series expansion for sigmoid shaped curves, and can be written as Equation 5 below.

$$M = A\left[\frac{a_w}{1-a_w}\right]^B \tag{5}$$

Where M is the moisture content (kg/kg dry solid), A and B are constants. Boquet *et al.,* 1978 considered the Oswin equation to be the best one for describing the isotherms of starchy food, and a reasonably good fit for meat and vegetables.

This equation was also used by Labuza, *et al.*, 1972 to relate the moisture contents of non-fat dry milk up to a_w=0.5.

Model Evaluation Methods

The relationship between the equilibrium moisture content and the water activity of the product was predicted by using the equations representing the models commonly used in foodstuff. These models which incorporate the temperature effect have been adopted as standard equations by the American Society of Association Executives (ASAE) for the description of sorption isotherms (ASAE, 1997). The constants were estimated by fitting the mathematical model to the experimental data, using a non-linear regression analysis with Microsoft Excel 2013 software. The quality of the fitting of different models was evaluated by calculating the correlation coefficient (R^2), the mean relative percentage deviation modulus E in percentage (%) and the root mean square error (RMSE) between the experimental and predicted equilibrium moisture content (Boquet *et al.*, 1978 and Basu *et al.*, 2006). The deviation modulus and root mean square error and correlation coefficient are expressed as Equations 6, 7 and 8 below.

$$E = \frac{100}{N} \sum_{i=1}^{N} \frac{|X_{eq,exp} - X_{eq,pre}|}{X_{eq,exp}} \tag{6}$$

$$RMSE = \left[\frac{1}{N} \sum_{i=1}^{N} \frac{(X_{eq,exp} - X_{eq,pre})^2}{X_{eq,exp}} \right]^{\frac{1}{2}} \tag{7}$$

$$R^2 = \frac{(X_{eq,\,exp})}{X_{eq,\,exp} + X_{eq,\,pre}} \tag{8}$$

Where N is the number of observations; $X_{eq,exp}$ and $X_{eq,pre}$ are the experimental and predicted values of the equilibrium moisture content, respectively. The mean relative percentage deviation modulus is widely adopted throughout the literature, with a modulus value below 10 % indicative of a good fit for practical purposes (Lomauro *et al.*, 1985; Kaymak-Ertekin and Gedik, 2004). In the same way, the smaller the RMSE value, the better the fit of the model.

According to Kaleemullah and Kallappan (2004), Aviara *et al.*, 2004) and Basu *et al.*, 2006, statistical parameters like R^2, E and RMSE may not be sufficient evidence for the goodness of fit of a moisture sorption model based on experimental data, but the nature of the residual plots should be considered in addition. A model was considered acceptable if the residuals ($X_{eq,exp} – X_{eq,pre}$) are uniformly scattered around the horizontal value of zero, showing no systematic tendency towards a clear pattern.

Results and Discussions

Moisture Sorption Isotherm of *Garcina Kola* at Varied Temperatures

The experimental data for the equilibrium moisture content (EMC) and water activities as shown in Table 2 below were plugged into nonlinear regression graphs using Microsoft Excel 2013. Figures 1 – 4 below are the adsorption isotherms; they show that the moisture sorption isotherms were temperature dependent. The equilibrium moisture at a given water activity, decreased with increase in temperature. This was in agreement with the theory of physical sorption (Iglesias *et al.*, 1975; Hassian *et al.*, 2001). Implying that *garcina kola* became less hygroscopic with increasing temperature. This was similar to the report on starch (Al-Muhtaseb *et al.*, 2004), pitahya fruit (Ayala *et al.*, 2011) and coffee (Corrêa *et al.*, 2010). The adsorption isotherms exhibited Type II curve. It was consistent with previous researchers on bananas and plantains (Johnson and Brennan, 2000; and Yan *et al.*, 2008).

Table 2: **Summary of the Equilibrium Moisture Content and Water Activity at Varied Temperatures for *Garcina Kola***

30 ºC		40 ºC		50 ºC		60 ºC	
a_w	EMC	a_w	EMC	a_w	EMC	a_w	EMC
0.113	0.050	0.112	0.049	0.111	0.048	0.111	0.045
0.324	0.070	0.316	0.068	0.305	0.066	0.293	0.065
0.432	0.081	0.432	0.080	0.432	0.079	0.432	0.077
0.560	0.110	0.532	0.100	0.509	0.097	0.497	0.095
0.679	0.112	0.661	0.120	0.645	0.117	0.631	0.114

0.691	0.127	0.673	0.125	0.657	0.120	0.643	0.118
0.751	0.200	0.747	0.199	0.744	0.197	0.745	0.190
0.836	0.330	0.823	0.310	0.812	0.290	0.803	0.285
0.970	1.835	0.964	1.830	0.958	1.790	0.952	1.750

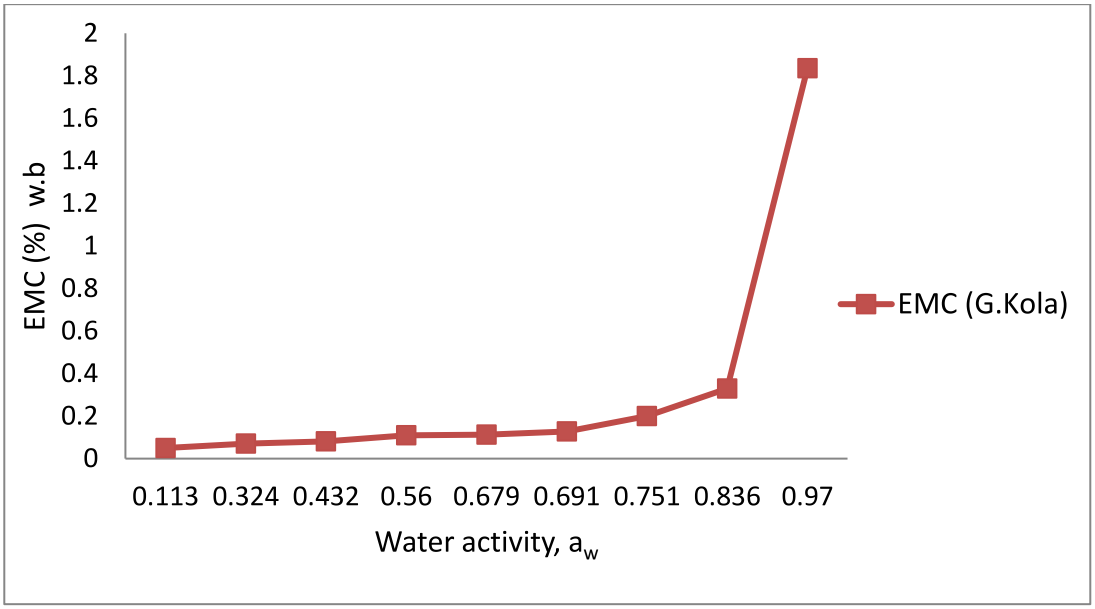

Figure 1: Adsorption isotherm of *garcina kola* at 30 °C

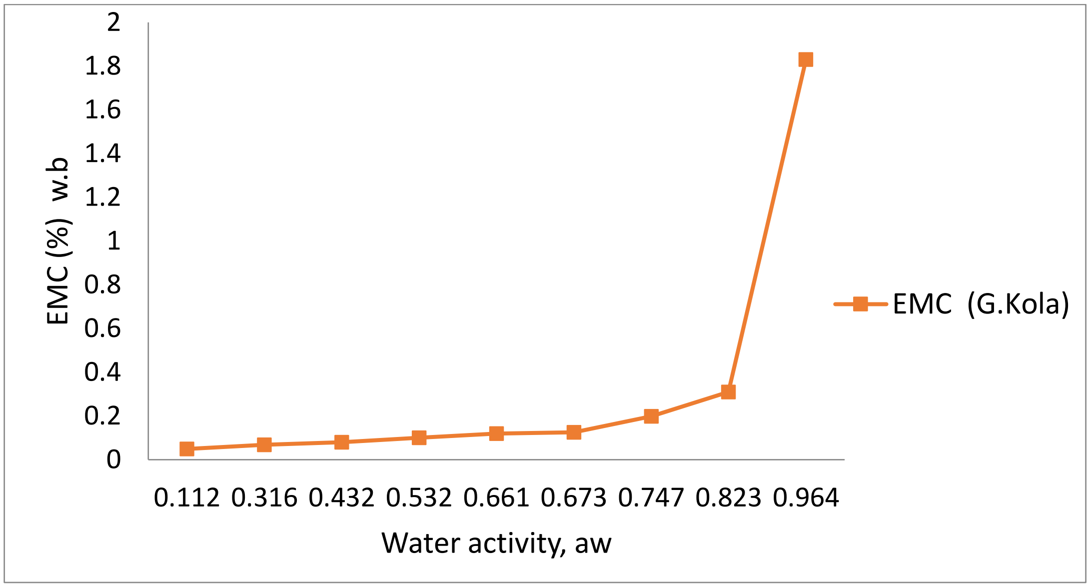

Figure 2: Adsorption isotherm of *garcina kola* at 40 °C

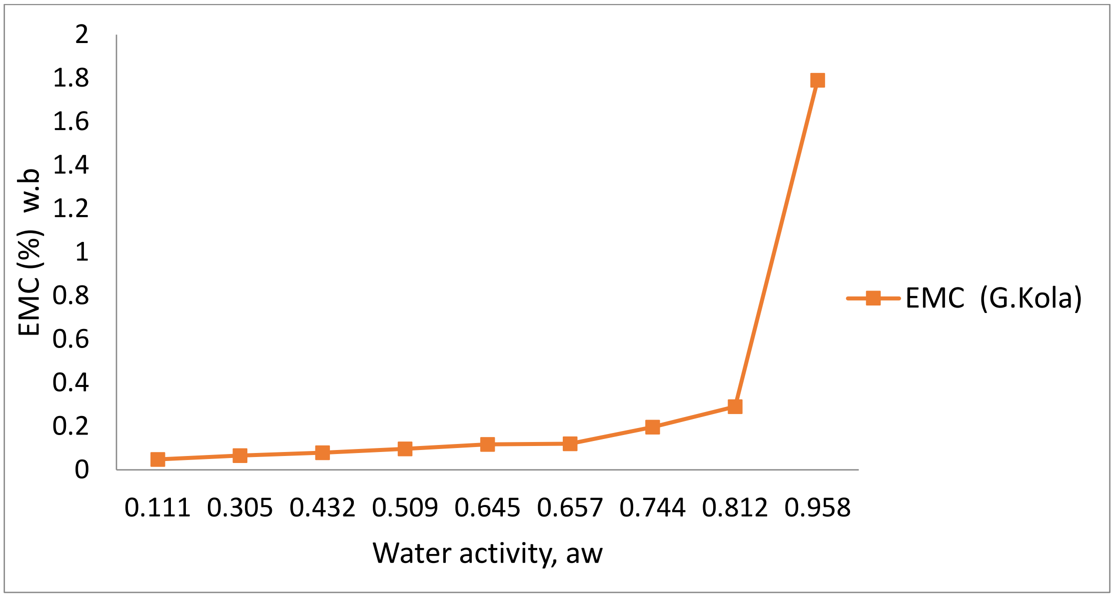

Figure 3: Adsorption isotherm of *garcina kola* at 50 ºC

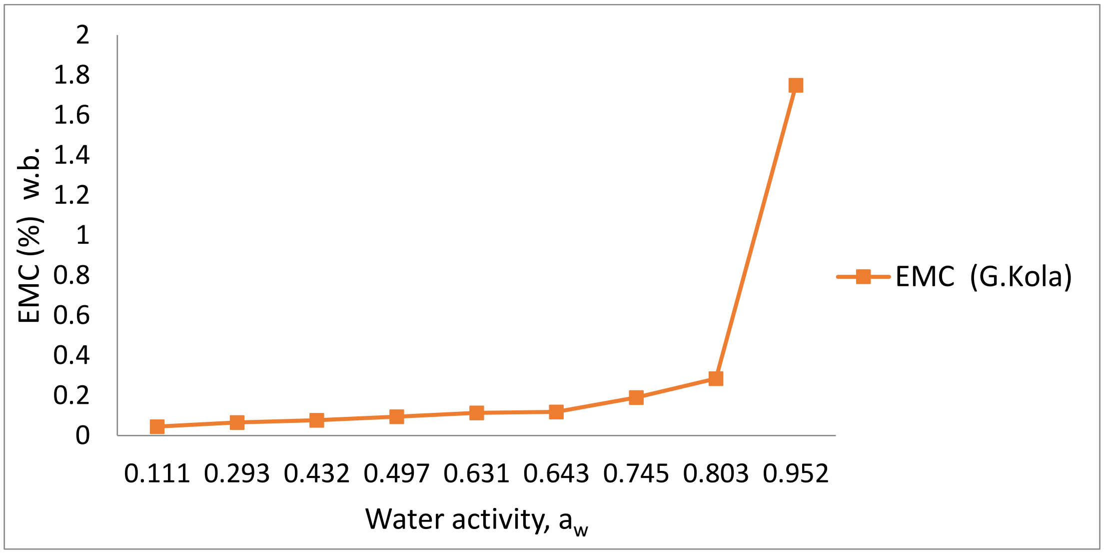

Figure 4: Adsorption isotherm of *garcina kola* at 60 ºC

Fitting Models to Moisture Sorption Isotherms *Garcina Kola*

Table 3 below shows the model constants and statistical errors for adsorption isotherms using *garcina kola* as case study. It was observed that the relationship between monolayer for BET and GAB were not totally consistent. GAB monolayer values almost fell within the monolayer for starchy foods which generally range from 0.032 – 0.16 (kg water/kg dry matter) (Siripatrawan and

Jantawal, 2006) except for Timmermann *et al.*, (2001). BET monolayer values were less than the GAB'; but at 30 and 40 °C, the constant C values for BET were lesser than the constant K values of GAB in the adsorption isotherms at the temperatures.

Also, the GAB constant values for adsorption were less than unity. This shows that they all fell within the recommended range of $0.7 < k < 1$ (Timmermann *et al.*, 2001). Provided that the water activity level of 0.6 corresponds with equilibrium moisture content of 0.11 (kg water/kg dry-matter) and equivalent to 10% (w.b) on the adsorption isotherm at 30 and 40 °C, the GAB monolayer values satisfy the condition. This implies that the equilibrium moisture content for microbiologically shelf-stable dried *garcina kola* is equal to 10% (w.b) at 30 and 40 °C.

The three models fitted to describe both the adsorption isotherm for *garcina kola w*ere: BET, GAB and Oswin. The models with $R^2 > 0.95$, $E \leq 10\%$ and low values of RMSE were considered to be the best fitting models to describe *garcina kola* moisture isotherm. The results for statistical errors used to evaluate the best fitting models together with model constants are given in Table 3 below. It was observed from tables that all models fitted had low values for RMSE, which indicated that all models could be used to describe adsorption moisture isotherms for *garcina kola*. However, the computed values of E % indicated that none of the three models E-value was lesser than 10 %. This indicated that although the RMSE had low values, R^2 values were less than unity, for all models, The E-values eliminated the three models because the values were greater than 10 %. On the basis of monolayer, BET and GAB model best describe the adsorption moisture isotherm at 30 and 40 °C. From the observation of the results, the three models described all the adsorption moisture isotherms for *garcina kola*.

Table 3: Estimated Parameters by Nonlinear Regression for BET, GAB and OSWIN Models *for Garcina Kola*

Models	Constants	Temperatures (°C)			
		30	**40**	**50**	**60**
BET	M_o (g/g dry solid)	0.181576	0.080867	0.041238	0.032293
	C	0.552673	0.917647	1.386102	1.706528
	R^2	0.99835	0.998261	0.997453	0.993035
	RMSE	0.02211	0.022701	0.026876	0.045547

	E (%)	12.56221	16.31022	17.69961	10.38048
GAB	M_o (g/g dry solid)	0.072897	0.050659	0.029747	0.023466
	K	0.989682	0.999365	0.974841	1.007904
	G	1.002062	1.174062	1.666545	1.796874
	R^2	0.996416	0.998076	0.997479	0.997517
	RMSE	0.032727	0.023936	0.026832	0.026348
	E (%)	16.50219	21.07163	20.83303	25.12812
OSWIN	A	0.079592	0.068519	0.065333	0.062802
	B	0.903281	0.999387	1.058778	1.114019
	R^2	0.99678	0.998234	0.99791	0.997649
	RMSE	0.030881	0.022931	0.02446	0.025406
	E (%)	11.87691	18.26048	21.06275	23.0462

Conclusion

This study has specifically provided the following information on moisture sorption isotherm for varied temperature of bitter kola (*garcina kola*) for the first time, and the following have been revealed:

i. Bitter kola isotherms exhibited type II curves behavior which is characteristic of foodstuffs as reported by Johnson and Brennan, 2000 and Yan *et al.*, 2008. The equilibrium moisture content decreases with increase in temperature at a given water activity, indicating that bitter kola becomes less hygroscopic with increase in temperature giving a clear stability domain after drying leading to extensive shelf life. This complements Muranga, (1998), who attributed the extensive shelf life to low sugar and fat.

ii. The equilibrium moisture content of *garcina kola* decreases with increase in temperature at a given water activity, indicating that they become less hygroscopic at high temperatures giving a clear stability domain of *garcina kola* after drying leading to extensive shelf life. This compliments Muranga (1998), who attributed the extensive shelf life to low sugar and fat.

iii. For shelf stability of dried *garcina kola*, it is recommended to dry it to moisture content below or equal to 0.11 (g water/g dry matter) which is equivalent to 10 % (w.b).

iv. On the basis of monolayer, BET and GAB models best describe the adsorption moisture isotherm for *garcina kola* at 30 and 40 ^oC.

v. The three models properly described all the adsorption and moisture isotherms for *garcina kola*.
vi. The relationship between monolayer for BET and GAB for all cases were consistent with Timmermann *et al.*, (2001).
vii. The data obtained from this study are useful in characterization, selection of appropriate packaging material, design of processing machines and generally in a post-harvest handling of bitter kola.

Recommendation

From literature survey during this study, it was noted that the information in some areas of bitter kola were inadequate. Based on this fact, further research is recommended in the following areas:

i. Sorption isotherms of bitter kola pellets.
ii. Diffusion models for predicting the drying behaviour of bitter kola.
iii. Effects of relative humidity on drying rates of pelletized bitter kola.
iv. The use of other models to provide wider range results for better comparative analysis of experimental data.

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