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THE STUDY OF SORPTION ISOTHERMS FOR VARIED TEMPERATURES OF COCOYAM (*TARO*)

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Abstract

The sorption isotherms of cocoyam (taro; colocasia esculenta) pellets were determined experimentally using the static gravimetric method at 30 °C, 40 °C, 50 °C and 60 °C for water activity ranging from 0.113 – 0.970. The equilibrium moisture content increased with increasing water activity at a given temperature and decreased with increase in temperature. The experimentally obtained data were used to evaluate the suitability of Brunaeur-Emmet-Teller (BET), Guggenheim-Anderson-de Boer (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 experimental values were consistent in their relationship but differ when compared with other starchy food materials. The sorption isotherm curves obtained were sigmoidal (typical type II isotherm) and the results demonstrated that the three models adequately predicted the equilibrium moisture content of cocoyam. At the end of the analysis, it was observed that the BET and Oswin model best describe the adsorption moisture isotherm at 30 °C, 40 °C and 50 °C respectively.

Keywords: *Sorption Isotherms, Cocoyam, Static gravimetric method, Equilibrium moisture content, Monolayer moisture content*

Introduction

Cocoyam is regarded as the third most important root crop after yam and cassava in West Africa (Obomegheive *et al.*, 1998). It is a staple food for millions of people living in the tropics. Onwueme and Simha (1991) reported that cocoyam cultivars have yield potentials for 37-75 tonnes/hectare and the corms and cormels are rich in minerals, vitamins and digestive starch grains. Despite these nutritional benefits, cocoyam is less valued in areas like Eastern Nigeria where it is produced in abundance (IITA, 1992; FAO, 2006). According to Kordylas (1990), about 30-40 species of cocoyam have been identified but only 5-6 specie produce edible parts. Two genera of cocoyam are widely cultivated in Africa; these are namely taro (*Colocasia esculenta*) and tannia (*Xanthosoma sagittifolium*). Cocoyam is one of the under exploited tropical plants though with promising quality. However, research and development on cocoyam have been meager in Nigeria when compared with other tropical root crops like yam and cassava (Onwuka and Eneh, 1998). Among the reasons for the underutilization of cocoyam is due to the presence of calcium oxalate raphide; the irritant which causes itching effect felt throughout the throat when consumed (Purseglove, 1983). Another reason is that cocoyam is prone to pre-harvest and post-harvest diseases, which reduce storage stability and quality of the tubers (Hahn *et al*., 1987). To overcome these losses, Onyeike *et al*., (1995) reported that the corms and cormels may be processed into flour. Kwarteng and Towler (1994) reported that the flours store much longer than the unprocessed tubers of cocoyam. Though cocoyam flour stores better than the corm due to highly reduced moisture content, conversion into flour is not an end to the storage problem. According to Leniger and Beverloo (1975) and Desrosier and Desrosier (1977), all food products are inherently unstable and quality retention depends upon a number of factors including storage temperature, storage relative humidity and storage time. Rockland and Nishi (1980) also reported that maximum stability of natural products had been associated with minimum total moisture until definitive studies on shelled walnuts demonstrated that there was a narrow optimum moisture range, corresponding to a broader water activity (a_w) range, above and below which kernels deteriorated at more rapid rate; and that analogous optimum a_w values are observed for other natural products. Thereafter, it became generally accepted that a_w is more closely related to the physical, chemical and biological properties of foods and other natural products than its total moisture content. Recent works (Owuamanam *et al.,* 2010; Nwanekezi *et al*., 2010: Nurtama and Lin (2010) have studied the moisture sorption isotherm of cocoyam flour, which according to Idlimam *et al*., (2008) are important to improve the conditions of several processes such as dehydration, packaging or storage. According to Iwuoha and Kalu (1995), proper cooking eliminates the harsh and sharp irritation in the throat and mouth while the post-harvest losses would be obviated by prompt processing of the harvested tubers into cocoyam flour. According to Enwere (1998), the cormels of cocoyam are used traditionally as soup thickeners. Some skeletal works have been reported on some proximate and functional properties as well as their industrial application (Osisiogwu *et al*., 1974; Olaofe *et al*., 1998).

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).

The objectives of this study are to determine the experimental sorption isotherm for cocoyam pellets at 30 ^{o}C, 40 ^{o}C, 50 ^{o}C and 60 ^{o}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

Cocoyam (*taro*) 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 ^{o}C until constant weight. The dried samples were further reduced into pellets of 2 mm.

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 ^{o}C at the selected temperatures of 30 ^{o}C – 60 ^{o}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 *taro pellets* 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 (1998) and Lewis (1976) as given in Table 1 below.

Table 1: Water Activity Values of Saturated Salt Solutions at Different Temperatures

Saturated salt solutions	Temperatures (^{o}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.

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

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.

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

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.

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

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

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.

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

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.

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

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

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

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 *Taro* 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 *taro* 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 *Taro***

30 ºC		40 ºC		50 ºC		60ºC	
a_w	EMC	a_w	EMC	a_w	EMC	a_w	EMC
0.113	0.010	0.112	0.007	0.111	0.005	0.111	0.004
0.324	0.015	0.316	0.010	0.305	0.007	0.293	0.006
0.432	0.018	0.432	0.013	0.432	0.009	0.432	0.008

0.560	0.027	0.532	0.024	0.509	0.020	0.497	0.018
0.679	0.052	0.661	0.048	0.645	0.044	0.631	0.041
0.691	0.053	0.673	0.049	0.657	0.045	0.643	0.043
0.751	0.062	0.747	0.058	0.744	0.052	0.745	0.049
0.836	0.117	0.823	0.113	0.812	0.111	0.803	0.109
0.970	0.700	0.964	0.570	0.958	0.483	0.952	0.409

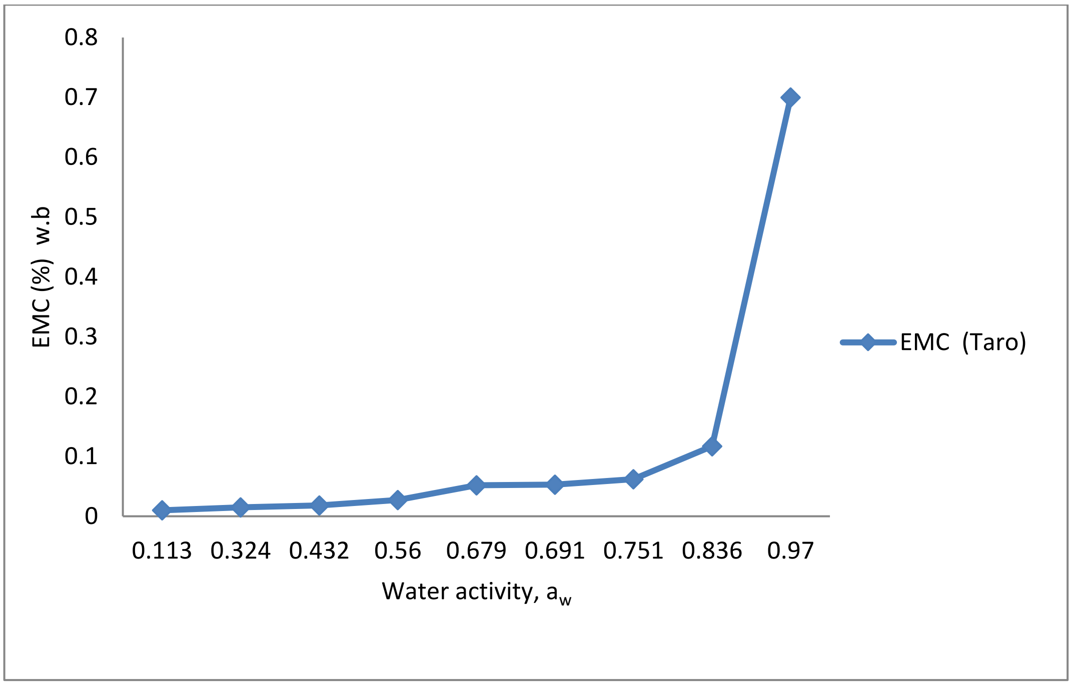

Figure 1: Adsorption isotherm of *taro* at 30 ºC

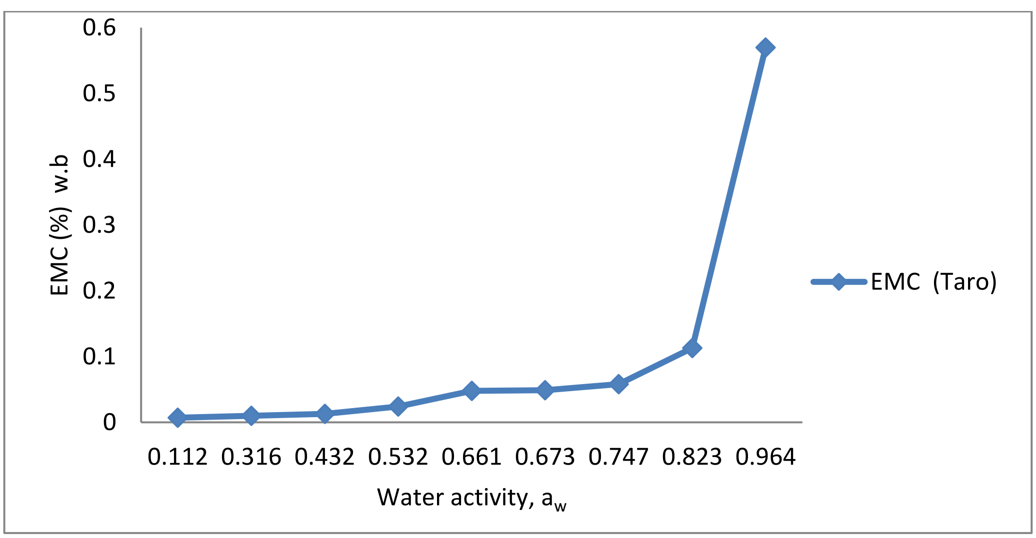

Figure 2: Adsorption isotherm of *taro* at 40 ºC

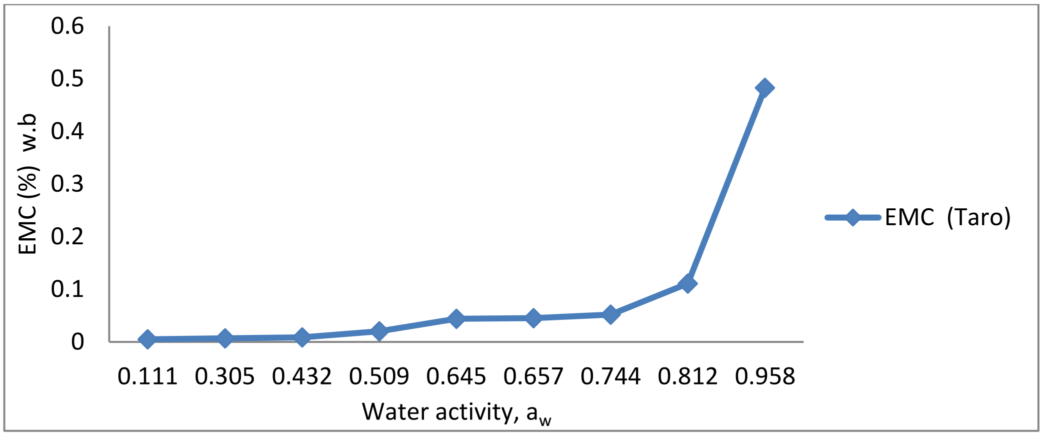

Figure 3: Adsorption isotherm of *taro* at 50 ºC

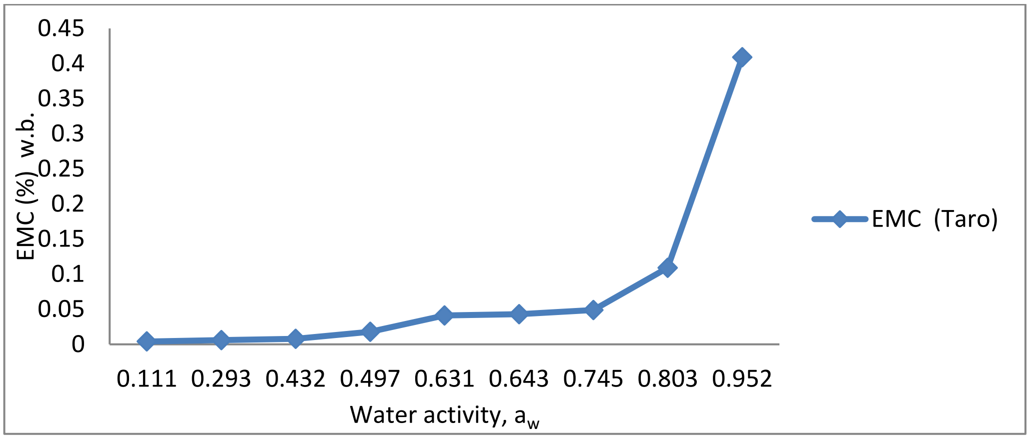

Figure 4: Adsorption isotherm of *taro* at 60 ºC

Fitting Models to Moisture Sorption Isotherms for *Taro*

Tables 3 below shows the model constants and statistical errors of adsorption isotherms for *taro*. These data were generated using the BET, GAB and Oswin models. It was observed that the relationship between monolayer for BET and GAB for adsorption were consistent with Timmermann *et al.*, (2001), for which the BET monolayer was less than the GAB monolayer; and the constant C for BET was greater than the constant K of GAB in the adsorption isotherms.

All the BET and GAB monolayer values approximately fell within the monolayer for starchy foods which generally range from 0.032 – 0.16 (kg water/kg dry matter) (Siripatrawan and Jantawal, 2006).

Also, the GAB constants for adsorption were 0.9913, 0.99025, 0.877883 and 0.989999 at 30 ˚C, 40 ˚C, 50 ˚C and 60 ˚C respectively. This shows that they all fall within the recommended range of 0.7 < k < 1 (Timmermann *et al.*, 2001). The GAB monolayers for adsorption isotherm at the experimental temperatures were between the ranges of 0.02872 to 0.024434. These values are below the recommended safe water activity level of 0.6 (Labuza *et al.*, 1972; Yan *et al.*, 2008; Sahu & Tiwari, 2007). The water activity level of 0.6 corresponds with equilibrium moisture content of 0.11 (kg water/kg dry-matter) which is equivalent to 10% (wet base) on the adsorption isotherm. This implies that the equilibrium moisture content for microbiologically shelf-stable dried *taro* pellets is less than 10% (wet base).

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 *taro's* moisture isotherm. It was observed from Table 3 that all models fitted had low RMSE values, R^2 values were less than unity, for all models and E values fluctuated. BET and Oswin model best describe the adsorption moisture isotherm at 30 °C and 40 °C and 50 °C. Therefore the three models described all the adsorption moisture isotherms for *taro*.

Table 3: Estimated parameters by Non-linear Regression for BET, GAB and OSWIN Models for *Taro*

Models	Constants	Temperatures (^{0}C)			
		30	40	50	60
BET	M_0 (g/g dry solid)	0.021510	0.021155	0.021111	0.020672
	C	1.291558	1.284853	1.182593	1.206428
	R^2	0.999672003	0.999034687	0.997019764	0.993727462
	RMSE	0.003786	0.005265	0.007858	0.009655
	E (%)	7.517906	1.07209	13.1379	19.396
	K	0.9913	0.99025	0.877883	0.989999
	G	1.210937	1.090866	1.154837	1.150999
	R^2	0.994268	0.995156	0.997222	0.993003
	RMSE	0.0016255	0.0012121	0.007544	0.00989
	E (%)	14.9403	19.5608	16.0394	33.1579
	A	0.024294	0.024151	0.023377	0.023377

OSWIN					
	B	0.971778	0.962395	0.962395	0.961963
	R^2	0.999258	0.999171	0.996423	0.998151
	RMSE	0.005787	0.004874	0.004874	0.008363
	E (%)	6.461344	0.47165	0.47165	15.1069

Source: Computed by the researcher from calculated data

Conclusion

This study has specifically provided the following information on moisture sorption isotherm for varied temperature of cocoyam (*taro*) pellets 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 taro 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 *taro* 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 *taro* after drying leading to extensive shelf life. This compliments Muranga (1998), who attributed the extensive shelf life to low sugar and fat.

iii. The relationship between the monolayer for BET and GAB for adsorption are consistent with Timmermann *et al*., (2001), for which the BET monolayer was less than the GAB monolayer; and the constant (C) for BET was greater than the constant K of GAB in the adsorption isotherms.

iv. The BET and GAB monolayer values were not within the monolayer for starchy foods which generally range from 0.032 – 0.16 (g water/g dry matter) (Siripatrawan & Jantawal, 2006).

v. BET and Oswin model best describe the adsorption moisture isotherm at 30 ^oC, 40 ^oC and 50 ^oC.

vi. 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 cocoyam.
vii. The three models properly described all the adsorption and moisture isotherms for *taro*.

Recommendation

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

i. Drying characteristics of cocoyam subjected to repetitive wetting and drying cycles.
ii. Diffusion models for predicting the drying behaviour of cocoyam.
iii. The use of other models to provide wider range results for better comparative analysis of experimental data.

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