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ORIGINAL RESEARCH ARTICLE

MOISTURE SORPTION CHARACTERISTICS OF DEHYDRATED IN-SHELL AFRICAN WALNUT *(TETRACARPIDIUM CONOPHORUM)*

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ABSTRACT

Moisture sorption isotherms are useful thermodynamic tools for determining water interactions within food systems and providing information that can assist in optimizing food processing operations such as drying, mixing, packaging and storage, as well as to maximize retention of quality parameters such as colour, aroma, texture, and nutrient. Moisture sorption isotherm characteristics of African walnut were evaluated at three different temperatures (28, 33 and 38°C) and relative humidity range of 11.20 - 97.00 % using gravimetric method; five mathematical models (GAB, BET, Peleg, Smith and Ferro Fontan) were fitted into the experimental data. Sorption isotherms of the dehydrated walnut gave type II (S-shaped) isotherms according to BET classification. Temperature had significant effect on the equilibrium moisture content (EMC). A nonlinear regression analysis method was used to evaluate the constants of sorption models. The models were evaluated statistically by calculating coefficient of determination (R^2), the mean relative percentage error (P) and the reduced chi-square (λ^2). The BET model gave the best fit for the obtained data among the tested models with R^2 value of 0.9892. Calculated monolayer moisture (M_o) content from BET ranged from 5.018 to 7.922% db for adsorption and 9.842 to 10.143% db for desorption respectively.

1.0 Introduction

African walnuts (*Tretracarpidium conophorum*) can be described as rounded, stone fruits of the walnut tree with single seed. The fruit of the walnut is enclosed in a hull which is green, leathery, and fleshy. After harvest, this hull is usually removed to reveal the wrinkly walnut shell that encloses the kernel, and the shell is then removed to obtain the kernel (Atungulu *et al.*, 2013). Primarily, walnut plant is cultivated in Nigeria for the nuts, which is traditionally eaten after boiling (Akpuaka and Nwankwor, 2000; Ndie *et al.*, 2010).

Biochemically, walnut is composed of polyunsaturated fatty acids, especially linoleic and oleic acid, it is also high in protein content (Savage *et al.*, 2001), which makes it of significant economic value and medicinal importance for human health; due to these facts, there has been an increasing interest in its consumption and utilisation. Some of the other beneficial components it contains include plant protein (for example, arginine, leucine), carbohydrates (for example, dietary fibre),

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vitamins (for example, vitamin A and E), pectic substances, minerals (magnesium, potassium, phosphorus, sulphur, copper and iron), plant sterols and phytochemicals (Savage *et al*., 2001; Colaric *et al*., 2006; Ogunmoyole *et al*., 2011).

Although African walnuts have high socio-economic potentials, the product is still under-utilised in Nigeria, especially at industrial level. This can mainly be attributed to the fact that there is lack of storage facilities for the walnut market which has been hampering the production on a full scale and exploration of its inherent potentials (Ekwe and Ihemeje, 2013). Thus, in order to overcome the challenge of spoilage and inconsistent seasonal availability, there is need for development and provision of appropriate storage facilities for the African walnut (Babalola, 2011) as well as appropriate preservation methods.

Deterioration of food occurs during handling or due to mechanical, physical, chemical or microbial damage (Rahman, 1999; Mujumdar, 2004); microbial growth depends on the storage conditions and the moisture level in the product. Among the commonly employed methods for preserving food such as freezing, vacuum packing, canning, preserving in syrup, food irradiation, addition of preservatives, the most popular is dehydration (Jangam *et al*., 2010).

1.1 Research Justification

Dehydration which removes water from food materials will result in its stability and as well reduce storage and transportation cost. To a great extent, most dried foods quality depends upon their physical, chemical and microbiological stability, which is mainly a consequence of the relationship between the equilibrium moisture content (EMC) of the food material, and its correspondence water activity (a_w) or relative humidity at a given temperature (Guilan *et al*., 2007).

Moisture sorption isotherms (MSI) is the terminology usually used to express the relationship between equilibrium moisture content and equilibrium relative humidity (ERH) or water activity of materials being studied. It is worthy of note to know that this relationship is complex and unique for each product due to different interactions which may be colligative, capillary or surface effects between the water and the solid components of the product at different moisture contents. According to Al-Mahasneh *et al*., 2011, MSI are useful thermodynamic tools for determining water interactions within food systems and providing information that can assist in optimizing food processing operations such as drying, mixing, packaging and storage, as well as to maximize retention of quality parameters such as colour, aroma, texture, and nutrient. Several authors (Johnson and Brennan, 2000; Chowdhury *et al*., 2005; Akanbi *et al*., 2006; Samapundo *et al*., 2007; Oyelade *et al*., 2008) have reported some of the importance of knowing the sorption characteristics of products as being essential in respect to storage stability and food product acceptability, drying process modelling, design and optimization of drying equipment, aeration, calculation of moisture changes which may occur during storage and for selecting appropriate packaging materials for dehydrated products. Isosteric heat of sorption, often referred to as differential heat of sorption, is used as an indicator of the state of water adsorbed by the solid particles (Fasina *et al*., 1997; Togrul and Arslan, 2007).

Water sorption properties of foods can be predicted using water sorption isotherm equations. Guilan *et al*., 2007 noted that many empirical and semi-empirical equations describing the sorption characteristics of foods have been proposed in the literature; these equations are suitable for some food products only, or for selected ranges of a_w, and part of these equations clearly show the effect of temperature on sorption isotherms. According to Togrul and Arslan (2007), among the models which were reviewed, 23 common equations for fitting sorption isotherms to different food materials were reported, some of the models took into consideration the effect of temperature; these include modified Chung-Pfost (Chung and Pfost, 1967), modified Henderson (Henderson, 1952), modified Halsey (Halsey, 1948) and modified Oswin (Oswin, 1946) models. Brunauer–Emmett–Teller (BET) and Guggenheim–Anderson–de Boer (GAB) equations were reported to be the most popular food isotherm equations (Cadden, 1988; Togrul and Arslan, 2007).

While the sorption characteristics of other exotic walnut of the Juglandaceae family (*Juglans regia, microcarpa, hindsii* cultivars) have been studied extensively, there is dearth of information on the sorption characteristics of African walnut (*Tetracarpidium conophorum*) specie available in Nigeria and other neighbouring African countries. The study of sorption isotherm properties of African walnut will provide information that can be used directly to know the storability, solve process design problems (e.g. dryer design and operations), predict energy requirements and determine its proper storage conditions (Ekwe and Ihemeje, 2013).

1.3 Objectives

This study therefore experimentally determined the sorption characteristics of dehydrated in-shell African walnut kernels at three different temperatures of 28, 33, and 38 °C, evaluated the suitability of five moisture isotherm model equations and investigated the nature of the moisture sorption hysteresis.

2.0 Materials and Methods

2.1 Materials

Raw unshelled walnuts were obtained from Ogbese market, Ondo State, Nigeria. All other reagents used were of analytical grade.

2.1.1 Sample Treatment

The bulk quantity of walnuts was cleaned and divided into two portions. One portion of the walnuts used for adsorption isotherm was dried in an air-draught oven at 120 °C for 10 hours to obtain a moisture content of about of 4 % (dry basis). The other portion used for desorption process was first dampened by placing in known amount of water so as to allow them pick up moisture for 4 days This enabled the moisture content to be raised to a stable and uniform level.

2.2 Methods

2.2.1. Experimental sorption isotherm

Water sorption isotherms (adsorption and desorption) were determined by gravimetric method which involved exposure of samples to different salts (LiCl, CH_3COOK, $MgCl_2.6H_2O$, K_2CO_3, $Mg (NO_3)_2.6H_2O$, NaBr, $SrCl_2.6H_2O$, NaCl, $(NH_4)_2SO_4$, $BaCl_2.2H_2O$) at a_w of 0.112 to 0.970 as shown in Table 1. The salts were employed as saturated solutions to give constant water activity environment. The samples were placed on perforated lid in different desiccators containing the salts. After which the desiccators were placed in a temperature-controlled chamber at different temperatures of 28, 33 and 38°C covering the main range of possible storage condition (to investigate the effect of different temperatures).

Samples were weighed consistently daily until a constant weight was achieved. Moisture content was determined by AOAC vacuum oven method (AOAC, 1990) at 60°C. An analytical balance (model) with a sensitivity of 0.1mg was used to measure water uptake. This procedure was performed in triplicate.

Table 1: Amount of water and salt used to prepare the different saturated salts and expected water activity

No.	Name	Salt (g)	Distilled water (g)	a_w (30°C)
1.	LiCl	112	63	0.112
2.	CH_3COOK	126	79	0.226
3.	$MgCl_2.6H_2O$	300	100	0.327
4.	K_2CO_3	300	135	0.431
5.	$Mg(NO_3)_2.6H_2O$	225	34	0.528
6.	NaBr	300	120	0.577
7.	$SrCl_2.6H_2O$	300	75	0.708
8.	NaCl	300	90	0.752
9.	$(NH_4)_2SO_4$	300	120	0.800
10.	$BaCl_2.2H_2O$	375	105	0.903
11.	K_2SO_4	20	100	0.970

2.2.2 Determination of moisture content

Moisture content was determined by gravimetric method (AOAC, 2000). Five gram (5g) of the sample was pre-weighed (W_1) in a petri dish and placed in an oven at 105 °C for 3 hour. The sample was removed from the oven, cooled in a desiccator, and re-weighed. It was taken back to oven for another 1 hour, cooled and weighed, this was repeated until a constant weight was achieved and recorded as (W_2). All analyses were carried out in triplicates.

Moisture percentage was calculated as shown in Equation 1:

$$moisture\ (\%) = \frac{(W_1 - W_2)}{W_1} \times 100 \tag{1}$$

2.2.3 Data Analysis: Mathematical Modelling of Moisture Sorption Isotherm

Five mathematical equations namely; GAB, BET, Peleg, Smith and Ferro Fontan were used for describing desorption and adsorption isotherms of in-shell walnut in the range of temperature 28, 33 and 38^{0}C. The expressions and the parameters of the five models used to fit the data are presented in Table 2.

The goodness of fit of the data to the equations was evaluated by the criteria of correlation coefficient (R^2), the mean relative percentage error (*P*) and the reduced chi-square (X^2) presented as in equations 2, 3 and 4 respectively. The higher the R^2 value and the lower the X^2 and P values, the better is the goodness of the fit.

$$R^2 = \frac{\sum_{i=1}^{n}\left(M_{\exp,i} - M_{pre,i}\right)\sum_{i=1}^{n}\left(M_{pre,i} - M_{\exp,i}\right)}{\sqrt{\left[\sum_{i=1}^{n}\left(M_{\exp,i} - M_{pre,i}\right)\right]^2\left[\sum_{i=1}^{n}\left(M_{pre,i} - M_{\exp,i}\right)^2\right]}} \tag{2}$$

$$P = \frac{100}{N}\sum_{i=1}^{N}\frac{M_{\exp,i} - M_{pre,i}}{M_{\exp,i}} \qquad (3)$$

$$X^2 = \frac{\sum_{i=1}^{N}(M_{exp,i} - M_{pre,i})^2}{N-n} \qquad (4)$$

where, $M_{exp,I}$ = *i*th experimentally observed equilibrium moisture content, $M_{pre,I}$ = *i*th predicted equilibrium moisture content, *N* = number of observations and *n* = number of constants (Kaya and Kahyaoglu, 2007). Where A, B, C and D are parameters of the equations, T is temperature (°C), X_e is equilibrium moisture content (kg/kg d.b.), and a_w is the water activity (Kaya and Kahyaoglu, 2007).

Table 2: Mathematical models used to describe moisture adsorption and desorption isotherm behaviours of the dehydrated African walnut

Model name	Equilibrium Moisture Content Model equation
Smith	$X_e = A - B\ln(1 - a_w)$
GAB	$X_e = \frac{CABa_w}{(1 - Ba_w)(1 - Ba_w + ABa_w)}$
Ferro-Fontan	$X_e = \left\{1 \Big/ \left[\frac{1}{\alpha}\ln\left(\frac{\gamma}{a_w}\right)\right]^{\frac{1}{r}}\right\}$
Peleg	$X_e = Aa_w^{\ B} + Ca_w^{\ D}$
BET	$X_e = \frac{ABa_w}{(1 - a_w)(1 + (A - 1)a_w)}$

3.0 Results and Discussions

3.1 Moisture Sorption Isotherms of Dehydrated Walnut: Effect of Temperature on Sorption Isotherms

The isotherms shown in figures 1 and 2 were obtained by plotting the corresponding mean EMC of three replicates against a_w at each temperature for adsorption and desorption processes respectively. The effect of temperature on the moisture sorption isotherm reveals that there was an initial increase in the equilibrium moisture content as temperature increased to 33°C and then the equilibrium moisture content decreased as temperature further increased to 38°C at constant a_w (0.226 – 0.800), but there was decrease in the equilibrium moisture content with increasing temperature, at a constant a_w (0.903 – 0.970); the decrease in EMC with increasing temperature signifies that the product became less hygroscopic with increasing temperature which means it can absorb more moisture at lower temperatures than at higher temperatures at constant relative humidity environment (Ariahu *et al.,* 2006). According to Ronald *et al.*, (2005), this can be explained by the change in the excess enthalpy of water binding, dissociation of water, or increase in solubility of solute in water as temperature increases. Also, this trend may be due to a reduction in the total number of active sites for water binding as a result of physical and/or chemical changes in the product induced by temperature (Mazza and LeMaguer, 1980; Moreira *et al.*, 2008; Saad *et al.*, 2014). The effect of temperature on the sorption isotherm is of great importance (Falade and Awoyele, 2005), this is due to the fact that foods are exposed to a range of temperatures during storage and processing, and also a_w changes with temperature. Temperature affects the mobility of the water molecules and the dynamic equilibrium between the vapour and adsorbed phases (Al-Muhtaseb *et al.*, 2004).

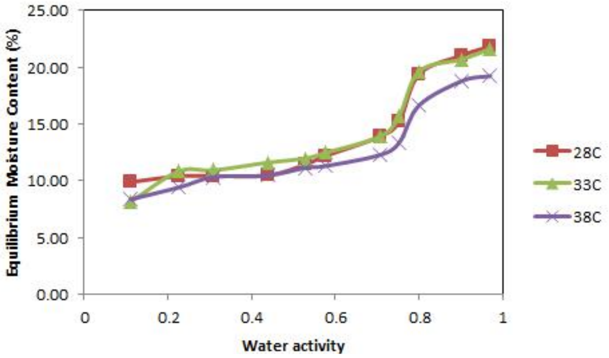

Figure 1: Effect of temperature on the adsorption isotherm of dehydrated African walnut

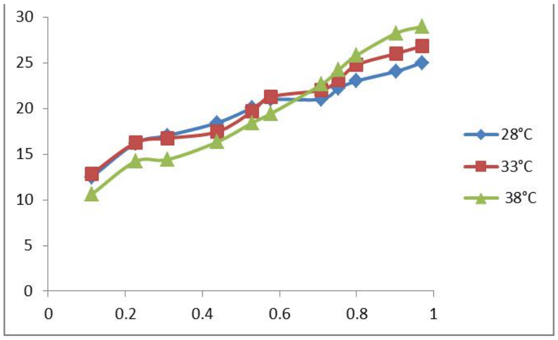

Figure 2: Effect of temperature on the desorption isotherm of dehydrated African walnut

The monolayer moisture content (M_O) for each temperature, calculated using the BET and GAB models, is presented in Table 3. In general, the M_O value calculated using the BET model was lower than that calculated using the GAB equation this is in agreement with the reports of other researchers (Erbas *et al*., 2005; McMinn *et al*., 2007; Lee and Lee, 2008).

For adsorption, M_O decreased as temperature increased while it decreased for desorption isotherm. The behaviour of M_O decreasing with increase in temperature been ascribed to a reduction in the number of active sites due to physical and chemical changes induced by temperature (Moreira *et al*., 2008). The M_O is the minimum moisture content covering hydrophilic sites on the material surface, and it is a necessary data for achieving storage with minimum quality loss for long time. Furthermore, at this condition, the rates of spoilage reactions, except for oxidation of unsaturated fats, are minimal. Therefore, at a given temperature, the safest a_W level is that corresponding to M_O or lower (Moreira *et al*., 2008)

Table 3: Estimated parameters and fitting criteria of the models applied to experimental sorption data of dehydrated African walnut

Model names	Constants	Adsorption			Desorption		
		28°C	33°C	38°C	28°C	33°C	38°C
Smith	A	9.3806	9.4023	9.6824	15.1562	14.5851	11.8149
	B	-8.1272	-9.5799	-10.5799	-11.3994	-12.6404	-17.7076
	R^2	0.9992	0.8758	0.8758	0.7906	0.8929	0.9499
	P	0.0000	0.0211	1.164E-10	1.707E-15	0.0000	8.574E-10
	X^2	1.69E-17	1.34E-02	4.00E-19	8.08E-29	0.00E+00	1.25E-17
BET	C	-6.02E+00	-8.93E+00	-9.02E+00	-1.13E+01	-1.12E+01	-1.62E+01
	M_O	5.018	5.856	7.922	10.143	9.913	9.842
	R^2	0.9027	0.9392	0.9892	0.987	0.9771	0.9771
	P	-2.36E-08	0.0008222	0.0001921	0.0027622	1.63E-12	1.63E-12
	X^2	8.79E-14	1.90E-09	8E-05	3E-05	8.13E-14	8.13E-14
GAB	C	-3.79E-07	-2.38E-07	-2.38E-07	-8.35E-07	-1.12E-07	-1.12E-07
	K	0.8219	0.73913	0.73913	0.7688	0.1622	0.1622
	M_O	8.642	9.2241	14.8728	20.3271	17.9138	17.9138
	R^2	0.943	0.869	0.962	0.922	0.9731	0.9731
	P	-8.36E-10	3.59E-08	4.921E-08	1.67E-15	1.13E-15	1.13E-15
	X^2	1.53E-13	0.001	1E-05	8.01E-23	3.03E-23	3.03E-23
Ferro-Fontan	A	4.4426	5.3367	6.0169	11.0965	7.03742	4.1174

	Y	2.5528	2.7230	2.9101	4.8628	5.0022	7.9389
	R	0.216736	0.2451	0.2699	6.05E-02	9.58E-02	9.87E-02
	R^2	0.8375	0.8892	0.8892	0.9856	0.9697	0.9812
	P	3.36E-10	0.05979	1.192E-06	-0.003498	0.0098708	0.0330662
	X^2	1.17E-03	1.07E-01	2.81E-01	3.39E-04	1.83E-03	1.86E-02
Peleg	K_1	3.9892	6.9166	7.6596	8.1121	7.62	6.18
	K_2	10.84	11.89	13.901	12.3797	14.8789	16.1923
	n_1	1.8529	2.50449	2.82829	2.4099	2.2645	2.2917
	n_2	0.00079	0.00085	0.00091	0.00081	0.00099	0.0010
	R^2	0.9003	0.8540	0.8540	0.9895	0.9616	0.9715
	P	-8.36E-05	0.0214	0.0369261	0.0008715	0.0277175	0.0277175
	X^2	0.0000	0.0137	0.0000	0.0000	0.0144	0.0144

3.2 Modeling of Sorption Isotherms

Table 3 showed the result of the nonlinear regression analysis of adsorption and desorption isotherms of in-shell walnut obtained at 28°C, 33°C, and 38°C. The values of constants of the five models, that is, GAB, BET, Smith, Ferro-Fontan, Peleg, fitted to the desorption and adsorption data along with their standard error (P), the correlation coefficient (R^2), and the reduced chi-square (X^2) for the studied temperatures are given. Examination of the results revealed that some of the models are acceptable for predicting the EMC.

In considering the models with lowest standard error and the highest coefficient of correlation, the BET gave the best fitting for adsorption at 33°C and 38°C and Smith model was best fit at 28°C, while Peleg models gave the best fitting of desorption isotherms at 28°C, BET gave the best fitting at 33°C and Ferro Fontan gave the best fitting at 38°C. The R^2 of the models that fit the isotherms ranged from 0.903 to 0.999.

Lomauro *et al.*, (1985) pointed out that when percentage average relative deviation is less than 5, the fit is considered to be excellent. It is well-known that the fit becomes better as the coefficient of determination 'R^2' approaches 1. Also, the smaller the standard errors of models the better is the fit. Considering all these three criteria for the isotherms of the walnut at the three temperatures, the sorption data were in good agreement with GAB and other tested models. However, BET model seems to be excellent to represent the experimental sorption data. Comparison of the experimental and predicted isotherms for dehydrated walnut over the range of temperature and a_w commonly encountered in the tropics in food storage structures are shown in figures 3-5 for the adsorption isotherms and figures 6-8 for the desorption isotherms.

3.3 Moisture Sorption Hysteresis of Dehydrated African Walnuts

The experimental adsorption and desorption isotherms (change in EMC with a_w) obtained at 28, 33, and 38°C for African walnut are shown in Figures 9, 10, and 11. The EMC at each a_w represents the mean value of three replications. The temperature chosen is suggestive of average ambient temperature at which most food products are stored in tropics. The results indicated that the adsorption isotherms of the dehydrated walnut exhibit about type II curve. The adsorption isotherms at the temperatures lay below their desorption counterparts and both enclosed hysteresis loop. Hysteresis has been related to the nature and state of the components in a food, reflecting their potential for structural and conformational rearrangements (Yan *et al.* 2008). The sorption isotherms have the sigmoidal-shaped profile according to the BET classification. These curves are typical of plant products as reported by Kouhila et al., (2002), Ait-Mohammed *et al.*, (2004) and Idlimam *et al.*, (2008). Hysteresis between adsorption and desorption existed over almost the entire range of water activity at 28°C. Similar behaviour of adsorption and desorption isotherms was observed for 33°C and 38°C. The hysteresis effect was more pronounced at lower temperatures in which water content on the desorption isotherm is higher than that on the adsorption side at the same water activity, this is one of the reasons for difference in moisture content between the two closures.

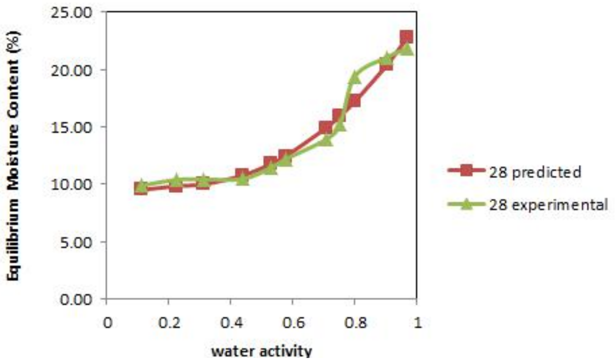

Figure 3: Comparison of experimental and GAB predictive adsorption isotherms of dehydrated African walnut at 28°C

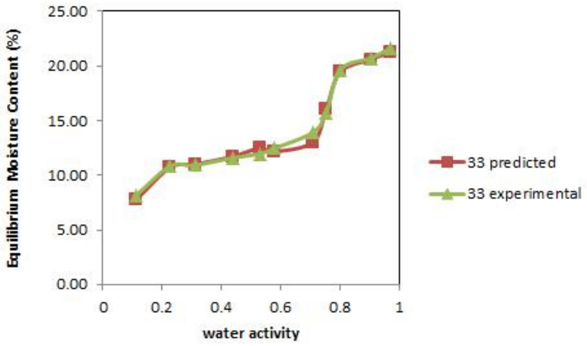

Figure 4: Comparison of experimental and GAB predictive adsorption isotherms of dehydrated African walnut at 33°C

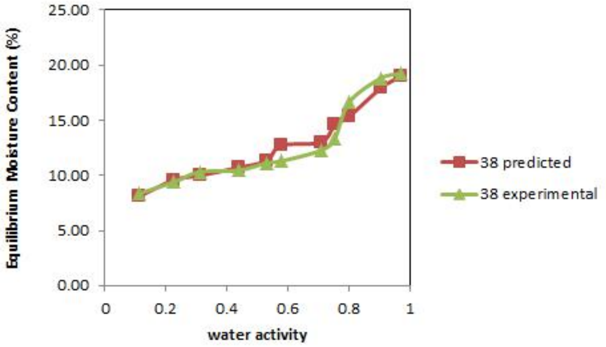

Figure 5: Comparison of experimental and GAB predictive adsorption isotherms of dehydrated African walnut at 38°C

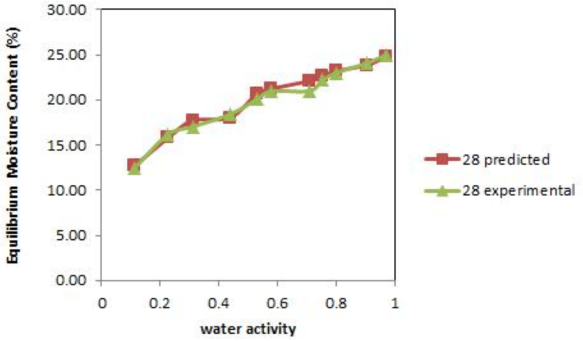

Figure 6: Comparison of experimental and GAB predictive desorption isotherms of dehydrated African walnut at 28°C

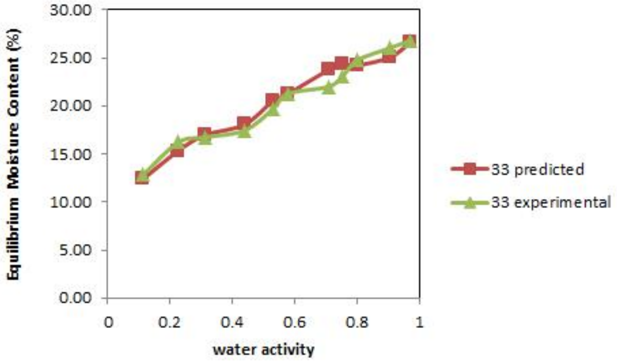

Figure 7: Comparison of experimental and GAB predictive desorption isotherms of dehydrated African walnut at 33°C

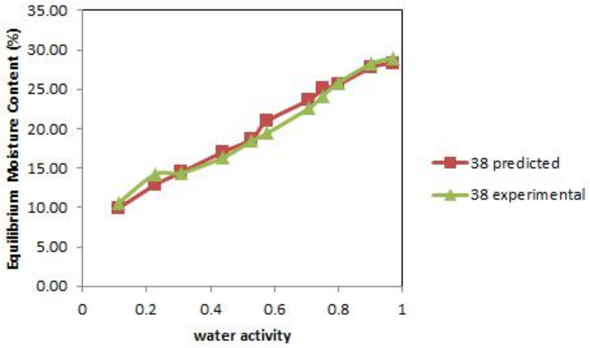

Figure 8: Comparison of experimental and GAB predictive desorption isotherms of dehydrated African walnut at 38°C

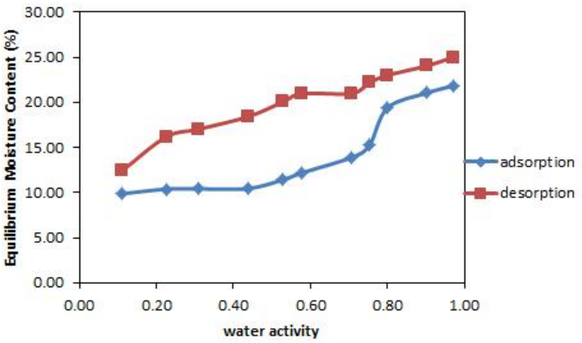

Figure 9: Adsorption and desorption isotherm of dehydrated African walnut at 28°C

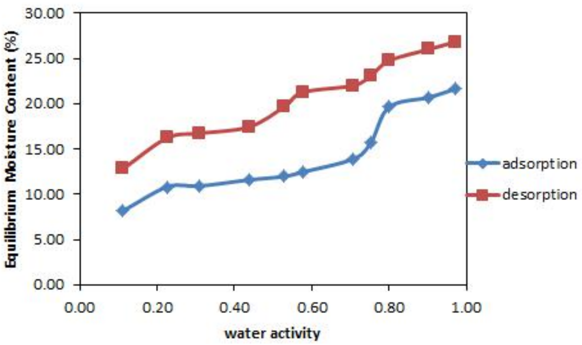

Figure 10: Adsorption and desorption isotherm of dehydrated African walnut at 33°C

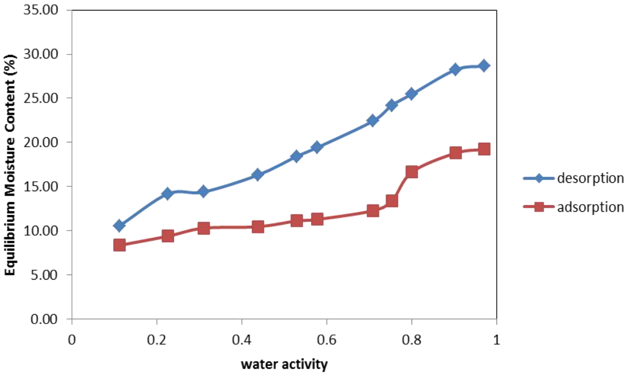

Figure 11: Adsorption and desorption isotherm of dehydrated African walnut at 38°C

points is that, during drying (desorption), some solutes may supersaturate below their crystallization water activity and thus hold more water as a_w is lowered (Barbosa-Carnovas *et al.,* 2007; Saad *et al.*, 2014). . The swelling of polymeric materials during moisture adsorption can also lead to hysteresis (Raji and Adeniran, 2011). Iglesias and Chirife (1976) recognized that it is not possible to give a single explanation of the hysteresis phenomena in foods due to food being a complex biological material.

The figures revealed that the equilibrium moisture content increased with water activity at constant temperature. This may be due to the fact that vapor pressure of water present in foods increases with that of surroundings (Shivhare *et al.*, 2004). In the first segment (with low water activity) of the S-shaped sorption isotherm curves, walnut kernels sorbed relatively lower amounts of moisture. However, larger amount of moisture was absorbed at higher a_w. Similar behaviour has been reported by other authors for different foods (Sanni *et al.*, 1997; Lee and Lee, 2008). Two bending regions are noted, one around 0.1 to 0.3 and another at 0.5 to 0.6. The isotherm is therefore divided into three zones. According to Aguilera and Stanley (1999), in zone I (a_w between 0.05 and 0.2) minimal water is contained in the product, and the water molecules present are tightly bound to active sites (e.g., polar groups in molecules) mainly by hydrogen bonding. In zone II (a_w between 0.2 and 0.5) the water is more loosely bound, initially as multilayer above the monolayer; later, as moisture content increases, this water successively fills micro-pores and macro-pores in the system. In this region, chemical and biochemical reactions requiring solvent water start to take place because of the increased mobility of solutes. In zone III (a_w between 0.6 and 0.9), excess water is present in macro-capillaries, exhibiting nearly all the properties of bulk water. Microbial growth becomes a major deteriorative reaction in this region (Saad *et al.,* 2014). According to Vanden Berg and Bruin (1981), a general sigmoid sorption isotherm can be divided into three different parts; ranges I (a_w 0–0.22), II (a_w 0.22–0.73) and III (a_w 0.73–1.0). In ranges II and III, water molecules penetrate newly created pores of the already swollen structure and are mechanically entrapped in the void spaces. Therefore, water uptake particularly at higher a_w would be markedly influenced by the stability of the micro-porous structure.

4.0 Conclusions

Moisture sorption isotherms were determined for dehydrated African walnut at three different temperatures of 28, 33 and 38°C. There was a significant effect of temperature on the equilibrium moisture content (EMC) in the range of temperatures studied. The EMCs were found to decrease with increasing temperature at constant a_w; they were also found to increase with increasing a_w at constant temperature. The isotherms obtained are sigmoidal in shape and showed evident effect of hysteresis. In general, BET model provided a better fit to the experimental data than other tested models, thus it was found to be the most appropriate equation for representing the sorption isotherms. M_o increased with temperature for adsorption, while it decreased with increasing temperature for desorption.

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