

Moisture sorption isotherms of dehydrated (African oil bean) *Pentaclethra macrophylla* Benth. cotyledons

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Abstract: Sorption isotherms of (African oil bean) *Pentaclethra macrophylla* benth. cotyledons were determined by static gravimetric method at different temperatures, in the range from 28°C to 48°C. Equilibrium moisture content (EMC) data were analysed using various sorption models usually applied to dried food materials. Experimental data were analysed by a thermodynamic approach to obtain such properties as net isosteric heat, net equilibrium heat, differential and integral entropy that provide a deeper understanding of the properties of water and energy requirements associated with sorption process. Sorption isotherms of dried African oil bean cotyledons showed type II isotherm. Adsorption and desorption isotherms were best described by Guggenheim–Anderson–DeBoer ($R^2 = 0.98$). The monolayer moisture content (M_o) decreased as drying temperature increased and the value ranged from 0.0877 to 0.9966 g H₂O 100 g⁻¹ DM.

Keywords: African oil bean, sorption isotherms, sorption models, equilibrium moisture content, hysteresis

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1 Introduction

The African oil bean (*Pentaclethra macrophylla* Benth) is of large leguminous woody plant, it belongs to the family *Fabaceae* and sub-family *Mimosoidae* (Keay, 1989; Orwa et al., 2009). It usually has about eight flat glossy brown edible seeds per pod (Enujiugha et al., 2003). The raw seed is a potential source of edible protein and calories, containing the twenty essential amino acids and essential fatty acids that make up more than 80 percent of the fatty acids in the oil (Enujiugha and Agbede, 2000). Available literatures indicate variations in cooking time, fermentation

duration as well as in slice sizes (Enujiugha, 2000; Mbajunwa, 1993; Aju and Okwulehie, 2005). The shelf life of boiled and fermented seeds had also been reported to be very short leading to putrefaction within few days after processing (Mbajunwa, 1993). In other to increase the shelf life of African oil bean seeds, different preservation techniques are being employed including the manipulation of storage temperature, fermentation, roasting, addition of preservatives and canning (Enujiugha, 2000).

Dried food products are prone to adsorption of moisture during storage, and this could affect their shelf-life and packaging requirements. It is quite germane to study the moisture sorption isotherms of dehydrated products in order to provide the required information. Moisture sorption properties of foods have been shown to be influenced by food composition, processing, treatment, temperature and relative humidity (Alhamdan and Hassan, 1999).

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Knowledge of the water adsorption characteristics is needed for shelf life predictions, and determinations of critical moisture and water activity for acceptability of products that deteriorate mainly by moisture gain, and are important in drying, packaging and storage characteristics (Al-Mahasneh and Rababah, 2007; Iguedjal et al., 2008; Moreira et al., 2010). Lipid or protein food products might develop off-odour as result of either lipid oxidation of fat-containing foods or protein degradation (Wasowicz et al., 2004; Chotimarkorn and Silalai, 2008). However, high moisture content and water activity are the main factors that are responsible for quality loss of most foods (Lee and Lee, 2008). The relationship between water content in food equilibrium with water activity or equilibrium relative humidity at constant temperature and pressure are valuable for predicting rate of deterioration of dry food materials (Kaymak-Ertekin and Sultanoglu, 2004; Oyelade et al., 2008). A number of models has been suggested to describe the relationship between the equilibrium moisture content and relative humidity (Moreira et al., 2010; Lee and Lee, 2008). These models could be classified as theoretical and semi-empirical models. The effectiveness of any model depends on the physiochemical characteristics of the food materials. The Guggenheim-Anderson-DeBoer (GAB) equation is widely used for most food materials (Torğrul and Arslan, 2007; Peng et al., 2007). The Henderson and Chung and Pfost equations were suitable for fibrous and starchy materials while the modified Halsey fitted well for high oil and protein materials (Corzo and Fuentes, 2004; Chowdhury et al., 2006). Modelling of sorption isotherms confer benefits on foods including convenience for design drying process, shelf life prediction and packaging material selection (Doporto et al., 2012). Moreover, moisture sorption isotherm affects physical, sensory, colour, rehydration and water holding capacity (WHC), chemical and microbiological attributes of dried products (Lee and Lee, 2008). In addition, the rate of deterioration does not only depend on moisture content of the dried food, but oxidation of its components (Shrestha et al., 2007). Lipid oxidation can be initiated by moisture migration and the

amount during storage. Thus, moisture adsorption can also be used as index of quality and shelf life determinations because of the impact of lipid oxidation on flavour, taste and decreasing consumers' acceptability (Lin et al., 1998; Lu et al., 2011; Demirkesen et al., 2010).

The objective of this study was to determine the moisture sorption isotherms characteristics of dried African oil bean cotyledons over a range of temperatures and humidities commonly experienced in the tropical environment and the correlate experimental sorption data with well-known sorption isotherms models and state the best predictive model for dried African oil bean cotyledons.

2 Material and methods

2.1 Material

African oil bean seeds (*Pentaclethra macrophylla* Benth) were purchased from a Local Conservation Officer at Ilaro (6.8954° N, 3.0126° E), South Western Nigeria. Two kilograms of the seeds were dehulled by parboiling the seeds for 30 minutes at 103.42 kPa using a pressure cooker (Masterchef, YB 208, Japan) to aid removal of the seed coats. The dehulled seed (cotyledon) were boiled in pressure cooker at 103.42 kPa for 6 hour (Enujiugha, 2003). The cotyledons were washed in six changes of fresh water for 2 hour (Enujiugha, 2003). The heater of the drier (Uniscope SM9080, England) was switched on and the drier was allowed to run for 30 minutes in order to allow drying air to attain the desired temperature (60°C). Boiled cotyledons were weighed and were dried at 60°C till equilibrium weight was achieved. The initial moisture content was determined using method of AOAC (2000).

2.2 Determination of sorption isotherms

For adsorption isotherm, single cotyledon of the dried African oil bean were weighed and dried to 4% MC_{db} (Moreira et al., 2010). For desorption isotherm, twenty single cotyledon of the dried African oil bean were placed in a desiccator having relative humidity of 97%, thereby making the dried sample to pick up moisture at 25°C for 4-5 days (Moreira et al., 2010). The initial moisture content was determined using method of AOAC (2000). Water

sorption isotherms were determined by exposing samples to saturated salt solution of known relative humidity. Eleven saturated salt solutions were prepared by mixing salt and distilled water in closed containers and stirring them once a day for 7 days. Salts used are: LiCl; CH₃COOK; MgCl₂.6H₂O; K₂CO₃; Mg(NO₃)₂.6H₂O; NaBr; SrCl₂.6H₂O; NaCl; (NH₄)₂SO₄; BaCl₂.2H₂O; K₂SO₄. Water activities of slushes (saturated salt solutions) as described by Kaya and Kahyaoglu (2007) at 30°C are shown Table 1.

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 ₃ COOK	126	79	0.226
3.	MgCl ₂ .6H ₂ O	300	100	0.327
4.	K ₂ CO ₃	300	135	0.431
5.	Mg(NO ₃) ₂ .6H ₂ O	225	34	0.528
6.	NaBr	300	120	0.577
7.	SrCl ₂ .6H ₂ O	300	75	0.708
8.	NaCl	300	90	0.752
9.	(NH ₄) ₂ SO ₄	300	120	0.800
10.	BaCl ₂ .2H ₂ O	375	105	0.903
11.	K ₂ SO ₄	20	100	0.970

Moisture adsorption and desorption isotherms were determined by static gravimetric method at different temperatures (28°C, 38°C and 48°C). Sample dishes were placed in desiccators which contained saturated solutions of known constant relative humidity. Samples were weighed twice a day until weight change less than ± 0.001 g. On attainment of equilibrium weight, the equilibrium moisture content for all samples was determined using method of AOAC (2000).

2.3 Modelling sorption isotherm characteristics

In order to predict moisture sorption behavior of samples, the BET (Brunauer, Emmett & Teller), GAB, Smith, Ferro-Fonta, Peleg and Oswin models (Table 2) were used to fit experimental curves (Kaya and Kahyaoglu, 2007). The parameters of the model used were estimated by a non-linear regression produce of Datafit software version 8.2 by Oakdale Engineering. The criterion used to evaluate goodness of fit of the experimental sorption data was defined by coefficient determination (R^2), the mean relative

percent error (P) and the root mean square error ($RMSE$) (Kaya and Kahyaoglu, 2007):

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

$$P = \frac{100}{N} \sum_{i=1}^N \frac{M_{exp,i} - M_{pre,i}}{M_{exp,i}} \quad (2)$$

$$RMSE = \left[\frac{1}{n} \sum_{i=1}^n (M_{exp,i} - M_{pre,i})^2 \right]^{\frac{1}{2}} \quad (3)$$

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.

Table 2 Mathematical models used to describe moisture adsorption and desorption isotherm behaviors

No.	Name	EMC Model	Reference
1	Oswin	$M_e = A \left(\frac{a_w}{(1-a_w)} \right)^C$	(Kaymak-Ertekin and Gedik, 2004)
2	BET	$M_e = \frac{CM_o a_w}{(1-a_w)(1+(C-1)a_w)}$ $M_e = k a_w^n + k_2 a_w^{n_2}$	(Barbosa-Cánovas and Vega-Mercado, 1996)
3	Peleg	$M_e = \frac{CkM_o a_w}{(1-ka_w)(1-ka_w + cka_w)}$	(Medeiros <i>et al.</i> , 2005)
4	GAB	$M_e = A - B \ln(1-a_w)$	(Barbosa-Cánovas and Vega-Mercado, 1996)
5	Smith	$M_e = \left\{ 1 / \left[\frac{1}{\alpha} \ln \left(\frac{\gamma}{a_w} \right) \right]^{\frac{1}{r}} \right\}$	(Barbosa-Cánovas and Vega-Mercado, 1996)
6	Ferro-Fontan		(Barbosa-Cánovas and Vega-Mercado, 1996)
			(Durakova and Menkov, 2005)

Where M_e = Equilibrium moisture content; M_o = Monolayer moisture content; a_w = Water activity; A , B , C , γ , α , n and k , are model constant.

3 Result and discussions

3.1 Moisture sorption isotherms of dried African oil bean cotyledons

The experimental adsorption and desorption isotherms of African oil bean cotyledons at 28°C, 38°C and 48°C were presented in Figures 1-3. The adsorption and desorption isotherm curves showed a typical sigmoid shape confirming type II classification which is characteristic nature of biological material, which sorbs relatively small quantity of moisture at lower water activities and large amount at high relative humidity (Aviara et al., 2004). The equilibrium moisture content (EMC) increased with increasing water activity (a_w) or relative humidity at constant storage temperature. The difference in EMC between adsorption and desorption isotherms gave hysteretic phenomenon. The hysteresis might be attributed to the effect of irreversible physico-chemical changes that occurred during drying of the African oil bean (Arslan and Toğrul, 2005). Low hysteresis magnitude, found in sorption experiment conducted at 48°C might be due to high energy level of absorbed water molecules. Therefore, temperatures influence movement of water molecules to the surface of the food or sorption sites faster than low storage temperature resulting in least hysteresis magnitude. However, moisture migration rate also depends on the degree of adsorbent porosity and moisture gradient between surrounding air and adsorbent interface (Guillard et al., 2004).

Figure 4 shows the effect of storage temperature on the moisture sorption of African oil bean cotyledon. There was slight increase in equilibrium moisture content of the oil bean cotyledons as water activity increases signifying minimal effects on the storage stability of the dried African oil bean. This is agreement with similar findings reported by Arslan and Toğrul (2005) which stored walnut kernels at 25°C, 35°C and 45°C. The higher the temperature, the lower the equilibrium moisture at constant water activity (a_w). This is explained by the higher excitation state of water molecules at higher temperature decreasing the attractive

forces between molecules of water and the components of the oil bean or thermal agitation (Kapseu et al., 2006). The lower equilibrium moisture content at higher temperature has also been explained to be due to the activation of water molecules which causes them to break away from the water binding sites, thus lowering the equilibrium moisture content (Oluwamukomi, 2008; Tarigan et al., 2006; Aviara et al., 2006). Dried African oil bean became less hygroscopic at higher temperature. This will enhance the storage life of the dehydrated African oil bean cotyledons because with increasing storage temperature, the sample adsorbed less water at constant relative humidity. Similar observation have been reported in chestnut (Moreira et al., 2010). The increase in temperature coupled with increase in water activity has been linked to factors responsible for quality loss in foods linked, oxidative rancidity, non-enzymatic and enzymatic browning (Barbosa-Cánovas and Vega-Mercado, 1996). A number of studies have considered the effect of environmental conditions and water activity on the lipid degradation (Sun et al., 2001; Akanbi et al., 2006; Cunningham et al., 2007).

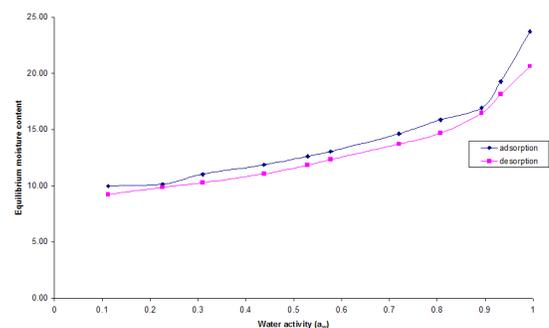


Figure 1 Adsorption and desorption isotherms of dried African oil bean cotyledon at 28°C

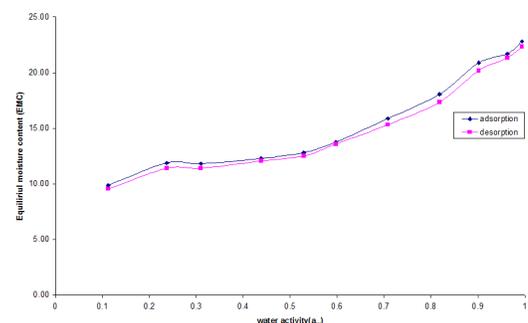


Figure 2 Adsorption and desorption isotherms of dried African oil bean cotyledon at 38°C

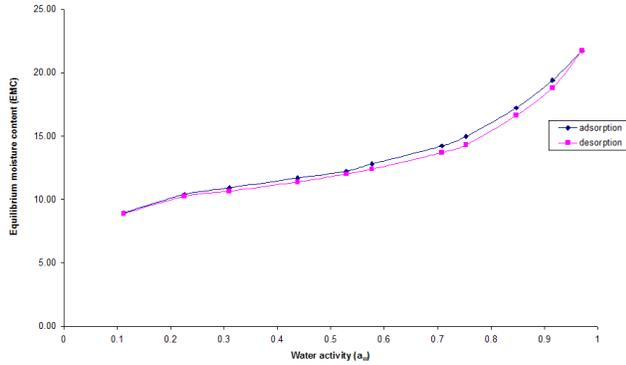


Figure 3 Adsorption and desorption isotherms of dried African oil bean cotyledon at 38°C

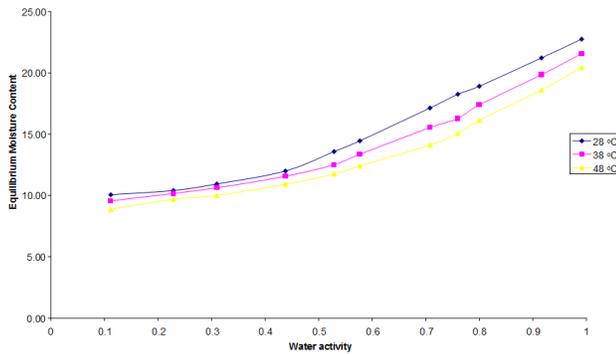


Figure 4 Adsorption isotherms for African oil bean stored at different temperature

3.2 Mathematical modelling for sorption isotherm of the African oil bean cotyledons

The experimental sorption equilibrium moisture content data of African oil bean were fitted into 10 models. Six models out of the models were selected based on the criteria: coefficient of determination (R^2), the mean relative per cent error (P), and the root mean square error ($RMSE$). Better goodness of fit was indicated by higher R^2 and lower P and $RMSE$ values. The Smith, GAB, Ferrotan-Fontan, BET, Peleg and Oswin models gave acceptable fit values over the range of temperature studied (i.e. 28°C - 48°C). Monolayer moisture content (M_o) ($\text{g H}_2\text{O } 100 \text{ g}^{-1} \text{ DM}$) obtained using GAB and BET models is given in Table 3. The monolayer moisture content of the samples decreased with increasing storage temperature for adsorption, however, M_o increased with increasing storage temperature for desorption. The M_o values are important for the determination of the storage conditions and controlling the possible deterioration reactions. It is the amount of water

needed to form a continuous, adsorbed monolayer over the surface of dried foodstuff (Lomanro et al., 2020). The M_o values ranged between 0.0877 and 0.0996 $\text{g H}_2\text{O } 100 \text{ g}^{-1} \text{ dry matter}$ for GAB and 0.0931 and 0.1056 $\text{g H}_2\text{O } 100 \text{ g}^{-1} \text{ dry matter}$ for BET and were found to be comparable to those found in the literature (Moreira et al., 2010; Vázquez et al., 2001; Ayranci and Duman, 2005). The C (7.22 – 6.7326) parameter and K (2.00 – 2.41) parameter increased with increasing temperature. The reduction in the values of C and K parameters of GAB model as temperature increases revealed that the binding energies associated with the mono and multilayer sorption of water to the African oil bean decreased with temperature increase. The result is in agreement with the findings reported by Resio et al. (1999), Akanbi et al. (2006) and Moreira et al. (2010).

Table 3 Estimated parameters and fitting criteria of the models applied to experimental sorption data of African oil bean cotyledon

Models	Constant	Adsorption			Desorption		
		28°C	38°C	48°C	28°C	38°C	48°C
Smith	A	10.0512	9.6706	9.3600	10.3477	9.5882	9.3806
	B	-9.9552	-9.3780	-8.148	-9.3766	-9.5420	-9.8127
	R^2	0.8910	0.8810	0.9010	0.8754	0.9618	0.9999
	P	5.45E-	1.14E-	4.88E-	0.0006	0.0002	-6.71E-
	$RMSE$	10	09	10	0.3255	0.0521	10
		0.0264	0.0300	0.0254			0.0269
GAB	C				7.220	6.9730	
	K	6.4800	6.2700	6.1700	2.000	2.2100	6.7326
	M_o	1.0200	1.2400	1.9000	0.0910	0.0967	2.4100
	R^2	0.0877	0.0831	0.0816	0.9680	0.9860	0.0996
	P	0.9830	0.9621	0.9520	0.0125	0.0080	0.9987
	$RMSE$	0.0008	0.0092	0.0095	0.0576	0.0754	0.0006
Ferro-Fontan	α				3.0875	2.8765	
	γ	2.7964	2.8840	3.4032	4.6955	6.3829	2.3590
	R^2	6.0805	3.7686	3.4033	0.9320	0.9195	8.1510
	P	0.8560	0.8690	0.8630	0.0445	0.0658	0.7980
	$RMSE$	0.0776	0.0656	0.0580	0.7257	0.7718	0.0627
		0.8019	0.7713	0.7450			0.7558
BET	C				30.451	26.4012	
	M_o	28.510	25.400	21.200	0.0994	0.0996	18.1224
	R^2	0.0943	0.0971	0.0931	0.9513	0.9723	0.1056
	P	0.9583	0.9643	0.9644	0.0371	0.0329	0.9027
	$RMSE$	0.0688	0.0382	0.0380	0.0022	0.0016	0.2689
	k_1	0.0049	0.0031	0.0029	28.2220	30.5954	0.0064
Peleg	k_2	14.499	12.512	11.082	25.0054	27.4398	32.4391
	n_1	10.543	11.158	10.579	8.3328	8.2917	30.2381
	n_2	2.8360	3.0000	3.2650	0.8446	0.9275	8.1229
	R^2	0.0600	0.0920	0.0830	0.9046	0.8869	0.9643
	P	0.9570	0.9490	0.9630	0.0213	0.0241	0.9012
	$RMSE$	0.0006	-0.001	0.0006	0.6340	0.6432	-0.0981

		0.3255	0.3692	0.3255			0.8120
	<i>N</i>				13.64	16.64	
Oswin	<i>K</i>	13.500	12.943	12.209	0.1630	0.1830	18.76
	<i>R</i> ²	0.1720	0.1700	0.1690	0.9046	0.8869	0.2460
	<i>P</i>	0.9570	0.9490	0.9630	0.0213	0.0242	0.9072
	<i>RMSE</i>	0.0006	-0.0011	0.0006	0.6340	0.6432	0.0181
		0.3255	0.3692	0.3255			0.6200

Monolayer moisture content estimated using GAB model was similar to that of BET model. The similar monolayer moisture contents could be due to the low initial moisture content of the dried African oil bean cotyledon. Based on the values of *R*² (0.9830), *P* (0.0008), *RMSE* (0.0061) values, the GAB model showed the best correlations for the experimental sorption data for African oil bean cotyledon throughout the range of water activity (0 – 0.970) studied.

4 Conclusion

The moisture sorption isotherms of dried African oil bean cotyledons are of type II as classified by BET. The effect of temperature on moisture sorption isotherm was identified. The equilibrium moisture content decreases with increasing temperature at the same water activity following the trend of most of the food materials on storage. GAB model was the best suitable model for describing the moisture sorption isotherm behaviour of dried African oil bean cotyledons. Monolayer moisture content of the oil bean cotyledons was temperature independence.

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