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Isotherm Models of Tacca Starch (*Tacca Involucrata*) at Ambient Temperature Using Some Common Packaging Materials

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A B S T R A C T

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Looking into the relationship between the air relative humidity (water activity a_w) and equilibrium moisture content of food materials is important in maintain good keeping quality and process operations. The adsorption isotherms for tacca starch using different packaging materials (low density polyethylene (LDP), high density polyethylene (HDP) and plastic) were investigated. Concentrated acid (H_2SO_4) solutions ranging from 69.50-164.98 ml were used to vary the micro-climate in the study at ambient temperature and a_w (0.10-0.80) which is usually experienced in the tropical environment. The experimental data were compared with seven widely recommended models in the literature for food adsorption isotherms (GAB, Oswin, Modified Oswin, BET Hasley, Smith and Henderson). The moisture adsorption isotherms were sigmoidal in shape and was influenced by temperature. The modified Oswin model was found to be most adequate for HDP packaging material while Oswin model was found suitable for plastic and LDP packaging materials.

1. Introduction

Mostly natural food contains water in abundance, which the water has many roles in food processing such as solvent, mobilize, reactant reaction medium etc. and, while the chemistry has impact on food quality and food reaction. Otherwise water in food is believed to be free and bound water (Al-Muhtaseb et al., 2002) hence, water is an important factor for deterioration of food and affect its shelf-life also.

Controlling the food moisture content during processing and storage is very important since water plays a critical role in food reactions and food quality. Knowledge of sorption isotherm has a great importance in food dehydration (Budi and Jenshinn, 2010). The connection between the equilibrium moisture content in a biological materials and the relative humidity of ambient air at constant temperature is called a sorption isotherm. It is well known that most hygroscopic biological materials exhibit hysteresis in the adsorption and desorption isotherms.

According to Ricardo, et al., (2011) the characteristics of sorption isotherm in food can be classified to five different types including Type 1: Langmuir or similar isotherm, Type 2: sigmoidal sorption isotherm, Type 3: Flory-Huggins isotherm, Type 4: .

described the adsorption of a swellable hydrophilic solid until a maximum of site hydration is reached, and the last type is Brunauer-Emmett-Teller (BET). Normally sorption characteristic on the type 2 and 4 was shown in majority of food products. In the food dehydration, the sorption properties are very pivotal to shelf-life of food materials which are useful for quantitative approach prediction. From the previous researches, the study of sorption isotherm in food products with the use of mathematic model to prediction with the use of two or three fitting parameters to describe moisture sorption isotherms (Al-Muhtaseb et al., 2002). Hence, different food products has their specific models that are best for them. So selection of the model should be suitable for type of food products that effect to high efficiently. The common equations that are used for describing sorption models in food products including the Langmuir equation, BET equation, Oswin model, Smith model, Halsey model, Henderson model, Iglesias-Chirife equation, GAB model, and Peleg model (Ricardo et al., 2011).

Starch is one of the most abundant natural biological materials in nature. It is cheap, available, renewable, and biodegradable polymer produced by many plants as a source of stored energy. It can be found in different parts of the plants which includes the leaves, stem, roots, bulbs, nuts, stalks. Starch can be found in different plants depending on the plant origin but

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particularly tuber plants which includes from cassava, potato, yam and tacca among others. It is a renewable and almost unlimited resource material. Apart from tuber plants, it can be found in rice, wheat, corn e.t.c. It has found wide use in the food, textiles, cosmetics, plastics, adhesives, paper, and pharmaceutical industries. In the food industry, starch has a wide range of applications ranging from being a thickener, gelling agent, to being a stabilizer for making snacks, meat products, fruit juices (Manek, et al., 2005).

The numerous industrial usage of starch is characterised on its availability at low cost, high caloric value, inherent excellence physicochemical properties and the ease of its modification to other derivatives. The relevance of starch in the industry is spawn by its starch morphology and physicochemical characteristics which are typical of its biological origin (Gebre-Mariam and PC Schmidt, 2006). In 2000, the world starch market was estimated to be 48.5 million tons, including native and modified starches. The value of the output is worth €15 billion per year (Le Corre et al., 2010). The current sources of commercial starch are corn (maize), wheat, potato, cassava and rice and the global share of corn starch accounts for more than 80 %, whereas cassava starch is only 7.5 % (Patil, 2012).

If Africa must compete in the global starch production, newer sources must be exploited among the carbohydrate crops that are not fully utilized as staple food materials, one of such plants is the generic plant *Tacca*. The common names are Polynesian arrowroot and Bat flower. They are native to tropical regions of Africa, Australia, and South-eastern Asia, and have been domesticated in the Pacific island nations.

The plant *Tacca involucrata* is a wild plant that contains starch which is eaten when the flour is being cooked with almost 0% fat, usually by the villagers or rural dwellers in the Northern Nigeria as their food. It belongs to the family Taccaceae. It is a perennial herb with a round swollen tuber underneath the ground from which shoots out a leaf stalk up to 1 m long. The plant had a long history of being a major source of carbohydrate in the savannah belt of Nigeria until the advent of cassava when its importance waned. It is now harvested wild. The tuber is first boiled to remove the toxic element and then converted to flour and employed in a variety of food preparations. It is an under-utilized crop with a high potential as a source of industrial starch (Omojola, 2013). The starch has to be dried to lower moisture content and processed into flour for easy and long-term storage (Budi and Jenshinn, 2010).

Therefore, moisture sorption isotherm information is required for drying and storage. The objectives of this work are (a) to obtain data on adsorption isotherm of tacca starch flour at ambient temperature and (b) to compare fittings of seven sorption isotherm models and choose the best model.

2.0 Materials and Methods

2.1 Materials

The tacca tubers were gotten from Along hill 2, OAU, Ile-Ife, Osun State Nigeria, Olokemeji Road, Eruwa, Oyo State Nigeria. The packaging materials were gotten from a local market in Akure, Ondo State Nigeria. The dessicators, digital weighing balance (AND EK-410i, A&D CO., LTD, JAPAN), Hot-air Oven and Incubator (Model IR701SG, Lec Refrigerator Plc, Britain) were gotten from Food Processing Laboratory, Food Science and Technology Department, Federal University of Technology Akure, Ondo State. Analytical grade of concentrated Sulphric acid and sodium metabisulphite solution were gotten from PASCAL Limited.

2.2 Methods

2.2.1 Preparation of tacca starch

The tacca starch was obtained according to the method described by Ukpabi et al., 2009. Fresh roots of *Tacca involucrata* were peeled and washed thoroughly with water to remove dirt. The washed tubers were soaked in sodium metabisulphite solution (2 L 1% w/v) at room temperature (27 °C) overnight. Thereafter, the tubers were removed and wet milled into a slurry using a crusher. The paste was dispersed in a large volume of 1 % sodium metabisulphite solution and filtered through muslin cloth. The suspension was centrifuged at 3500 rpm for 10 minutes to facilitate the removal of dirt. The supernatant was carefully decanted and the mucilage scraped off. The process was repeated for three times with the mucilage on the starch scraped continuously until a pure starch was obtained. The resulting starch was dried in the sun and further dried at 60 °C in a hot air oven, pulverized, weighed and stored in sample bottles for analysis. The starch obtained was a brilliant white, crystalline, non-hygroscopic powder.

2.2.2 Moisture content determination

The AOAC (1990) procedures were followed to determine the moisture content for tacca starch samples. Determination was in triplicates.

2.2.3 Humidity control

The static gravimetric method was used to bring about the required equilibration with concentrated sulphuric acid (H₂SO₄) to create storage atmosphere according to the method described by Oyelade, 2008. Various quantities of H₂SO₄ used to make up a 250 ml of desiccant with deionised water was prepared at 30 °C to obtain water activities (aw) of 0.1, 0.20, 0.40, 0.60 and 0.80 respectively.

2.2.4 Determination of adsorption isotherms

The static gravimetric method was used. In triplicate, 20 g of tacca starch sample was weighed into different packaging materials (Ajibola, 1986). The prepared desiccant corresponding to each aw was dispensed into the desiccators used for the study. In this way the desiccators were maintained at water activity (aw) values of 0.1, 0.20, 0.40, 0.60 and 0.80 respectively. The desiccators were placed in Gallenkamp Incubator (Model IR701SG, Lec Refrigerator Plc, Britian) to maintain specific required temperature. Samples were monitored in the incubator for equilibration by weighing at intervals until constant weights were attained. Moisture content of the equilibrated samples were then found by calculation from the original moisture content and the known change in weight in dry basis form (Igbeka et al, 1975; Oyelade et al, 2001; Oyelade, 2008).

2.3 Isotherm equations and modelling

Seven widely recommended isotherm equations (GAB, BET, Oswin, modified Oswin (MOE), Henderson (HDE), Smith and Hasley that were investigated with the experimental data are

shown in Table 1. The excel package was used for the modelling of the equilibrium moisture content data. In order to determine the goodness of fit of models, it is evaluated using three different parameters namely residual sum of squares (RSS), standard error of estimate (SEE) and coefficient of determination (R2) (Oyelade, 2008). These indicators of model fit are defined in equations (8) to (10).

$$RSS = \sum_{i=1}^n (M_{cal} - M_{pred})^2 \tag{8}$$

$$R^2 = 1 - \frac{\sum_{i=1}^n (M_{cal} - M_{pred})^2}{\sum_{i=1}^n (M_{pred} - M_{cal})^2} \tag{9}$$

$$SEE = \sqrt{\frac{\sum_{i=1}^n (M_{cal} - M_{pred})^2}{df}} \tag{10}$$

where, M_{cal} = experimental equilibrium moisture content,
 M_{pred} = predicted equilibrium moisture content,
 n = number of experimental unit
 (degree of freedom) = n - 1,
 RSS = residual sum of squares
 SEE = standard error of estimate.

Table 1: Humidity Control of concentrated sulphuric acid (H₂SO₄) acid at different Temperatures.

Relative humidity (%)	Quantity of H ₂ SO ₄ at 20 °C (ml)	Quantity of H ₂ SO ₄ at 30 °C (ml)	Quantities of H ₂ SO ₄ at 40 °C (ml)
10	164.43	164.98	165.50
20	146.17	146.73	147.07
40	120.89	121.37	121.28
60	97.74	98.01	97.03
80	69.89	69.50	66.19

Table 2: Isotherm Models for Fitting Experimental Data

MODELS	EQUATIONS	REFERENCES
GAB	$X_{eq} = \frac{M_0 CK a_w}{(1 - K a_w)(1 - K a_w + CK a_w)}$ (1)	Andarade <i>et al.</i> , 2011
BET	$X_{eq} = \frac{CM_0 a_w}{(1 - a_w)(1 + (C - 1)a_w)}$ (2)	Andarade <i>et al.</i> , 2011
Oswin	$X_{eq} = A \left(\frac{a_w}{1 - a_w}\right)^B$ (3)	Vega-Galvez <i>et al.</i> , 2008
Modified Oswin	$X_{eq} = (a + bT) \left(\frac{a_w}{1 - a_w}\right)^c$ (4)	Vega-Galvez <i>et al.</i> , 2008
Smith	$X_{eq} = C + n \ln(1 - a_w)$ (5)	Andarade <i>et al.</i> , 2011
Halsey	$X_{eq} = \left(-\frac{C}{\ln a_w}\right)^{1/n}$ (6)	Andarade <i>et al.</i> , 2011
Henderson	$X_{eq} = \left(-\frac{\ln(1 - a_w)}{C}\right)^{1/n}$ (7)	Andarade <i>et al.</i> , 2011

Where, M_0 = monolayer moisture content; a, b, c, n and k = unknown model parameters to be estimated; T = temperature, °C

3.0 Results and Discussion

3.1 Moisture sorption isotherms

The adsorption isotherm is shown in figure 1 which is determined by plotting the equilibrium moisture content against different water activities. The isotherm followed the sigmoid shape of type II classification by Brunamer et al. (1938) as reported by Budi and Jenshinn, 2010; Famurewa et al., 2012 and in which the curves are concave upwards; it takes into account the existence of multilayers at the internal surface of a material reported by Oyelade, 2008. This is typical of isotherms of products high in starch content as observed by Kumar (2000) and Oyelade, 2008.

The shape of the equilibrium isotherm can be divided into three parts depending on the type of fixation. At low RH the water molecules are bound in one layer to the surface of the pores by hydrogen bond or van der Waal forces. When all surfaces of the pores are covered with one layer of molecules, the building-up of the next layer starts. This is shown by a straight line in early phase of the curve. The thickness of the adsorbed water layer increases to a third or possibly a fourth layer with an increasing pore humidity. Capillary condensation is the last mechanism that takes place. This means that at a given relative humidity, all the pores with a certain radius will be filled with water. The equilibrium water content in the material is dependent not only on the relative humidity of the ambient air, but also on the temperature of the air hence, the shape of the sorption isotherm are influenced by the temperature.

3.2 Effects of Packaging Materials on the Sorption Study.

Packaging plays an important role in achieving the objectives of safety and waste prevention (Famurewa et al., 2013). From Figure 1, it was observed that there were interactive effects among the packaging materials used to store tacca starch. The plastic packaging material absorption of moisture was very low. This corroborates the report of Famurewa et al., 2013 that plastic materials are good for storing and packaging of food products. The absorption of moisture by different packaging material is a reflection of the porous structure of the material. High density polyethylene was the next and low density polyethylene gives the highest absorption rate. Moisture content of a food material has a preservative effect on the product during storage. The lower the moisture content of a product, the longer the shelf-life Sanni et al., (2008).

3.3 Evaluation of Isotherm Models

The models fitted for tacca starch are given in Table 1. The closeness of the predicted values from each model to the experimental data over aw ranges at the temperature levels are shown in Figures 2a to 2s for the various model and different packaging materials. The values of the parameters (estimated unknown values) for the models which are the co-efficient of fit (R2), REE and SEE are all shown in Table 2.

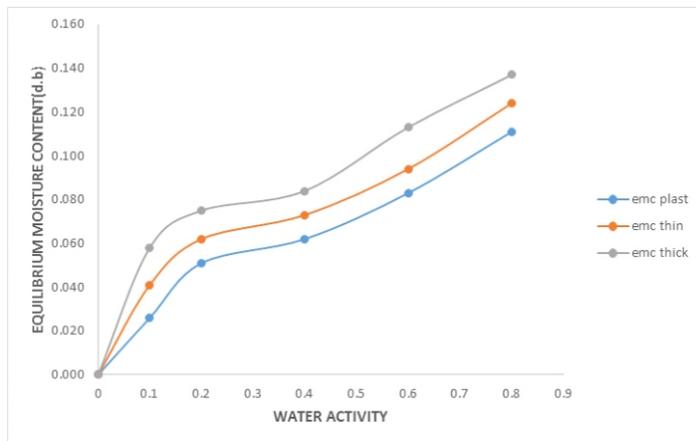


Figure 1: Sorption Isotherm for tacca Starch

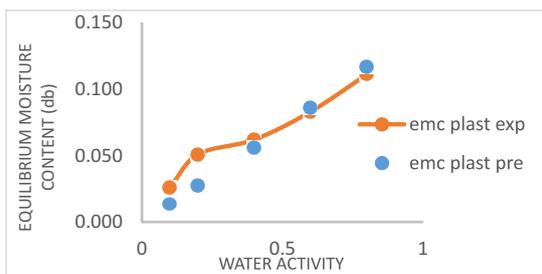


Figure 2a: GAB model graph for plastic container

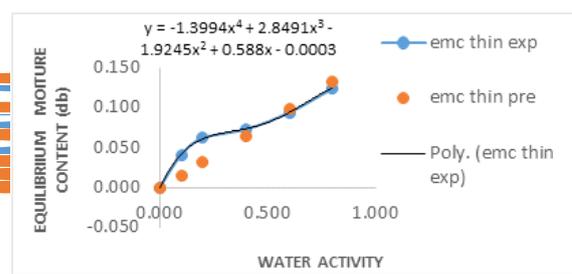


Figure 2b: GAB model graph for HDP

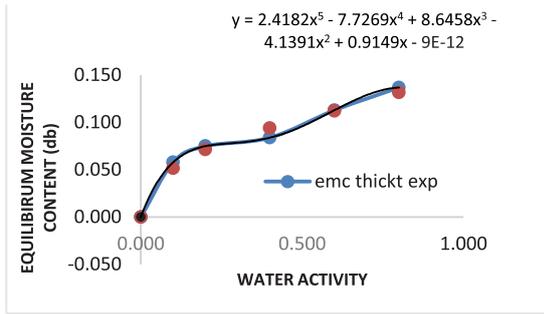


Figure 2c: GAB model for Thick container.

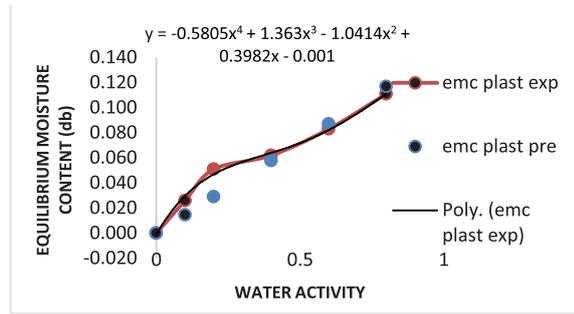


Figure 2d: BET model for plastic container.

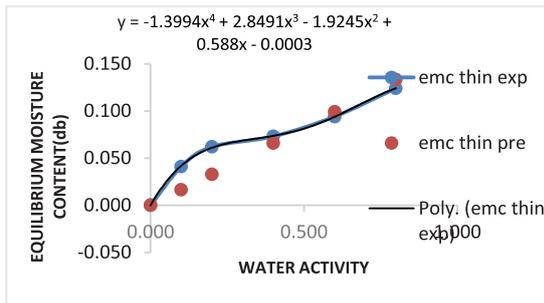


Figure 2e: BET model for HDP container.

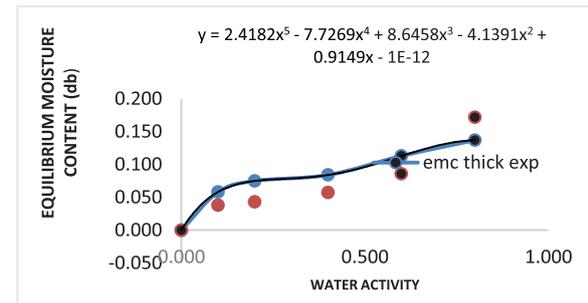


Figure 2f: BET model for LDP container.

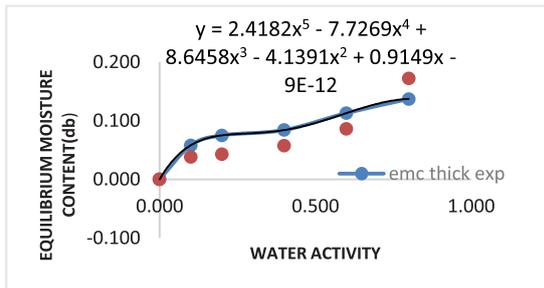


Figure 2g: BET model graph for LD container

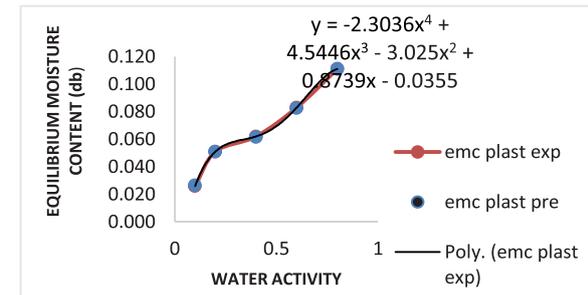


Figure 2h: Oswin model graph for plastic container

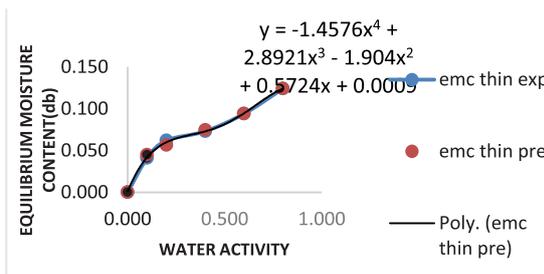


Figure 2i: Oswin model graph for HD container.

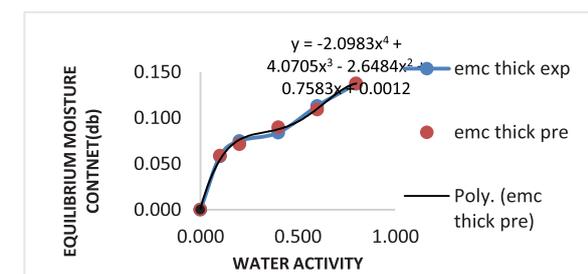


Figure 2j: Oswin model graph for LD container.

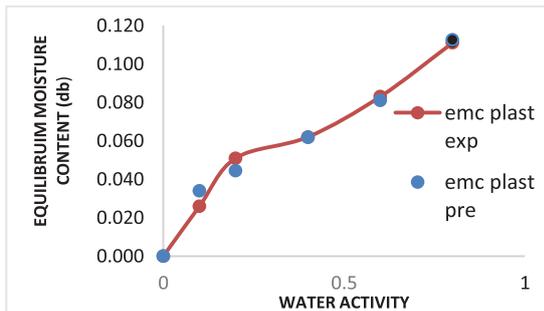


Figure 2k: Modified Oswin model graph of plastic container

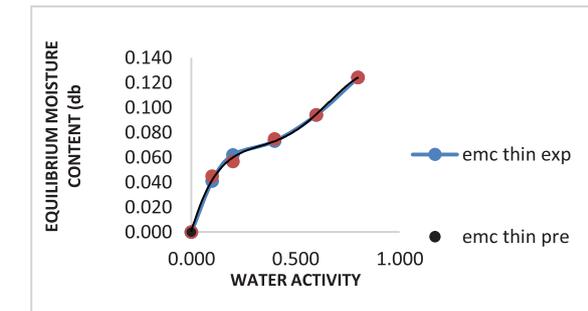


Figure 2l: Modified Oswin model graph of HD container.

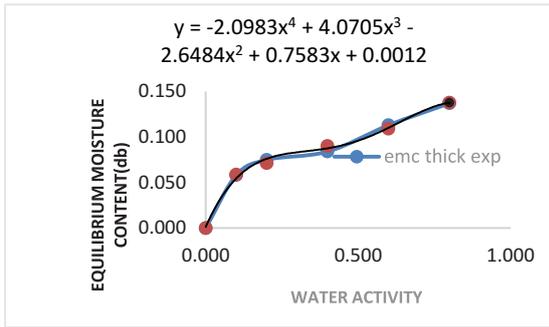


Figure 2j: Modified Oswin model graph for LDR container

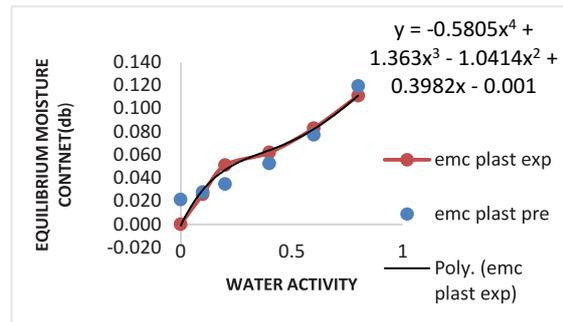


Figure 2k: Smith model graph for plastic container

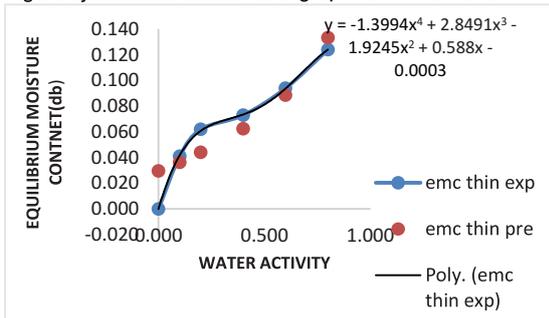


Figure 2l: Smith model graph for HDP container.

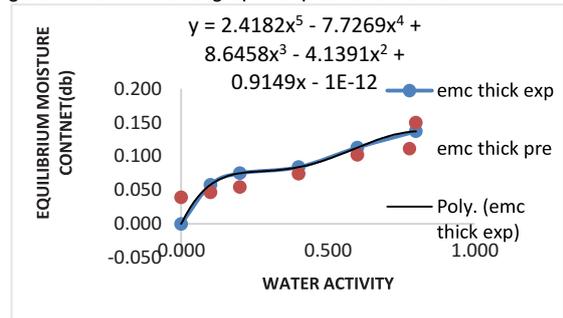


Figure 2m: Smith model graph of LDP container.

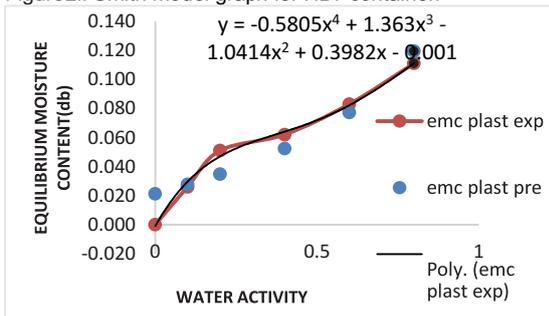


Figure 2n: Hasley model graph for plastic container

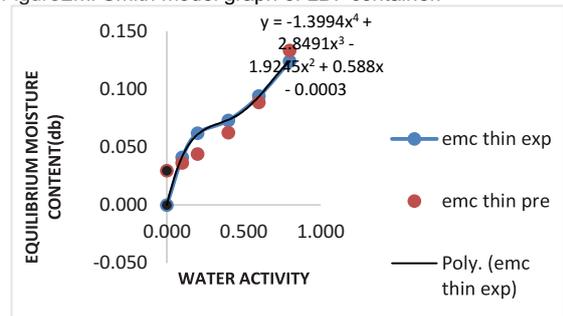


Figure 2o: Hasley model for HDP container.

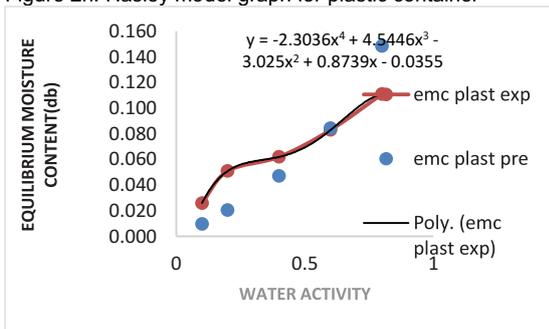


Figure 2p: Hasley model for LDP container.

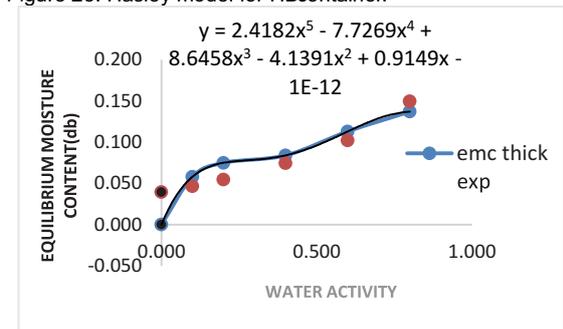


Figure 2q: Henderson model for plastic container

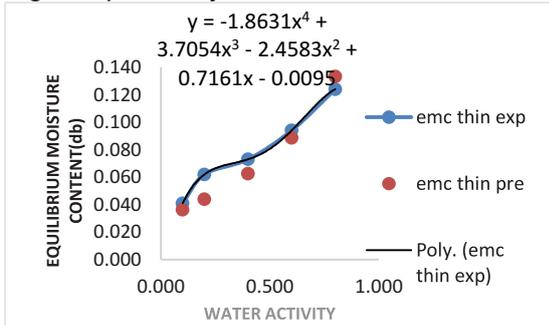


Figure 2r: Henderson model for HDP container

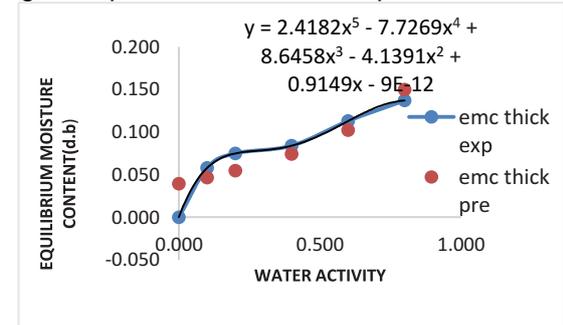


Figure 2s: Henderson model for LDP container.

Table 3: Estimated values for fitting models and model evaluation indicators For Tacca Starch at Ambient Temperature.

Models	Plastic Packaging	(HDP) Packaging	(LDP) Packaging
GAB(30)	K= 0.8830 C= 12.4357 Mo= 0.0607 RSS= 0.000789 SEE= 0.006818 R ² = 0.9492	K= 0.9423 C= 12.2588 Mo= 0.0597 RSS= 0.00719 SEE= 0.0105 R ² = 0.9135	K= 0.4329 C= 25.3079 Mo= 0.0691 RSS= 0.000185 SEE= 0.001225 R ² = 0.9841
BET	C= 0.5051 Mo= 0.0257 RSS= 0.000689 SEE= 0.00556 R ² = 0.9537	C= 0.4316 Mo= 0.02867 RSS= 0.00161 SEE= 0.00924 R ² = 0.9196	C= 0.2394 Mo= 0.0347 RSS= 0.00418 SEE= 0.01414 R ² = 0.8233
OSWIN	C= 0.7673 n= 0.9278 RSS= 0.00001 SEE= -0.000038 R ² = 0.9999	C= 0.0883 n= 0.2834 RSS= 0.000048 SEE=-0.00004 R ² = 0.9948	C= 0.0991 n= 0.2373 RSS= 0.000066 SEE= -0.0000051 R ² = 0.9941
MODIFIED OSWIN	c ₁ = 0.0212 c ₂ = -0.061 RSS= 0.00091 SEE= 7.49x 10E-13 R ² = 0.8838	a= 0.0838 b= 0.2834 c= 0.2834 RSS= 0.000048 SEE=-0.00004 R ² = 0.9948	a= 0.0991 b= 0.2834 c= 0.2373 RSS= 0.000066 SEE= -0.0000051 R ² = 0.9941
SMITH	c ₁ = 0.0212 c ₂ = -0.061 RSS= 0.00091 SEE= 7.49x 10E -13 R ² = 0.8838	c ₁ = 0.0295 c ₂ = -0.0644 RSS= 0.00145 SEE= 2.59x10E -11 R ² = 0.8425	c ₁ = 0.0393 c ₂ = -0.0685 RSS= 0.7806 SEE= -2.66x10E -11 R ² = 0.7806
HASLEY	c= 0.0212 n= -0.661 RSS = 0.0013 SEE = 7.49x10E-13 R ² = 0.8838	c= 0.0295 n= -0.0644 RSS= 0.00144 SEE= 2.50x10E-11 R ² = 0.8427	c= 0.0393 c _n = -0.0685 RSS= 0.00246 SEE= -2.66x10E-11 R ² = 0.7807
HENDERSON	c= 3.5 n= 3.099 RSS= 0.0013 SEE= 7.49x10E-13 R ² = 0.8838	c= 0.02 n= -0.0295 RSS= 0.8425 SEE= 2.59x10E-11 R ² = 0.8425	c= 0.0393 n= -0.0685 RSS= 0.00246 SEE= -2.66x10E-11 R ² = 0.7807

a, b, c and k are the model constants; RSS = residual sum of squares; SEE= the standard error of estimate; and R the co-efficient of fit.

3.4 *Effect of Water Activity on the Equilibrium Moisture Content*

Water activity had effect on the equilibrium moisture content of the samples. The equilibrium moisture content of the samples increased with an increase in water activity at constant temperature. These report are consistent with reports by other researcher (Alakili et al., 2009; Talla, 2012).

3.5 *Statistical Test Results*

Based on the closeness of R^2 to unity, the least values of RSS and SEE or closer to zero, the models were evaluated in terms of reliability of fit (Chowdhury et al., 2005, Aviara et al., 2006). The R^2 values of plastic packaging materials, thin (HDP) packaging materials, Thick (LDP) packaging materials of all the models ranged from 0.7806 to 0.9999. Hence, from table 2 above, the ranking was done on this fact and was observed that Oswin model was best for plastic packing material with R^2 value of 0.99991, RSS value of $7.27E-07$ and SEE value of $-3.8E-05$. The modified Oswin model with R^2 value of 0.99478, RSS value of $4.8E-05$ and SEE value of $-4E-05$ gave the best model for HDP packaging material which is similar to Chen, 2002 finding for product high in starch and corroborates with Oyelade, 2008 for lafun. Oswin model gave the best model for the thick LDP packaging material with R^2 value of 0.994126, RSS value of $6.6E-05$ and SEE value of $5.08E-06$ this finding is similar to the findings of Kaymak-Ertekin and Gedik, 2004 that Oswin equation are good for products high in starch such as potato.

3.6 *Monolayer Moisture Content*

The parameters for the value of tacca starch for the model used are presented in table 2. The importance of GAB and

BET models has always been used in determining the physiochemical explanations of their parameters. Especially the Monolayer moisture content is a parameter used in explaining GAB and BET model. The values of the monolayer moisture content for different packaging materials ranged from 0.02565 to 0.03437 for BET equation and 0.05972 to 0.06912 for GAB from Table 3.

4. Conclusion

The investigation of sorption isotherm of tacca starch at ambient temperature unveiled sigmoid shape type II typical for most food products. The equilibrium moisture contents for tacca starch increased with increase in water activity. At low water activity, very small amount of water was absorbed onto the active site but at high water activity, much more water was absorbed leading to rapid increase in equilibrium moisture content. Comparison between experimental results and predicted equilibrium moisture contents showed that Oswin model appears to be the most suitable in describing the experimental results of plastic and LDP packaging materials while Modified Oswin model was suitable for the thin (HDP) packaging material. The reliability sequence in which the evaluated models fitted the moisture sorption isotherms are Oswin, Modified Oswin, BET, Smith, GAB, Hasley and Henderson respectively for Plastic packaging material. The sequence for HDP packaging material are Modified Oswin, Oswin, Smith, Henderson, Hasley, BET and GAB respectively. While for LDP packaging material was Oswin, Modified Oswin, Hasley, Smith, Henderson, GAB and BET. The sorption isotherm of tacca starch showed that packaging materials had effect on the rate adsorption.

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