MOISTURE SORPTION ISOTHERM STUDY ON BREADFRUIT (*ARTOCARPUS ALTILIS*) FLOUR

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Abstract
Breadfruit is a seasonal and highly perishable food due to its high moisture content. Breadfruit is highly nutritious in nature, its yields in terms of food are superior to other starchy staples such as cassava and yam. The mature fruit is a good source of carbohydrate (84%) with starch constituting more than 60% of the total carbohydrate. Conversion of breadfruit into flour was a means of preserving and prolonging the shelf life of breadfruit yet it still becomes unstable by absorbing moisture (moisture sorption property) even after dehydration thereby reducing their shelf life hence a pertinent need to study the sorption isotherm properties of breadfruit flour. Matured Breadfruit was purchased at Osu market in Osun State and was processed into flour. The breadfruit flour was stored at 10, 27 and 40 ^{0} C relative humidity of 15%, 30%, 45%, 60% and 75% using the gravimetric method. Concentrated sulphuric acid (H_2SO_4) was used to create varying conditions. Various quantity of H_2SO_4 was used to make up a 250 ml of desiccant with deionized water, prepared at 10, 27 and 40^0 C to obtain water activities of 0.15, 0.30, 0.45, 0.60 and 0.75. Samples were monitored in the incubator for equilibration by weighing at intervals. Moisture content of the equilibrated samples were calculated and were taken as equilibrium moisture. Five isotherm equations BET, GAB, Halsey, Smith and Chung-Pfost were investigated with the experimental data. The result of the experiment and predicted equilibrium moisture content showed that GAB model fitted best for describing the experimental results of breadfruit flour isotherm at 10, 27 and 40^{0}C having the highest R^2 and Lowest SEE.

Key words: Moisture sorption isotherm, Mathematical modelling, Hysteresis Breadfruit

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INTRODUCTION

The quality of most foods preserved by drying depends to a great extent upon their physical, chemical and microbiological stability. This stability is mainly a consequence of the relationship between the equilibrium moisture content (EMC) of the food material and its correspondence water activity (aw), at a given temperature. Sorption isotherm describes the thermodynamic relationship between water activity and the equilibrium of the moisture content of a food product at constant temperature and pressure. The knowledge and understanding of sorption isotherms is highly important in food science and technology for the design and optimization of drying equipment, design of packages, predictions of quality, stability, shelf-life and for calculating moisture changes that may occur during storage. Sorption isotherm can be used to investigate structural features of a food product, such as specific surface area, pore volume, pore size distribution and crystallinity (Enujiugha *et al.,* 2006). Sorption isotherms can be generated from an adsorption process or desorption; the difference between these curves is defined as hysteresis (Raji *et al.*, 2011).

Many mathematical models has been used to describe the drying process of which thin-layer drying models are the most common models. According to Al-Muhtaseb (2002), mathematical models that describe drying mechanism of grain and food can also provide the required temperature and moisture content information. The most common equations used for describing sorption in food products includes Langmuir equation, the BET equation, the Oswin model, the Smith model, the Halsey model, the Henderson model, the Iglesias-Chirife equation, the GAB model, and the Peleg model (Sahin *et al*., 2006).

Breadfruit is a type of starchy fruit free from gluten and packed with nutrient. It is one of several "super foods" in ancient times which is high in carbohydrates and a good source of antioxidants, calcium, carotenoids, copper, dietary fibre, energy, iron, magnesium, niacin, omega 3, omega 6, phosphorus, potassium, protein, thiamine, vitamin A and vitamin C. Conversion of breadfruit into flour was a means of preserving and prolonging the shelf life of breadfruit (Famurewa, 2015), yet it still becomes unstable by absorbing moisture (moisture sorption property) even after dehydration thereby reducing their shelf life, its storage stability and resulting into its deterioration.
Attempt is, therefore, made in this study to evaluate the moisture sorption isotherm properties of breadfruit flour at varying temperature and relative humidity to determine the best storage condition that minimizes the rate of absorbing moisture thereby prolonging its shelf life.

MATERIALS AND METHODS

1. Breadfruit Flour Preparation: Matured Breadfruit was purchased from a local farm in Ile-Ife, Osun State, Nigeria. Other equipment that were used include slicer, stainless steel knife, bowl, weighing balance, aluminum foil paper, biomass fuelled dryer, hot air oven dryer, attrition milling machine and 0.35 mm mesh size. Thoroughly sorted breadfruits were cleaned, peeled with a stainless knife, and were sliced with manual slicer to 3 mm thickness and subjected to drying Hot air oven (Model IR701SG, Lec Refrigerator Plc, Britian) at 60 degree Celsius, it was dried until constant weights were achieved. The dried samples was milled with attrition mill and sieved with 0.35 mm mesh. The processed breadfruit flour was stored at selected temperature and relative humidity until constant mass of flour was attained using static gravimetric method. Figure 1 shows the steps in the preparation of breadfruit flour.

2. Determination of Equilibrium Moisture Content

The equilibrium moisture content of *breadfruit* flour was determined by static gravimetric method as applied according to Oyelade et al., 2001; Oyelade, 2008. For the adsorption process, the *breadfruit* flours were dehydrated in a hot air oven to bone dry. Duplicate samples, 3.0±.001g each of smoked dried, ovens dried and toasted *breadfruit* flour were weighed into moisture pans in the desiccators. The Concentrated sulphuric acid quantities used to make up a 250ml of desiccant with deionised water to achieve corresponding water activity prepared and stored at room temperature of 27°C, below room temperature (15 °C) and above room temperature (40 °C). The desiccators were maintained at aw values of 0.15, 0.30, 0.45, 0.60 and 0.75 respectively. The desiccators were kept at room temperature (15±20C, 27±2^{0}C and 40±2^{0}C). Each of the samples was being weighed at two days intervals using a digital balance until constant weight was obtained in three consecutive recordings, then the sample was assumed to be at equilibrium (±0.001g).The bone dry mass was determined by the oven-drying method for 8hrs at 105±5°C (AOAC, 1990).

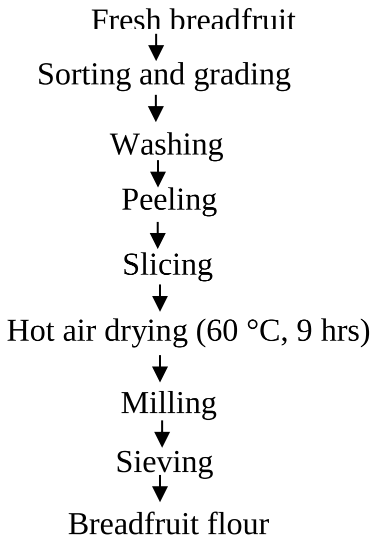

Fig. 1: Breadfruit flour processing

The time to reach equilibrium ranged between 10 to 21days depending on the water activity in each of the desiccators and the temperature of storage; those at higher water activities reaching equilibrium faster than those at lower water activities.

3. Determination of Sorption Isotherm

The equilibrium moisture contents were calculated from which the moisture sorption Isotherms were plotted for the dried *breadfruit* samples.

4. Sorption Equation

The experimental data were fitted to five commonly used models using linear regression analytical procedure. The models were the two parameter equations such as GAB (Guggenheim, 1966; Anderson, 1946; and de Boer, 1953), Halsey (Iglesias and Chirife, 1976), BET (BET, 1938), Smith (Smith, 1947) and Chung Pfost (Chung and Pfost, 1967).

These models were chosen because of their suitability for high carbohydrate foods, simplicity and ease of evaluation (Ajisegiri et al., 2007). The quality of fitness of the models was evaluated by calculating coefficient of determination (r^2). The equations of the five models are as follows;

a. GAB model

$$\frac{M_o CKaw}{(1-Kaw)(1-Kaw=CKaw)} \ldots\ldots\ldots\ldots\ldots eqn\ 1$$

where *M* is the moisture content (kg=kg dry solid), *M*0 is the monolayer moisture content; *C* and *K* are constants related to the energies of interaction between the first and further molecules at the individual sorption sites.

$$C = Co\exp\left(\frac{Ho-Hn}{RT}\right)\ldots\ldots\ldots\ldots eqn\ 2$$

$$K = koexp\left(\frac{Ho-Hn}{RT}\right)\ldots\ldots\ldots\ldots eqn\ 3$$

b. Hasley model

$$Me = M0(-A/(RT1naw))^{1/n}\ldots\ldots\ldots eqn\ 4$$

Where A and n are constants R is the universal gas constant T is the absolute temperature and Mo is monolayer moisture content.

Since the use of the RT term does not eliminate the temperature dependence of A and n, the Halsey equation was modified by Iglesias and Chirife, (1976) into the following form

$$Mw = Mo\left(-\frac{A}{Inaw}\right)1/n.\ldots\ldots\ldots\ldots eqn\ 5$$

c. BET model

BET equation is generally expressed in the form:

$$Mw = \frac{CMoaw}{(1-aw)(1+(C-1)aw)}\quad\ldots\ldots\ldots\ldots eqn\ 6$$

Where *M* is the moisture content (kg=kg dry solid), M_0 is monolayer moisture content (kg=kg dry solid), *aw* 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.

***d.* Smith model**

The Smith model can be written as:

$$Mw = C1 + C2\ 1n\ (1-aw)\ldots\ldots\ldots eqn\ 7$$

Where C1 is the quantity of water in the first sorbed fraction, and C2 is the quantity of water in the multilayer moisture fraction. This equation could be used in the water activity range from 0.5 to 0.95 in the case of wheat desorption and for various products (Ricardo *et al.*, 2011).

e. Chung P-fost model

$$Mw = \left(\frac{1}{C1}\right)1n\ (1n(ERH)(\frac{C2-T}{C2})\ldots..eqn\ 8$$

RESULTS AND DISCUSSION

1. Sorption Isotherms

The sorption isotherm have an S-shape profile (a type II isotherm according to Brunauer's classification) and correspond to multilayer formulation. Region I is represented by the monolayer moisture, which is strongly bond to material. Region II includes multilayer moisture which is under transition to natural properties of free water and is available for chemical reactions. The water in region III is in Free State, held in the voids, crevices and capillaries (Menkov *et al.*, 2007). The increasing trend of equilibrium moisture content agrees with the fact generally observed that the higher the value of water activity the more the quantity of adsorbed moisture. This corroborates Moore's (1979) observation that at higher water activity, more water is available for bonding at the active site of the solid. Isotherm which followed this principle have been reported in the literature (Oyelade 2008). The adsorption isotherm in this study was determined by plotting the equilibrium moisture content against different water activities, the sorption isotherm curve is similar to that reported by Ramanathan *et al.*, (1994) for compacted flour, Menkov *et al.*, (2007) for sesame and walnut flour, Oyelade, (2008) for lafun, Budi *et al.*, (2010) for taro flour.

Table 1.0 shows the Equilibrium Moisture Content values for the respective water activity and temperature. EMC values decreased with an increase in temperature at constant water activity and increased with an increase in relative humidity at constant temperature. This is similar to the work carried out by Menkov *et al.*, (2007) for sesame flour. 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. At increased temperatures water molecules get activated to higher energy levels, causing them to become less stable and break away from the water binding sites of the material, thus decreasing Equilibrium Moisture Content (Al-Muhtaseb *et al.*, 2004)

TABLE 1 Equilibrium Moisture Content (%) of Breadfruit flour at several temperature and water activity

Relative humidity (%)	EQUILIBRIUM MOISTURE CONTENT		
	10^0C	27^0C	40^0C
15	0.053	0.043	0.032
30	0.063	0.057	0.047
45	0.086	0.078	0.063
60	0.101	0.087	0.077
75	0.141	0.124	0.103

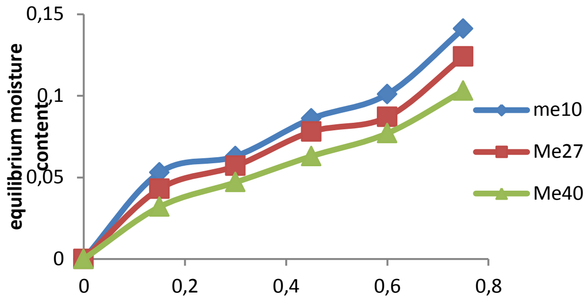

Figure 2. Sorption isotherm behaviour of Breadfruit flour at 10 ^{0}C, 27 ^{0}C and 40 ^{0}C

2. Fitting of Experimental Data to Sorption Isotherm Models

In models for breadfruit flour stored at 10^0C, R^2 ranges from 0.6561 to 0.9950 with the highest value for the B.E.T. The SEE for all models ranged from 0.0075 to 2.9984. Based on the highest R^2, least values of RSS and/or SEE, the models were evaluated in terms of reliability of fit (Chowdhury *et al.*, 2005). The G.A.B model gave the best fit among the equations tested having high R^2 of 0.9727 and SEE of 0.0075.

In models for breadfruit flour stored at 27^0C, R^2 ranges from 0.5576 to 0.9984 with the highest value for the B.E.T. The SEE for all models ranged from 0.0045 to 1.4304. Based on the highest R^2, least values of RSS and/or SEE, the models were evaluated in terms of reliability of fit (Chowdhury *et al.*, 2005; Oyelade, 2008). The G.A.B model gave the best fit among the equations tested having high R^2 of 0.9475 and SEE of 0.0045.

In models for breadfruit flour stored at 40^0C, R^2 ranges from 0.5020 to 0.9996 with the highest value for the B.E.T. The SEE for all models ranged from 0.0013 to 0.1255. Based on the highest R^2, least values of RSS and/or SEE, the models were evaluated in terms of reliability of fit (Chowdhury *et al.*, 2005). The G.A.B model gave the best fit among the equations tested having high R^2 of 0.9906 and SEE of 0.0013. The result is in agreement with cassava flour film plasticized with sorbitol which was best fitted with G.A.B model (Phan *et al.*, 2005)

3. Monolayer Moisture Content

The monolayer value of breadfruit flour for the models used is presented in table 2.0. The importance of GAB and BET models has always been used in determining the physiochemical explanations of their parameters (Igbabul *et al*., 2013). The values of the monolayer moisture content at different temperatures ranges from 0.0375 to 0.0449 for BET equation and 0.0477 to 0.0512 for GAB equation.

The monolayer moisture content represents the moisture content at which the water attached to each polar and ionic groups start to behave as a liquid-like phase. Exclusive attention is paid to the monolayer value. However, the values of the energy constants should not be overlooked nor ignored because they are simultaneous outputs of the regression processes and they influence the sigmoidal shape of the isotherms.

On the other hand, the third GAB constant k determines the profile of the isotherm at the higher water activity range, regulating the upswing after the plateau at medium water activity range. Higher values of k determine a more pronounced upswing. Timmermann *et al*., 2001. The monolayer moisture content Mo, can be determined from equilibrium sorption isotherm data by fitting BET, GAB stability of foods. The prediction of Mo has a significant importance for physical and chemical stability of foods. Below Mo values, food deterioration is expected to be extremely small, because water is strongly bound to the food and water is not involved in any deteriorative reaction (Ali *et al*., 2011).

Table 2 Estimated values of fitting models and model evaluation indicators of breadfruit flour

Models	10 ^{0}C	27 ^{0}C	40 ^{0}C
BET	c = 57.5387	c = 22.6047	c = 15.3611
	*M*o = 0.0477	*M*o = 0.0449	*M*o = 0.0375
	SEE = 2.9984	SEE = 1.4304	SEE = 0.0153
	E% = 17.6207	E% = 17.6582	E% = 18.6078
	R^2 = 0.9950	R^2 = 0.9984	R^2 = 0.9996
GAB	k = 0.8371	k = 0.7882	k = 0.7504
	c = 42.1962	c = 21.4638	c = 11.5501
	*M*o = 0.0529	*M*o = 0.0512	*M*o = 0.0477
	SEE = 0.0075	SEE = 0.0045	SEE = 0.0013
	E% = 5.6197	E% = 3.3946	E% = 1.1869
	R^2 = 0.9727	R^2 = 0.9475	R^2 = 0.9906
Halsey	k = 4.7020	k = 5.0809	k = 5.1870
	c = -1.7077	c = -1.7662	c = -1.6745
	SEE = 1.0415	SEE = 1.0232	SEE = 0.1255
	E% = -27.6077	E% = -25.6520	E% = -39.465
	R^2 = 0.6561	R^2 = 0.5576	R^2 = 0.5020
Smith	k = 0.0520	k = 0.0548	k = 0.0652
	c = 0.0310	c = 0.0398	c = 0.0464
	SEE = 0.0306	SEE = 0.0131	SEE = 0.0402
	E% = 14.5540	E% = 13.2940	E% = 58.3470
	R^2 = 0.7483	R^2 = 0.9771	R^2 = 0.9745
Chung-Pfost	k = -0.0562	k = 0.0480	k = -0.0416
	c = 0.0627	c = 0.0555	c = 0.0451
	SEE = 0.2013	SEE = 0.0202	SEE = 0.0185
	E% = 18.6003	E% = 22.2230	E% = 26.7271
	R^2 = 0.7483	R^2 = 0.6824	R^2 = 0.6599

C and k are the model constants,
RSS = residual sum of squares;
SEE = the standard error of estimate; and R^2 the coefficient of fit

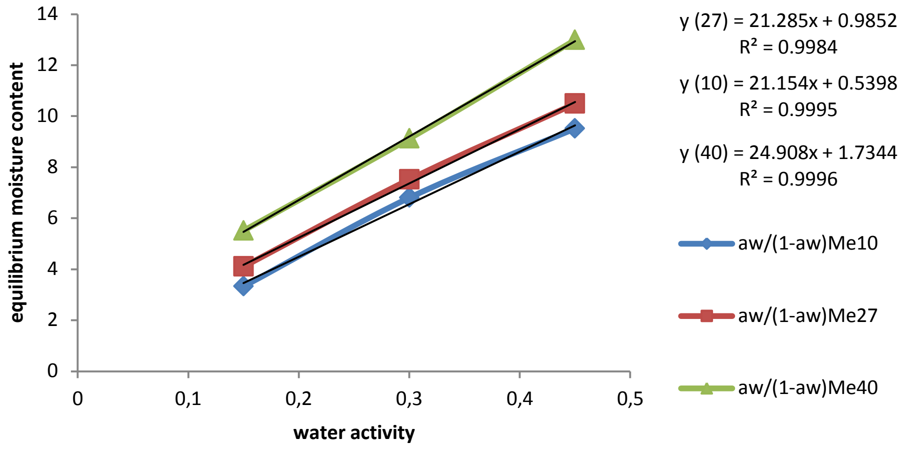

Figure 3: BET model graph at 10, 27 and 40^{0}C

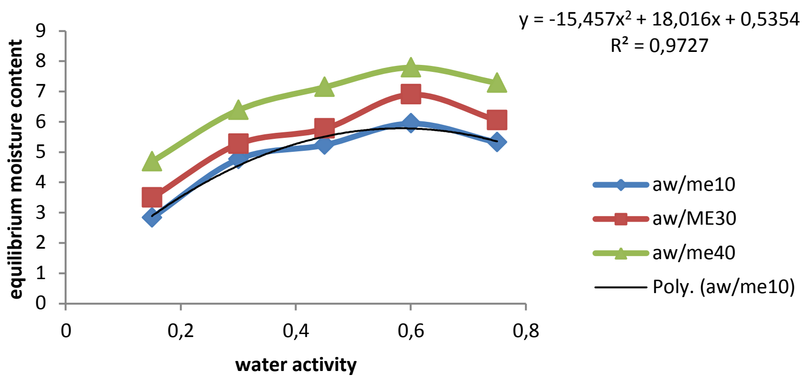

Figure 4: GAB model graph at 10, 27 and 40^{0}C

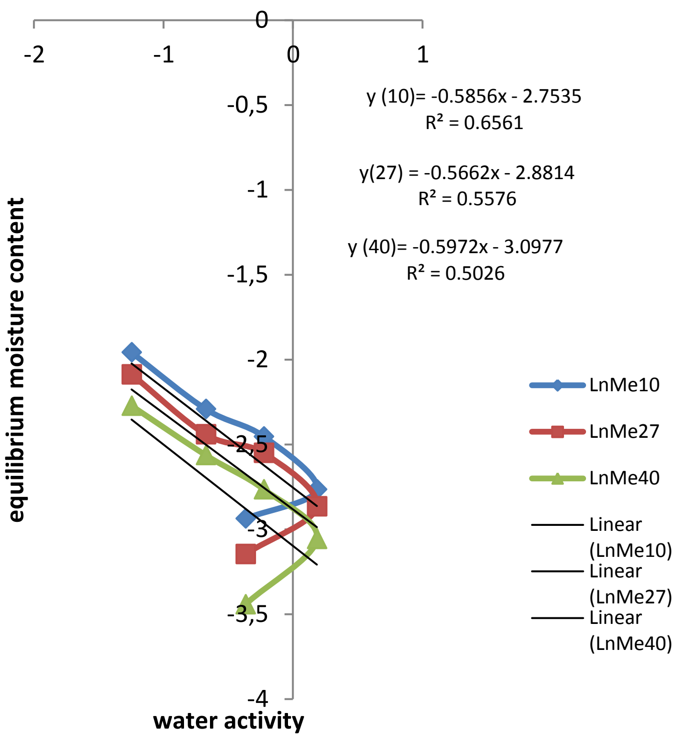

Figure 5: Halsey model graph at 10, 27 and 40^{0}C

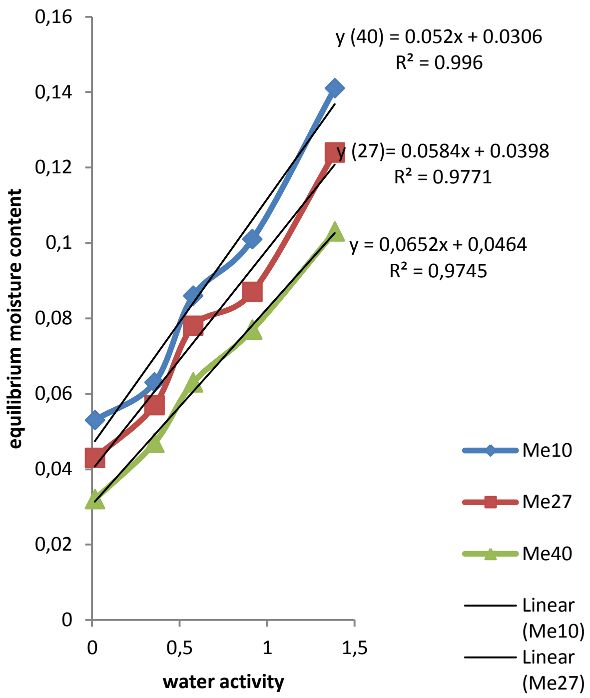

Figure 6: Smith model graph at 10, 27 and 40^{0}C

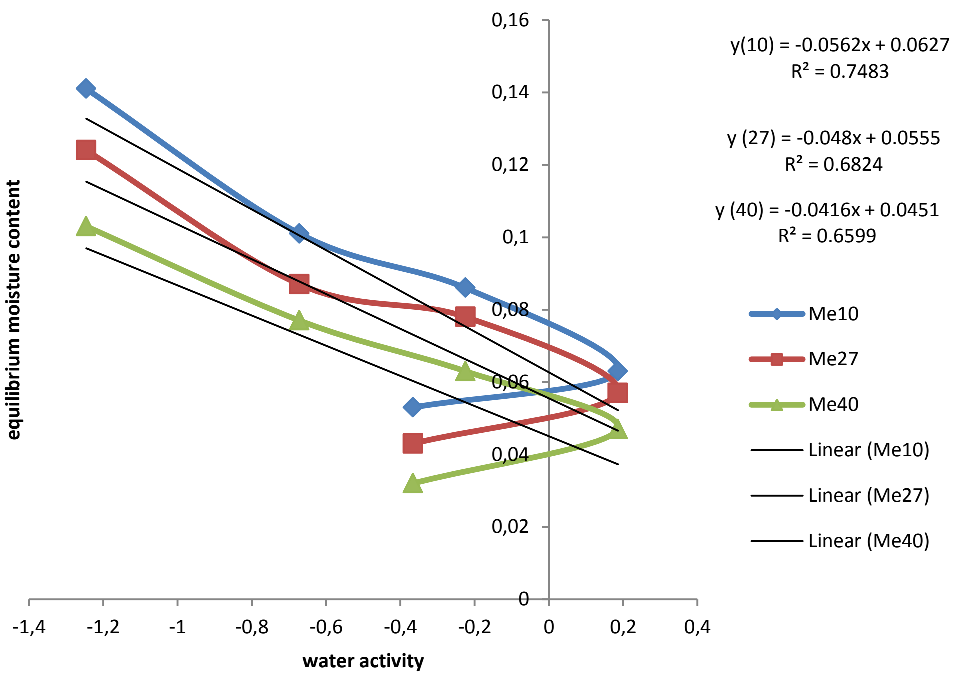

Figure 7: Chung-Pfost model graph for Breadfruit flour at 10, 27 and 40^{0}C

CONCLUSION

Based on the outcome of this study, quality attributes of breadfruit flour is significantly influenced by storage conditions.

The result of this study has established that at 27^0C, GAB model is suitable to predict the storage condition and sorption isotherm properties of *Artocarpus altilis* flour. This GAB model remains the most suitable even at 10^0C and 40^0C. The study has provided information on the sorption characteristics of *Artocarpus altilis* flour and models for its storage have been validated.

These information will assist food processors in the area breadfruit flour storage.

COMPETING INTERESTS

Authors have declared that no competing interests exist.

REFERENCES

[1] Ajisegiri, E.S.A., Chukwu, O., Sopade, P.A. (2007). Moisture- sorption study of locally parboiled rice. Au J. T., 11(2), 86 -90.

[2] Ali C. D., Hatice P., and Kadir B. B. (2012). Effect of drying methods on the moisture sorption isotherms and thermodynamics properties of mint leaves *Journal of Food Science and Technology* 49(4): 439-449

[3] Al-Muhtaseb A.H, NcMinn W.A.M., Magee T.R.A (2004). Sorption isotherms of starch powders, Part 1: Mathematical description of experimental data, *Journal of Food Engineering.* 61:297-307

[4] Al-muhtaseb, A. H., Mcminn, W. A. M. and Magee T. R. A. (2002), Moisture sorption isotherm characteristics of food products: a review. Trans IChemE, Volume 80, Part C, pp 118.

[5] Anderson, R. B., (1946),Modi. cation of the B.E.Tequation, *Journal of American Chemical Society*, 68: 686–691.

[6] Brunaer S, Emmett P.H, Teller E (1938). Adsorption of gases in multimolecular layer. *Journal of American Chemical Society* 60(2): 309-319

[7] Budi Nurtama and Jenshinn (2010). Moisture Sorption Isotherm Characteristics of taro Flour World Journal of Dairy & Food Sciences 5 (1): 01-06, 2010 ISSN 1817-308X IDOSI Publications, Department of Food Science, National Pingtung University of Science and Technology, Pingtung 91201, Taiwan

[8] Chowdhury M.M.I., Huda, M.D., Hossain, M.S (2005). Moisture Sorption Isotherm for Mungbean (*Vigna radiate*). *Journal of Food Engineering* 74 (4): 462-467

[9] de Boer, J. H., 1953, *The Dynamical Character of Adsorption* (Clarendon Press, Oxford, UK).

[10] Enujiugha V.N., Iyiola S.O., Oluwamukomi M.O., Gbadamosi S., Oluwalana I.B. and Lalude L.O., 2006. Sorption Isotherms of Some Nigerian Dry Condiment Powders. *Journal of Food Technology, 4: 242-248.*

[11] Famurewa J. A, Pele G. I, Esan Y. O., and Jeremiah B. P. 2016. Influence of Maturity and Drying Methods on the Chemical, Functional and Antioxidant Properties of Breadfruit (Artocarpus altilis). British Biotechnology Journal 16(1): 1-9, 2016, Article no.BBJ.25535

[12]Guggenheim, E. A., (1966). *Application of Statistical Mechanics* (Clarendon Press, Oxford, UK).

[13] Igesias, H.A., Chirife, J., Amodel W. (1976). For describing the water sorption behaviour of foods, *Journal of Food Science*, 41, 984.

[14] Menkov N.D and Durakova A.G (2007): Moisture Sorption Isotherm of Sesame flour, *Food, Technology Biotechnology. 45 (1): 96-100*

[15] Oyelade O.J (1997). Moisture isotherms of cowpea flour in relation to packaging. M.Sc project report, Uiversity of Ibadan, Ibadan Nigeria.

[16] Oyelade O.J. (2008). Equilibrium Moisture Content Models for Lafun. *International Journal of Food Engineering* 4(2) Article 4:1-14

[17] Phan D., Debeaufort, F., Luu, D. and Voilley, A. (2005). Functional properties of edible agar based and starch-based films for food quality preservation. *Journal of Agricultural and Food Chemistry*, 53: 973-981

[18] Raji A.O, Ojediran J.O (2011). Moisture of two variables of millet. *Food Bioprod Process.* 89 (3); 178-184

[19] Ramanathan S and Cenkwoski S. (1994). Sorption isotherm of flour and flow behaviour of flour as influenced by flow compaction. Department of Agricultural Engineering. University of Winnipeg, Manitoba, MB, Canada

[20] Sahin S, Gulum S. (2006). Physical properties of foods. New York, United States: *Springer;* pp 218
Smith S. E., (1947). The sorption of water vapour by high polymers, *Journal American Chemistry Society*, 69: 646.

[21]Timmermann, O., Chirife, J. and Iglesias, H. A., (2001). Water sorption isotherms of foods and foodstuffs: BET or GAB parameters? *Journal Food Engineering*, 48: 19–31.