Journal of Food and Agriculture
DOI: https://doi.org/10.4038/jfa.v17i1.5315

The Sorption Isotherm of Pretreated Breadfruit Starch Flour

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ABSTRACT

This study investigated the sorption isotherm of breadfruit starch with the view of providing sorption isotherm data that will enhance its storability and utilization in food formulation. The sorption isotherm of breadfruit starch flour at temperature levels (30, 35, 40, and 45 ℃) and water activity (ranging from 0.1 – 0.9) using sulphuric acid was experimentally investigated in an incubator until equilibrium moisture content (EMC) was obtained gravimetrically. The sorption data obtained was fitted into six different existing mathematical models using linear (Origin statistical procedure) and non-linear (Gauss-Newton) regression programs. The models were compared using the coefficient of determination (R^2), residual mean square error (RMSE) and the sum of square error (SSE). The sorption models' predictive parameters were used to determine the best-fitting model. The results showed that the Modified Henderson model and Halsey model are best suited to predict the sorption isotherm of breadfruit (*Artocarpus altilis*) flour. In conclusion, this research gives insights for understanding the sorption isotherm behaviour of the breadfruit flour which is crucial in predicting shelf-life stability, designing and optimization of drying equipment and choosing appropriate packaging material for the product.

Keywords: Breadfruit, Sorption isotherm, Starch flour.

INTRODUCTION

Starch is an important food ingredient and raw material used in food and non-food industries. It is used in food industries as additives for thickening, preservation and as a quality enhancer in baked foods, confectioneries, pastas, soups and sauces, and mayonnaises (Egharevba, 2019). Apart from its usage in the food and beverage industry, it has found extensive applications in many other industries such as health and medicine, agriculture, textiles, pulp and paper, chemicals and pharmaceuticals, biofuel production, mining and building (Przetaczek-

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Rożnowska, 2017). In pharmaceutical industries, it is used as diluent, disintegrant, lubricant and binder (Afolayan *et al.,* 2012).

Starch is a major source of energy in the human diet and therefore it is of great economic importance. Its conventional sources are corn, cassava, sweet potato, wheat, potato, sorghum, barley and rice (Laity *et al*., 2010). The highest-producing countries of starch are Thailand (43.71%), Vietnam (30.08%), Indonesia (11.71%), Brazil (2.49%), Laos (2.47%), Paraguay (2.18%), Cambodia (1.72%), Netherlands (1.66%), Germany (1.02%), and Nicaragua (0.57%). Due to ever-increasing demand for starches in modern food industries, there is a dire need to source for starch from other non-conventional sources which are readily available (Afolayan *et al.,* 2012).

Breadfruit, a non-conventional source of starch is a tree crop that grows in the tropics and sub-tropics. It is an excellent source of vitamin A, thiamin (B_1), riboflavin (B_2), niacin (B_3), folate, pantothenic acid (vitamin B_5), and minocycline. It contains trace amounts of only polyunsaturated fat which helps reduce low-density lipoprotein (LDL) cholesterol and reduces the risk of heart disease. The protein in breadfruit is composed of a combination of leucine and lycine. These amino acids are both used to build body proteins but the body cannot synthesize them on its own. The world's leading producers of breadfruit are Canada (12.45%), Thailand (7.02%), Poland (6.55%), Chile (6.11%), Peru (5.68%) United States (5.09%) and Netherlands (4.15%) (Nuga *et al.,* 2010). Breadfruit cultivation is distributed in countries across different continents of the world (about 20 countries in Africa, 32 countries in Asia, 14 countries in Oceania and 14 countries in South America (Nuga *et al.*, 2010).

In Africa, breadfruit is typically cultivated in West and Central African countries such as Tanzania, Zambia, Nigeria and Uganda (Nuga *et al.*, 2010; Baerts *et al.,* 2017). Despite its huge production rate, breadfruit is highly perishable and this accounts for its unavailability all year round. Therefore, processing it into different value-added products will minimize its postharvest losses.

Recent technological advances and modern food consumption patterns have led to spring up of many food processing industries in Nigeria.

This development has contributed significantly to starch consumption by these industries. In order to cater for these huge demands, food processing and other related industries depend largely on importation of starch. Therefore, utilizing potential of breadfruit as a source of starch can help in bridging the lacuna. This work then focuses on processing of starch from breadfruit and investigate its sorption isotherm properties with the view of providing engineering data that will enhance its processing and utilization in food formulation.

MATERIALS AND METHODS

Material Collection

Fresh and matured breadfruit was obtained from Obafemi Awolowo University Teaching and Research Farm, Ile-Ife, Nigeria and chemicals of analytical grades were used for this work.

Sample Preparation

The freshly harvested mature breadfruit was washed with clean water to remove the sticky latex and dirt. After washing, debarking operations were carried out using a stainless-steel kitchen knife and the sliced pulp was then dipped into sodium metabisulphite (1.25 g/L) solution to prevent enzymatic browning reaction (Adebayo *et al.*, 2021). The soaked pulp was drained and rinsed with portable water. The pulp was macerated at low speed using a Stephan universal machine (Western Germany, Model No-P33/E) for 5 min. A homogenate slurry obtained was sieved using an electrical SWECO separator (Belgium, Model No- S18). A stainless steel decanting bowl was then used to separate the filtrate which was subsequently left to settle for 3 h, after which the supernatant was discarded. The starch slurry was then dried using a hot air oven (Uniscope, SM9053, England) at 50 ^{0}C for 24 h. The starch flakes were then milled using a laboratory milling machine and sieved with a mesh size of 500 μm to get starch flour. The native starch obtained was stored in properly labelled zip-lock bags at room temperature till the time of usage.

Determination of Breadfruit Sorption Isotherms

The equilibrium moisture content (EMC) of the breadfruit starch flour was determined by gravimetric technique, in which sample weight was monitored continuously according to the methods documented by Okunola *et al.* (2015) at four different levels of temperature. Sorption isotherm data obtained was then fitted into existing sorption isotherm models using Design Expert 2.0.

Sulphuric acid solutions with water activity in the range of 0.1 – 0.9 was used to maintain the specified relative humidity inside the jars. Sulphuric acid has been used in sorption isotherm studies by other researchers (Peng *et al.*, 2007; Shindano, 2007). The desiccator lids were sealed with petroleum gel and placed at desired temperatures in GallenKamp DV 300 incubator (Weiss Gallenkamp, UK). A sample weight of 2 g was then weighed with analytical balance (Mettler-Toledo Inc., USA) having an accuracy of 0.0001 g and put in three replicates in the desiccators in all the experiments. A digital air oven (Memmert GmbH +Co KG, Germany) was used for moisture content determination at 105 ^{0}C ± 1 ^{0}C with the sample being weighed at 1 h intervals until a constant weight of ± 0.001 g is achieved (AOAC, 2005). The EMC was obtained as an average of three replicates on dry basis (d.b. %).

Modeling of Sorption Isotherms

Six sorption equations shown in Table 1 which have been established suitable for predicting equilibrium moisture content (EMC) for high carbohydrate foods over a wide range of water activity was used to analyze the experimental data in this report. The observed EMC and their respective a_w were then imputed into a linear regression program to predict EMC in the two parameter models using origin statistical procedure. A non-linear least grid search was used for the three parameter models using Guass-Newton method having two temperature levels incorporated for its applicability over the varying temperatures. The goodness of fit of these models was evaluated by statistical parameters of determination co – efficient (R^2) and residual mean square error (RMSE) and sum of squares error (SSE) which are defined as follow:

$$SSE = \frac{1}{N}\sum\left(MR_{exp,.i} - MR_{pre.,i}\right)^2 \quad (1)$$

$$R^2 = \frac{\sum_{i=1}^{N}\left(x_{pi}-\bar{x}_{ei}\right)^2}{\sum_{i=1}^{N}\left(x_{ei}-\bar{x}_{ei}\right)^2} \quad (2)$$

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

Table 1. Isotherm models for experimental data fitting.

Author	Model	Linear Form
Hasley (1948)	$M = -\frac{A^{1/B}}{T \ln a_w}$	$\ln M = \frac{\ln A}{BT} + \frac{\ln(-\ln a_w)}{B}$
Herderson (1952)	$M = -\frac{\ln(1 - \ln a_w)^{1/B}}{AT}$	$\ln M = -\frac{(-\ln(1 - a_w))}{B} + \frac{1}{B} \ln \frac{1}{AT}$
Chung and Pfrost (1967)	$M = \frac{1}{B} \ln \frac{A}{RT} - \frac{\ln(-\ln a_w)}{AT}$	$M = \frac{1}{B} \ln \frac{A}{RT} - \frac{1}{B} \ln(-\ln a_w)$
Modified Henderson (Thompson *et al.*, 1968)	$M = \frac{\ln(1 - a_w)^{1/C}}{-A(T + B)}$	$M = \frac{1}{C} \ln(-\ln(1 - a_w)) - \ln A\,(T + B)$
Modified Halsey (Iglesias and Chirife, 1976)	$M = \frac{e^{(A - BT)^{1/C}}}{-\ln a_w}$	$\ln M = \frac{(A + BT) + \ln(-\ln a_w)}{C}$
GAB (Guggenheim-Anderson-de Boer model, 1984)	$M_w = \frac{M_0 C K a_w}{(1 - K a_w)(1 - K a_w + C K a_w)}$	

M = EMC (decimal, dry basis), M_O = Monolayer moisture content (d.b); K = GAB model parameter; a_w = water activity (decimal); A, B and C, are parameters constant pertinent to each equation. T = Absolute temperature (K) R = Universal gas constant (J/mol). Source: Sahin and Sumnu (2006).

Where; x_{ei} and x are experimental and predicted equilibrium moisture contents, respectively and N is the number of experimental data. A model is considered acceptable if it has MRE value less than 10% (Kaymak-Ertekin and Gedik, 2004). Monolayer moisture content (M_o) will be determined using GAB model presented in (Table 1).

Isosteric Heat of Sorption

The net isosteric heat of sorption of differential enthalpy was determined from the experimental data using the Clausius-Clapeyron equation (Tsami *et al.*, 1990).

$$-\Delta h_d = R \left[\frac{d \ln a_w}{d\left(\frac{1}{T}\right)} \right]_x \quad (4)$$

Where: R is the gas constant and T is the absolute temperature (K) at the corresponding a_w level (Akanbi *et al.*, 2006). Re-plotting the experimental sorption isotherm in the form $\ln a_w$ versus $1/T$, for a specific moisture content, x, the slope of the regression line $-\Delta h_d/R$ provided a measure of the net isosteric heat of sorption. This procedure is based on the assumption that Δh_d is invariant with temperature, with measurement of the sorption isotherms at more than two temperatures being required for application of the method (McMinn *et al.,* 2007). The method is to determine the influence of temperature on the dynamic equilibrium between a_w, and moisture content, using Clausius-Clapeyron equation.

RESULTS AND DISCUSSION

The experimental sorption isotherm data curve of breadfruit starch flour is presented in Figure 1. Figure 1 shows that the equilibrium moisture content (EMC) of the sample increased as water activity (a_w) increased at each level of temperature investigated. Figure 1 further showed that as temperature increased, there was a decrease in the EMC values at all a_w levels. The sorption isotherm of breadfruit starch flour exhibited a sigmoid shape (Al-Muhtaseb *et al*., 2002) which further explained the fact that the EMC increased with increasing a_w. This was owing to the flour components' ability to absorb moisture via hydrogen bonding with water molecules (Kuye and Sanni, 2002). A similar pattern was documented for cassava starch (Perdomo *et al*., 2009). The sorption isotherm pattern indicated that when the a_w of the surrounding environment was higher, water molecules migrated from the environment into the sample until an equilibrium was reached. In addition to water bio-polar, its low-molecular weight also aids in the sorption of water at elevated a_w. Therefore, the result showed that breadfruit starch flour would be best stored at lower a_w levels. This becomes necessary due to the fact that foods are exposed to a distinct degree of temperatures during storage and processing: a_w fluctuates with temperature and the impact of temperature on the sorption isotherm is therefore a major concern to food processors.

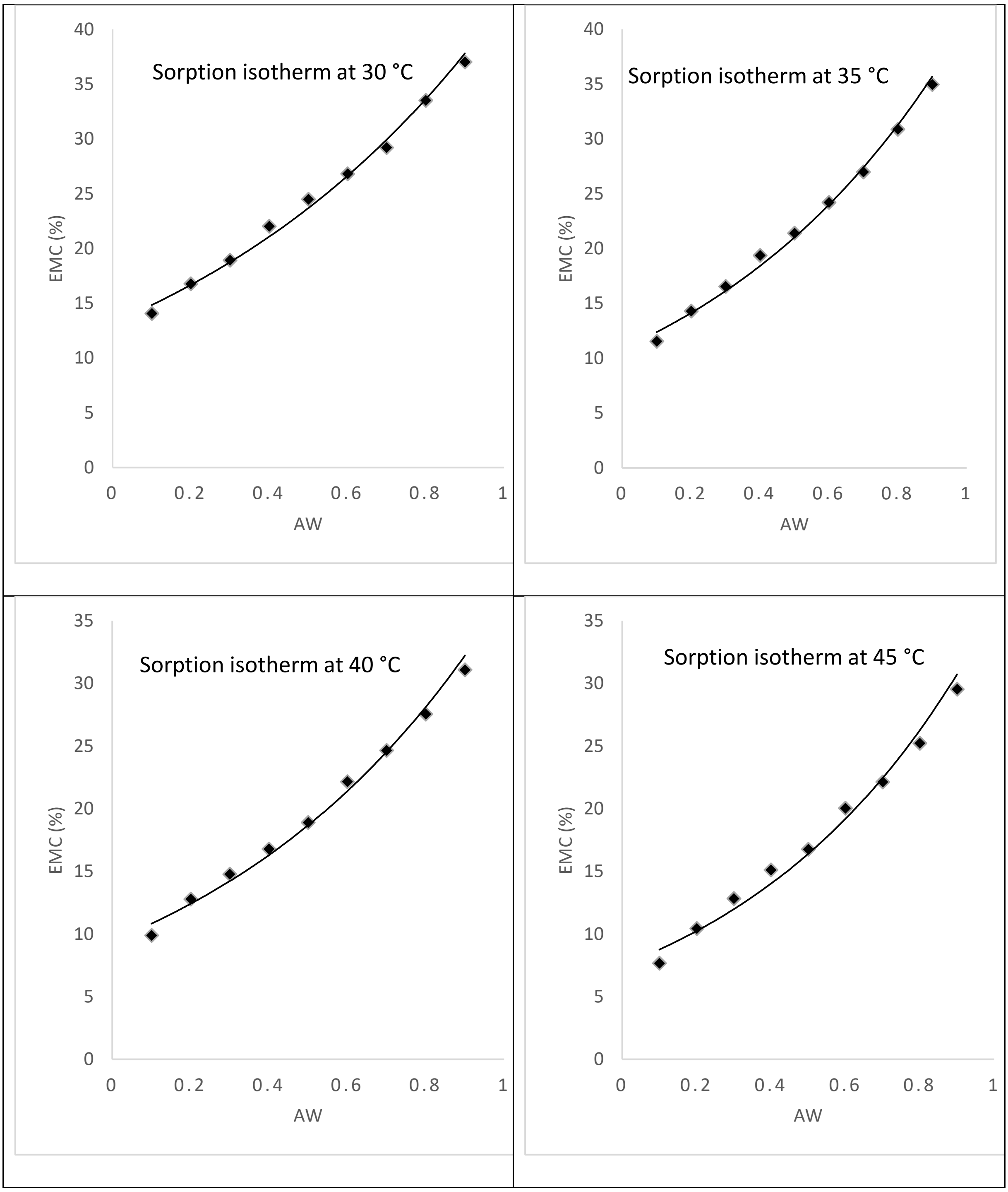

Figure 1. Experimental sorption isotherm curve of breadfruit starch flour.

Five known mathematical models, Halsey, Henderson, Chung and Pfost, Modified Henderson and GAB models, were used to correlate the experimental data. The model parameters were determined by applying the non-linear regression method as presented in Table 2. The goodness of fit of the equations was evaluated using the root mean square error (RMSE), sum of square error (SSE), and coefficient of determination (R^2). The R^2 values ranged from 0.9667 – 0.9968 with Modified Henderson and Hasley models having the highest and lowest values, respectively (Table 2). This showed that the Modified Henderson adsorption model fits the experimental data reasonably well, with the highest coefficient of determination value (COD) (0.9968).

The SSE values ranged from 1.2140 – 2.1733 with Modified Henderson and Chong and Pfost models having the highest and lowest values, respectively. This showed that the Chong and Pfost adsorption model fits the experimental data reasonably well, with the lowest SSE value (1.2140).

The RMSE values ranged from 1.1018 – 1.4742 with Modified Henderson and Chong and Pfost models having the highest and lowest values, respectively. This showed that the Chong and Pfost adsorption model fits the experimental data reasonably well, with the lowest RMSE value (1.2140). The SSE and RMSE values for Halsey model are 1.50175 and 1.2254. This shows that the Halsey model also fits this experimental process, having low values for the sum of square error and root mean square error. The GAB model also significantly fits with the experimental process having high R^2 value and low SSE and RMSE values with the values 0.9962, 2.53793 and 1.5931, respectively. Also, parameters such as the monolayer capacity (M_o), constant related to multilayer adsorption (c), and heterogeneity factor (k) were obtained as 0.0388, 0.060075 and 0.004475, respectively. The values obtained for each parameter were less than one which indicates that the flour had a relatively low capacity to form a monolayer of adsorbate molecule. It also indicates the weaker interactions between the flour molecules in the multilayer coverage. This suggests that the formation of additional layers of adsorbate on the surface of the flour is less favourable.

Isosteric heat of sorption of breadfruit starch flour plotted from ln

Table 2. Parameter results from sorption isotherm of breadfruit starch flour models.

Model Name	Sorption type	Model parameter	Values	R^2	SSE	RMSE
Hasley	Adsorption	A	22348	0.9667	1.50175	1.2254
		B	2.4193			
Henderson	Adsorption	A	0.000186	0.9941	2.40175	1.5497
		B	1.88375			
Chong and Pfost	Adsorption	A	11.6585	0.9962	1.2140	1.1018
		B	-7.05125			
GAB	Adsorption	M_O	0.0388	0.9962	2.53793	1.5931
		C	0.060075			
		K	0.004475			
Modified Henderson	Adsorption	A	0.0078	0.9968	2.1733	1.4742
		B	-23.9			
		C	-19.2			

a_w against 1/T using equation 4 is presented in Figure 2. Slopes, linear equation, and R^2 values from the graphs are presented in Table 3. The results showed that isosteric heats of adsorption decreased exponentially as the EMC increased indicating that at higher EMC levels, the strength of water binding decreased. This behaviour indicated that change in the amplitude of the interactions between the a_w and food components as isosteric heat approached zero (McMinn and Magee, 2003). Therefore, water vapour on the sorbent exhibits physical characteristics that are similar to those of pure water. Adsorption occurs mostly at the monomolecular layer, where the sorption sites are often active, at low moisture contents. (Aviara and Ajibola, 2002; McMinn and Magee 2003). A similar isosteric heat of sorption trend was exhibited by potatoes (McLaughlin and Magee, 1998). Table 3 shows the net isosteric heat of sorption ranges from 9.353 – 13.436 kJ/mol. The values of isosteric heat of sorption in this study increased with increasing temperature values. The values obtained are lower than those obtained for potato (Kaymak-Ertekin and Gedik, 2004).

The R^2 values indicate how well the logarithmic equation fits the data. A higher R^2 value (closer to 1) indicates a better fit. This suggests that the logarithmic equations are reasonably good at predicting the isosteric heat of sorption based on the given variable. Based on this, the best fit for the isosteric heat of sorption for breadfruit starch was at 45 ^{0}C having the highest R^2 value of 0.9709 of which there were other R^2 values of

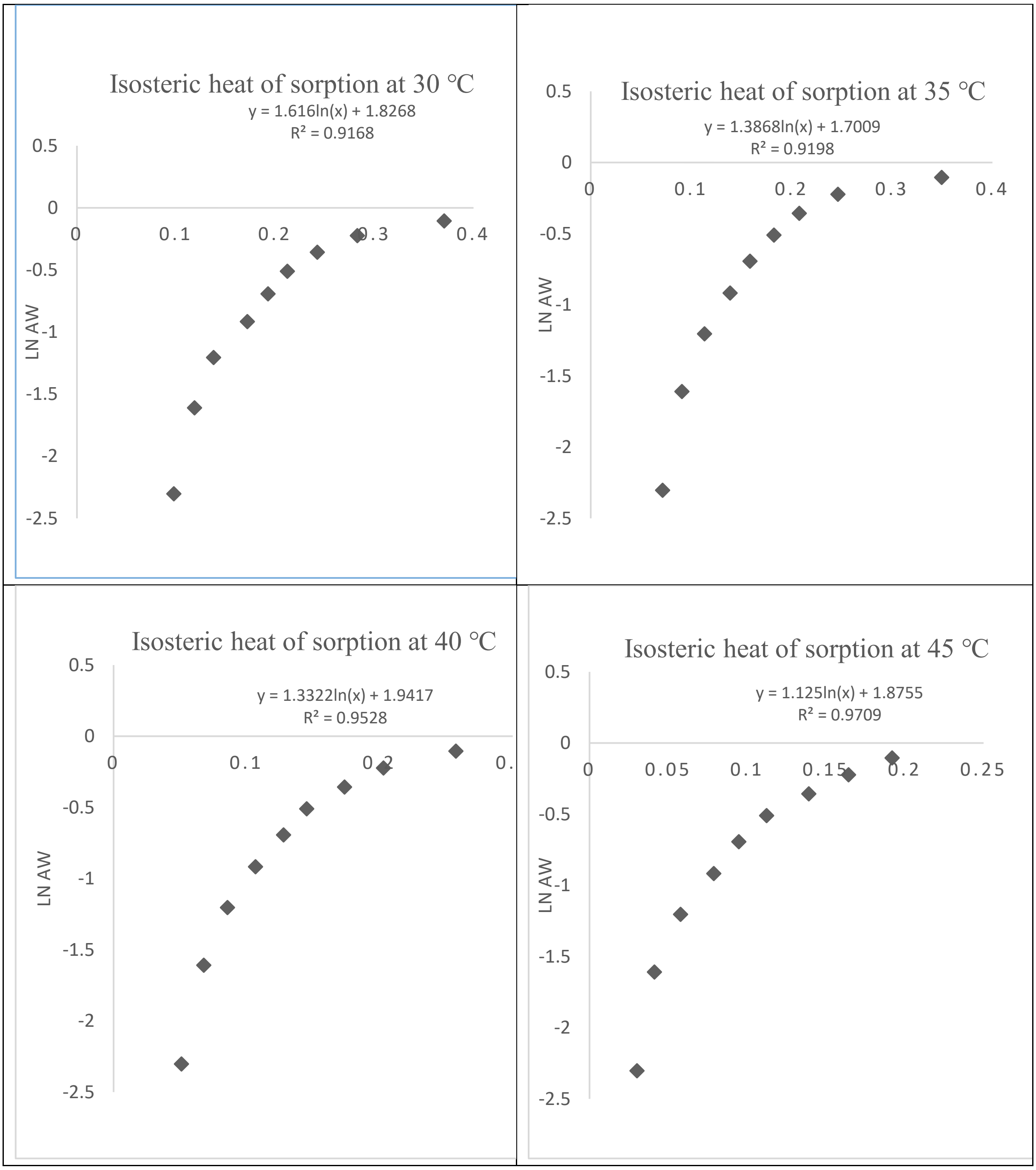

Figure 2. Isosteric heat of sorption of breadfruit starch flour.

Table 3. Isosteric heat of sorption of breadfruit starch.

a_w /1/T	$-\Delta h_d/R$	Logarithmic equation	R^2	Δh_d(kJ/mol)
30°C	-1.6161	Y = 1.616ln(x) + 1.8268	0.9168	13.436
35°C	-1.3868	Y = 1.3868ln(x) + 1.7009	0.9198	11.529
40°C	-1.2224	Y = 1.2224ln(x) + 1.6833	0.9176	10.163
45°C	-1.1250	Y = 1.1250ln(x) + 1.8755	0.9709	9.353

0.9168, 0.9198 and 0.9176 at 30 ^{0}C, 35 ^{0}C, and 40 ^{0}C respectively. In each equation, Y represents the dependent variable, which is Δh_d/R (the isosteric heat of sorption divided by the universal gas constant), the coefficient 'm' represents the slope of the line, x represents the independent variable, which is aw / 1/T. This is a combination of water activity and the inverse of temperature and the constants are the y-intercept. It indicates the rate of change of Δh_d with respect to the change in aw/1/T.

At 30 ^{0}C (Y = 1.616ln(x) + 1.8268), the coefficient 1.6161 is the slope of the line. It indicates that for every unit increase in Ln (a_w) / (1/T), the isosteric heat of sorption (Δh_d/R) increases by approximately 1.6161 units. The constant term 1.8268 is the y-intercept. This means that when aw / 1/T is zero, the isosteric heat of sorption is approximately 1.8268 times the universal gas constant.

At 35 ^{0}C (Y = 1.3868ln(x) + 1.7009), the coefficient 1.3868 is the slope of the line. It indicates that for every unit increase in aw / 1/T, the isosteric heat of sorption (Δh_d/R) increases by approximately 1.3868 units. The constant term 1.7009 is the y-intercept. This means that when aw / 1/T is zero, the isosteric heat of sorption is approximately 1.7009 times the universal gas constant.

At 40 ^{0}C (Y = 1.2224ln(x) + 1.6833), the coefficient 1.2224 is the slope of the line. It indicates that for every unit increase in aw / 1/T, the isosteric heat of sorption (Δh_d/R) increases by approximately 1.2224 units. The constant term 1.6833 is the y-intercept. This means that when aw / 1/T is zero, the isosteric heat of sorption is approximately 1.6833 times the universal gas constant.

At 45 ^{0}C (Y = 1.1250ln(x) + 1.8755), the coefficient 1.1250 is the slope of the line. It indicates that for every unit increase in aw / 1/T, the isosteric heat of sorption ($\Delta \boldsymbol{h_d}$/R) increases by approximately 1.1250 units. The constant term 1.8755 is the y-intercept. This means that when aw / 1/T is zero, the isosteric heat of sorption is approximately 1.8755 times the universal gas constant

CONCLUSION

This research work provided engineering data needed for understanding the sorption isotherm of breadfruit starch flour. Therefore, the following conclusion can be drawn from the results obtained.

The sorption isotherm behaviour of the breadfruit starch flour exhibits the sigmoid pattern. Sorption isotherm behaviour of the starch flour was temperature-dependent where the EMC values decreased as temperature values increased. Overall, the results revealed a best-fit and correlation between the experimental sorption isotherm data and the one predicted by Modified Henderson model and Halsey model.

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