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Isothermal adsorption modelling of *Pupuru* flour

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ABSTRACT

The amount of moisture adsorbed by stored food products usually varies with temperature and relative humidity at a constant pressure. This research was carried out to determine the sorption isotherm behaviour of *pupuru* flour in the temperature and water activity (a_w) ranges of 10−50 ºC and 0.10−0.93 using gravimetric method. The Guggenheim-Anderson-De Boer (GAB), Brunauer-Emmett-Teller (BET), Oswin and Smith sorption equations, formulated for the sorption characterization of starchy foods, were used to constitute the models. The results show that the sorption isotherm curve is similar to a Type II classification typical of starchy foods. Also, the sorption parameters (M_o, C and K) were generally higher for the products stored at 10 ºC and 0.10 a_w. The parameter *K* decreases with an increase in the temperature for the GAB and the Smith models, but remains inconsistent for the Oswin model. The M_o increases with an increase in the temperature for the GAB and Smith models. The GAB, Oswin and Smith models were generally adequate and valid with an MSE< 10% and a high degree of fitness (R^2 and R^2_{adj} > 90%). This indicates their ability to predict the sorption isortherm characteristics of the *pupuru* flour within its range of the a_w and the storage temperature.

Introduction

Pupuru is a fermented, dewatered, milled and toasted flour produced from cassava. It originates from the Ilaje people of the riverine area of Ondo State, South-west Nigeria (Shittu et al., 2001). The traditional processing of *pupuru* involves steeping the peeled cassava roots in water for four days after which the wet pulp is dried by spreading it on a raised platform, constructed using a raffia material locally known as *Aka*. Based on the processing method of *pupuru*, it is different from other cassava fermented products like *garri*, *lafun*, *fufu* and *akpu* (Osundahunsi, 2005). The fermented cassava mash can be mould into balls and smoke-dried. This is believed to impact some characteristics of flavour and aroma to this product (Shittu et al., 2003; Daramola et al., 2010). According to Famurewa et al. (2013), producing *pupuru* flour by toasting method can help solve the problem associated with making wet *pupuru* before smoking. A common problem is the deterioration of the product, and this is responsible for its occasional low market value and acceptability. The deterioration of the product may be associated with the activities of the spoilage organisms such as bacteria and fungi (Ilouno et al., 2016; Adebowale et al., 2017). An understanding of the sorption isotherm characteristics of the product is therefore critical to prevent the deterioration, especially before processing and storage (Oluwamukomi et al., 2008). The water sorption isotherm of foods is therefore of great importance.

A moisture sorption isotherm describes the thermodynamic relationship between water activity and the equilibrium moisture content of a food product at constant temperature and pressure (Erbaş et al., 2016). This relationship can be nonlinear or linearly constituted by two or more partial regression coefficients. Several mathematical models have been proposed for describing sorption isotherms of food products. Some of them were developed with a theoretical basis to describe adsorption mechanisms (Erbaş et al., 2016; Rockland and Stewart, 2013), whereas the others are just empirical or a simplification

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of more elaborate models. In some ranges of water activity, sorption isotherms can be approximated to linear equations (Yan et al., 2008). There are some semi-empirical equations with two or three fitting parameters which describe the moisture sorption isotherms of foods. The fundamental equations have been reported (Erbaş et al., 2016; Aguirre-Loredo et al., 2017; Mitrevski et al., 2017; Tejada-Ortigoza et al., 2017), but their application for predicting the adsorption and desorption characteristics depends on the nature of the food, water activity, equilibrium moisture content and the temperature of storage.
Guggenheim-Anderson-De Boer (GAB) and Brunauer-Emmett-Teller (BET) models have been applied most successfully to describe the equilibrium isotherm behviour of many food products (Tejada-Ortigoza et al., 2017). In fact, these models have been recommended by the European Project Group COST 90 on the Physical Properties of Foods as the fundamental equation for the characterization of water sorption of food materials (Samapundo et al., 2007). The adsorption and desorption isotherms of maize flour and yellow dent corn have been investigated by Oyelade et al. (2008) and Samapundo et al. (2007). The authors recommended the modified GAB model for predicting the safe moisture content for the storage of the products. Similarly, the effect of hydrophobic modifications on the adsorption isotherms of cassava starch has been reported (Cova et al., 2010). The authors reported that the water adsorption process was slower for modified starches. Although the storage stability of *pupuru* flour has been studied at room temperature (Famurewa et al., 2012), this study is limited, because the authors did not consider the storage effect at higher temperatures. Despite the wider application of the sorption isotherm models in predicting the sorption behaviour of stored food products, there are no reported research on their application to predict the sorption characteristics of *pupuru* flour. It is therefore imperative to determine the optimal storage conditions that would best maintain the quality attributes of the *pupuru* flour during storage and processing. Hence, the objective of this study was to determine sorption isotherm of *pupuru* at three different temperatures and water activities, and to validate sorption characteristics of the product using four different sorption equations for the starchy foods.

Materials and methods

Preparation of Pupuru

The preparation of the *Pupuru* flour was done as described by Famurewa et al. (2012). This involves peeling of the cassava tubers and washing them in clean water for 4 days at ambient temperature until they were softened. The water was changed every 24 hours to reduce the odour and allow the leaching of the antinutrient content out. After the fourth day, the water was drained and the fermented mash was pressed with a hydraulic press. The hard fibers were manually removed and the wet cassava meal sieved with a mesh to remove shaft. The meal was toasted in a wide aluminum pot for 20 min with a constant stirring to prevent burning and formation of lumps. Thereafter, the product was cooled and milled into flour. The sample of the *pupuru* flour was then packaged in HDPE film and kept in refrigerated storage for subsequent experimentation.

Determination of Equilibrium Moisture Content

The equilibrium moisture content of the *pupuru* flour was determined according to the procedures reported by Singh and Talukdar (2020) for static gravimetric analysis. The method involves the application of a saturated salt solution on the flour sample to establish a constant value of the water activity (a_w), until equilibrium is achieved between the atmosphere and the food. For the adsorption process, the *pupuru* flour samples were dehydrated in a hot air oven to bone dry state. Saturated solutions of LiCl, $MgCl_2$, $Mg(NO_3)_2$, Na_2NO_3, NaCl and KNO_3 salts were prepared and enclosed in a desiccator. The a_w of salt solutions were found to vary between 0.113 to 0.932 at three different temperatures, namely 10 °C, 30 °C and 50 °C. The sample of the *pupuru* flour (3 g) was placed into containers with the corresponding saturated salt solutions. Toluene was placed into the containers that had a_w values higher than 0.65 to prevent microbial growth. The desiccators were maintained at a_w values of 0.1 to 0.93. Each of the samples was weighed using a digital balance until a constant weight was achieved, thus indicating equilibrium. The bone-dry mass was determined by oven drying method (AOAC, 2005). The proceedure was repeated three times, and the average values and standard deviation of the equilibrium moisture content was computed at different temperatures and water activities of the *pupuru* flour samples.

Determination of Sorption Parameters of Pupuru Flour

GAB sorption model

The results of the moisture adsorption were fitted to the GAB equation (Fadeyibi et al., 2012; Bello et al., 2018). The GAB equation can be expressed as shown in Equation (1).

$$M_e = \frac{CkM_o a_w}{(1-ka_w)(1-ka_w+Cka_w)} M_o \quad (1)$$

The three parameters of the GAB model (c, k and M_o) were obtained from the second order polynomial expression, which was solved by multi-linear regression analysis to obtain α, β, γ, R^2 and E% (Xiong, 2002) according to equations (2) – (5).

$$\frac{a_w}{M_e} = \alpha {a_w}^2 + \beta a_w + \gamma \quad (2)$$

where

$$\alpha = \frac{k}{M_o}\left(\frac{1}{c-1}\right) \quad (3)$$

$$\beta = \frac{1}{M_o}\left(1-\frac{2}{c}\right) \quad (4)$$

$$\gamma = \frac{1}{M_o} Kc \quad (5)$$

The values of the GAB model parameters M_o, C and K, for each temperature, were therefore determined using equations (6) to (8).

$$M_o = \frac{1}{\sqrt{\beta^2-4\alpha\gamma}} \quad (6)$$

$$C = \frac{2\sqrt{\beta^2-4\alpha\gamma}}{-\beta+\sqrt{\beta^2-4\alpha\gamma}} \quad (7)$$

$$K = \frac{-\beta\pm\sqrt{\beta^2-4\alpha\gamma}}{2\alpha\gamma} \quad (8)$$

BET sorption model

The monolayer moisture content was also determined using the BET sorption model as expressed in equation (9) (Bello et al., 2018). The values of the equilibrium moisture content were plotted against water activity (a_w) to obtain sorption isotherm curves of the *pupuru* flour for different storage temperatures. The BET curves were obtained by plotting the graph $a_w/(1-a_w)M_e$ against a_w. The slope and intercept were determined from the BET curves using linear regression analysis. Monolayer moisture content (M_o) and the constant (C) were calculated from the analysis (Labuza and Altunakar, 2020).

$$M_e = \frac{M_o C a_w}{(1-a_w)(1+(C-1)a_w)} \quad (9)$$

$$\frac{a_w}{(1-a_w)M_e} = \frac{1}{CM_o} + \frac{a_w(C-1)}{CM_o}$$
$$\frac{a_w}{(1-a_w)M_e} = \alpha a_w + \beta$$

where

$$\beta = \frac{1}{CM_o}$$

$$\alpha = \frac{(C-1)}{CM_o}$$

M_o = monolayer moisture content,
C = energy constant,
M_e = equilibrium moisture content (moisture content adsorbed per dry weight) in gH_2O/g solids.

Oswin sorption model

The sorption data obtained were also fitted into the Oswin model as expressed in Equation (10). This model is essentially useful in describing the sorption isotherm behaviour of fat free and starchy foods. Thus, the model was used to determine the monolayer moisture content of *pupuru* flour.

$$M_e = C\left(\frac{a_w}{1-a_w}\right)^n \quad (10)$$

$$\ln M_e = \ln C + n \ln\left(\frac{a_w}{1-a_w}\right)$$
$$\ln M_e = \alpha \ln\left(\frac{a_w}{1-a_w}\right) + \beta$$

Thus, a plot of ln M_e versus ln (a_w/1-a_w) gives a straight line with slope n = k = α and intercept ln C = M_o = β.

Smith sorption model

The Smith sorption equation was used to predict the adsorption isotherm characteristics of the *pupuru* flour for different temperature conditions. The equation was used because it aids the understanding of the sorption behaviour of starchy foods which consist of a single and multiple layers of condensed water molecules. In this study, we computed the Smith sorption model parameters using Equation (11).

$$M_e = C_1 + C_2 \ln(1-a_w) \quad (11)$$

Putting $C_1 = M_o$, and C_2 = k, we have

$$M_e = M_o + k\ln(1-a_w)$$

where

C_1 = quantity of water in the first sorbed fraction,
C_2 = quantity of water in the multilayer moisture fraction.

Model Verification and Calibration

The validity of the GAB, BET, Oswin and Smith models for predicting the equilibrium and monolayer moisture contents of the *pupuru* flour for the different temperature conditions was tested. This was done by comparing the values with the corresponding values from an independent experiment (Ajisegiri et al., 2007). The fitness of the models was tested using the mean relative percentage error (MRE, %), and the coefficients of determination (R^2) and R_{adj}^2 as expressed in Equations (12) to (14), respectively.

$$MRE = \frac{100}{k}\sum_{i=1}^{N}\left(\frac{M_{ei}-M_{ci}}{M_{ei}}\right) \qquad (12)$$

$$R^2 = \sum\frac{(M_{pred}-M_{avg})^2}{(M_{avg}-M_{av})^2} \qquad (13)$$

$$R^2{}_{adj} = 1-\frac{(1-R^2)(k-1)}{k-p-1} \qquad (14)$$

where

M_{ei} = moisture content (% db)
M_{ci} = calculated moisture content (% db)
k = number of samples
p = number of independent variables
M_{pred} = model predicted moisture content (% db)
M_{avg} = average moisture content (% db)

Results and discussion

Sorption Isotherm Characteristics of Pupuru

The adsorption isotherm of *pupuru* samples are presented in Fig. 1. There was an increase in the equilibrium moisture content with an increase in the a_w. The sorption isotherm curve showed the typical sigmoid shape confirming type II classification which is the characteristic of the most biological material. This absorbs relatively small amount of water at lower activities and large amounts at high relative humidity. This agrees with the works of Kumar et al. (2005) for products rich in starch content. The moisture sorption characteristics of a product have been shown to be influenced by its composition, processing treatment, temperature and relative humidity (Ojuko and Olapade, 2018). The more the quantity of the adsorbed moisture, the higher the value of water activity.

Sorption Parameters of Pupuru Flour

The adsorption isotherm of the *pupuru* flour stored at different temperatures based on the GAB, BET, Oswin and Smith models are shown in Figs. 2–5, respectively. Quadratic relationships were established between the equilibrium moisture content and the a_w at different storage temperatures using the GAB and BET sorption models (Figs. 2 and 3). Also, linear relationships were established for the Oswin and Smith sorption models (Figs. 4 and 5). The magnitude of the a_w/M_e increases with the increase in the storage temperature until 0.6 a_w when it starts decreasing for the GAB models. The BET models, however, show a continuous s increase in the magnitude of the $a_w/(1-a_w)M_e$ with an increase in the storage temperature and a_w. An increase in the equilibrium moisture content with an increase in the storage temperature and a_w was observed for the Oswin and Smith models. The observed differences in the variable interraction may be due to the manner in which the models were constituted for sorption isortherm description. A similar result was reported by Arevalo et al. (2004) in their work on mathematical modelling of Inga pulp. Also, the research work of Oyelade et al. (2008) on the sorption isotherm of maize flour corroborates the findings of this study.

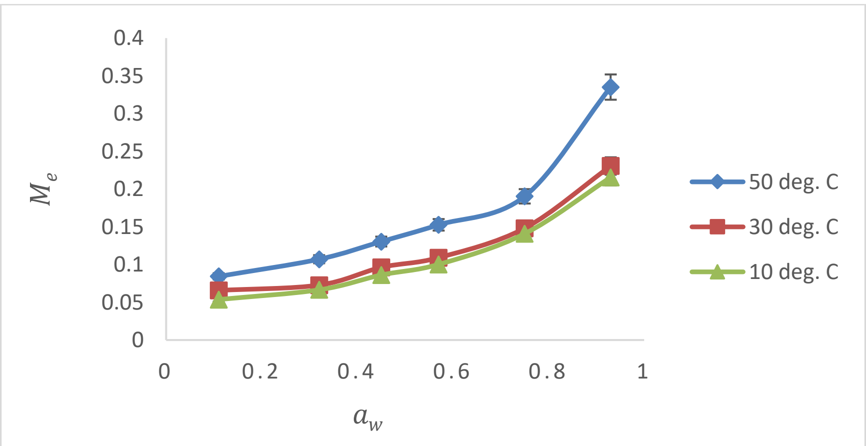

Fig 1. Adsorption Isotherm Curve of *Pupuru* flour at different temperatures

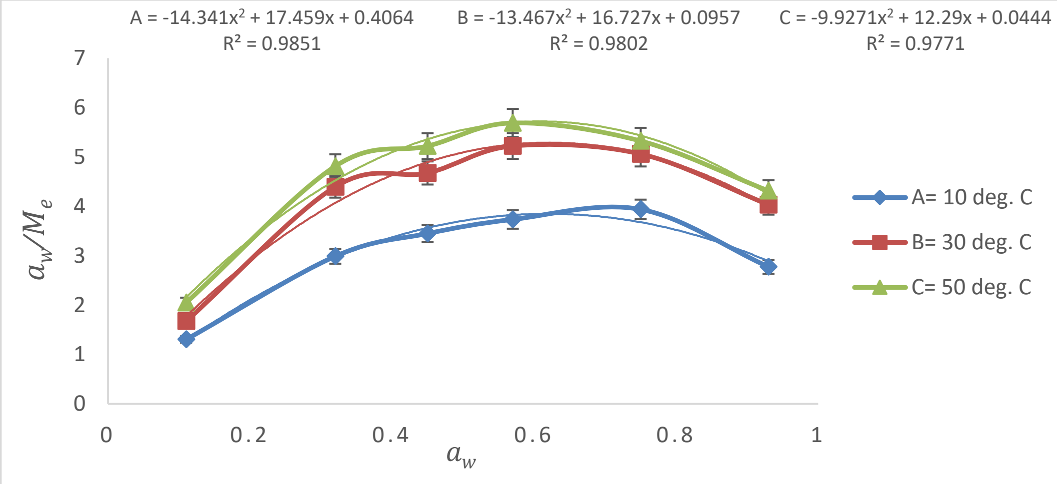

Fig 2. Sorption isotherm of stored *Pupuru* flour based on GAB model

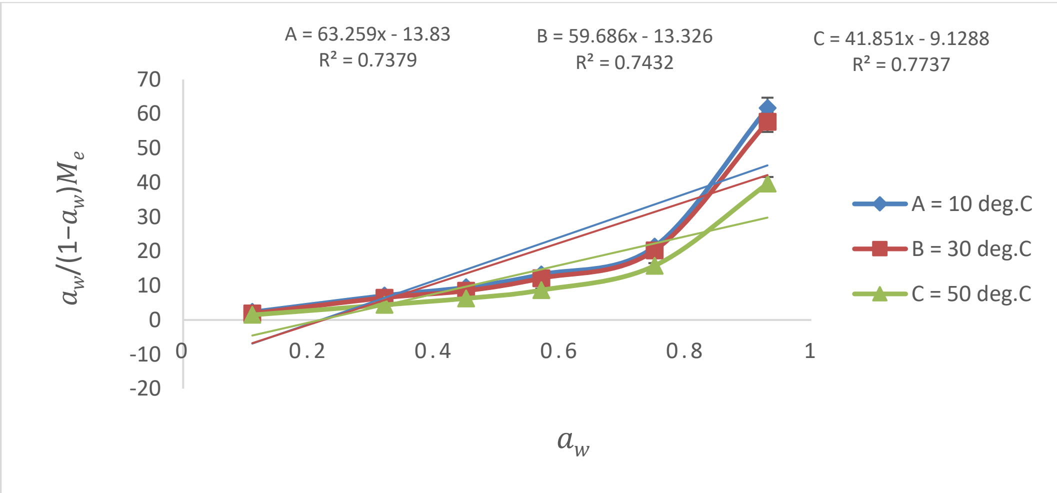

Fig 3. Sorption isotherm of stored *Pupuru* flour based on BET model

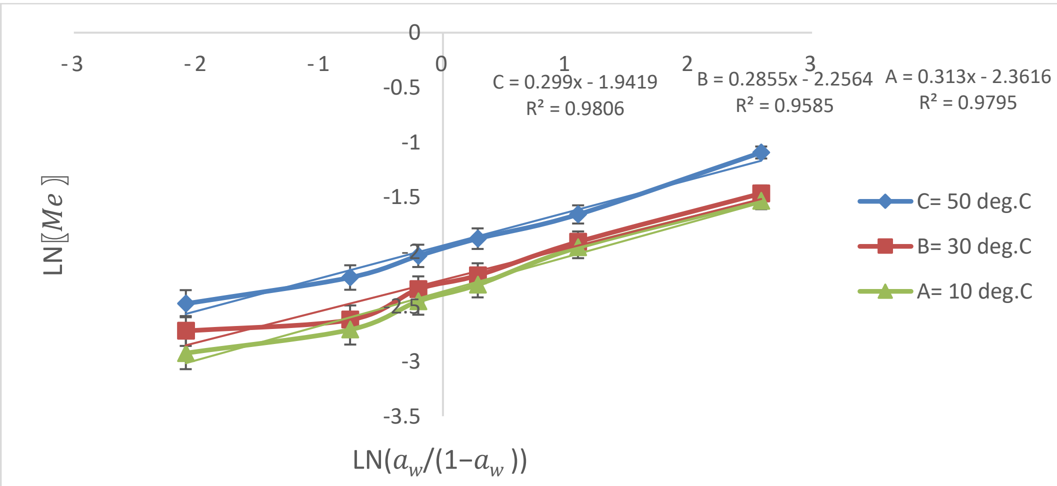

Fig 4. Sorption isotherm of stored *Pupuru* flour based on Oswin model

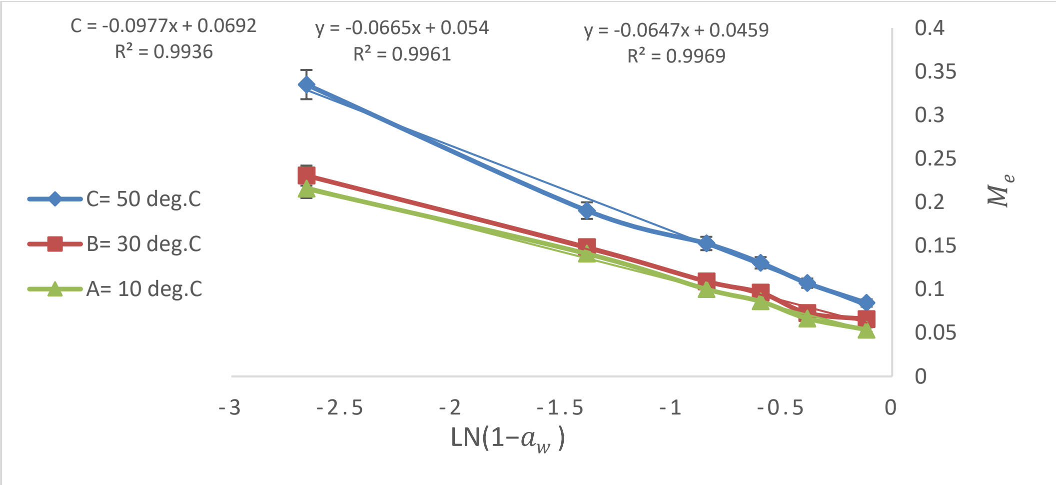

Fig 5. Sorption isotherm of stored *Pupuru* flour based on Smith model

The parameters of the sorption modelling of the *pupuru* flour at different storage temperatures are shown in Table 2. The magnitudes of the *α*, β and ϒ represent the coefficients of the terms of the models used for the sorption isotherm prediction. The values of the constants *α, β* and *γ* obtained from the curves (Abramovič and Klofutar, 2006) were used to calculate the three model parameters of C, *K* and M_o. The monolayer (M_o) values obtained were temperature dependent, and this agrees with previous studies (Abramovič and Klofutar, 2006; Fadeyibi et al., 2012). The M_o was found to decrease with an increase in the temperature for GAB, BET, and Oswin models. It is important to note that an increase in the temperature usually leads to a decrease in M_o (Arevalo-pinedo et al., 2004) and this was observed for the GAB, BET and Oswin models. Thus, the decrease in the M_o may indicate that the adsorbed molecules will require less kinetic energy to break away from their sorption sites. The values obtained were consistent with other similar unenriched cassava products such as *fufu* (0.043–0.049), tapioca (0.049– 0.058) and unenriched *gari* (0.057–0.060) (Kuye and Sanni, 2002; Oluwamukomi et al., 2008). The M_o was less than 0.1 kgkg^{-1} (db) in all the samples, which was the maximum value reported for the most food materials (Tejada-Ortigoza et al., 2017; Oluwamukomi et al., 2008; Kaymak- Ertekin and Sultanoglu, 2001). These values agree with literature values for other starchy products (Kuye and Sanni, 2002; Oluwamukomi et al., 2008). On the contrary, the M_o estimated from the Smith model increases with an increase in the temperature, and this may require high energy to process the product. Furthermore, the parameter *C* was found to increase with an increase in the storage temperature for the GAB model, but decrease for the BET model. However, the parameter *K* decreases with an increase in the temperature for the GAB and the Smith models, but remains inconsistent for the Oswin model. The decrease in the *K* values may indicate that the multilayer molecules of the *pupuru* flour may require less entropy for processing at higher temperature. This will usually result in the molecules requiring less kinetic energy to cause them to pull apart (Abramovič and Klofutar, 2006; Oluwamukomi et al., 2008). Thus, the GAB, BET and Oswin models are better estimators of the M_o, while the parameters *C* and *K* were better estimated using the GAB model.

Table 2. Parameters of sorption modelling of *Pupuru* flour at different storage temperatures

	Sorption Isotherm Model											
	GAB			BET			Oswin			Smith		
	Temperature (°C)			Temperature (°C)			Temperature (°C)			Temperature (°C)		
MP	10	30	50	10	30	50	10	30	50	10	30	50
α	-14.3	-13.5	-9.93	63.26	59.69	41.85	-2.36	-2.256	-1.942	-0.067	-0.067	-0.098
β	17.5	16.7	12.29	-13.83	-13.3	-9.13	0.313	0.286	0.299	0.046	0.054	0.069
ϒ	0.41	0.10	0.044	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
M_o	0.06	0.05	0.041	0.0202	0.021	0.020	0.313	0.286	0.279	0.046	0.054	0.069
C	55.3	219.4	348.7	-3.574	-3.68	-5.54	0.000	0.000	0.000	0.000	0.000	0.000
K	-19.0	-23.3	-26.4	0.000	0.00	0.000	-2.36	-2.256	-1.942	-0.067	-0.067	-0.098

k, c, are model constants; MP is model parameter; M_o *is monolayer moisture content;* α, β and ϒ are regression coefficients

Validation of Sorption Data

The results of the model verification and caliberation are shown in Table 3. The sorption data adequately fit the GAB and Smith models followed by the Oswin model and the least for the BET model. This is evident from the differences in the values of the R^2 and R_{adj}^2 of the regression equations obtained from the analysis of the 4 models. This means that the GAB, BET and Smith models can be used for describing the sorption behaviour of the *Pupuru* flour over the entire range of the a_w. The Oswin model may not be good for predicting the sorption isotherm of the *pupuru* flour because of its lower R^2 and R_{adj}^2 values relative to the other models tested. Also, the relationship between the actual and model predicted equilibrium moisture contents of the product is shown in Fig. 6. The predicted values closely match the actual values of the equilibrium moisture content of the product. All the models are adequate and valid for independent prediction of the equilibrium moisture content since the MSE values are generally less than 10% at the storage temperatures considered. This is consistent with the findings of Oluwamukomi et al. (2008) for unenriched *gari* product and Kuye and Sanni (2002) for *lafun* food product. Thus, the model can be used for predicting the sorption isotherm behaviour of the *pupuru* flour within its range of a_w.

Table 3. Verification data of the sorption models for *pupuru* flour at different temperatures

	Sorption Isotherm Model											
	GAB			BET			Oswin			Smith		
	Temperature (°C)			Temperature (°C)			Temperature (°C)			Temperature (°C)		
	10	30	50	10	30	50	10	30	50	10	30	50
R^2	0.985	0.98	0.977	0.738	0.743	0.774	0.980	0.959	0.981	0.997	0.996	0.994
R_{adj}^2	0.981	0.975	0.971	0.673	0.679	0.718	0.975	0.949	0.976	0.996	0.995	0.993
MSE	0.011	0.007	0.002	0.006	0.005	0.005	0.005	0.004	0.001	0.004	0.003	0.006

MSE is mean square error, R^2 is coefficient of determination and R_{adj}^2 adjusted coefficient of determination

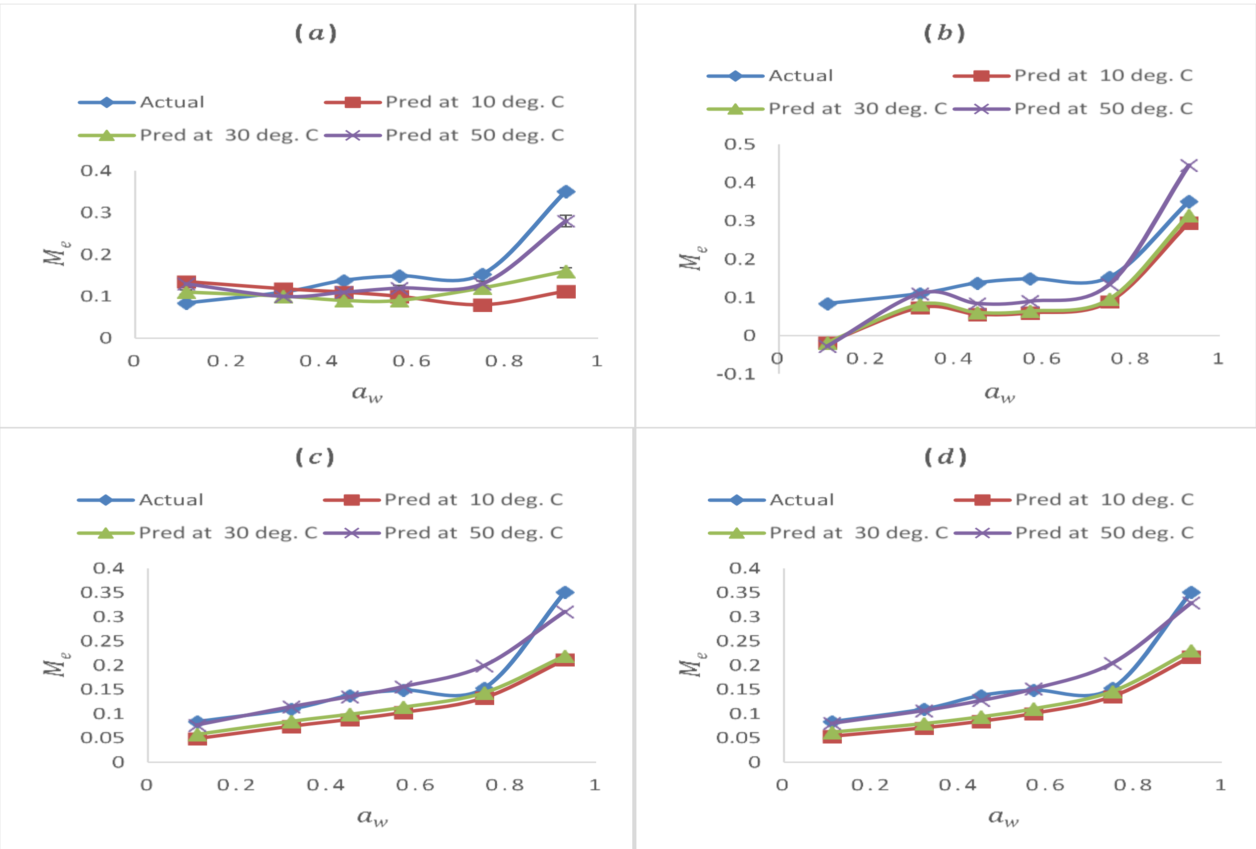

Fig 6. Relationship between the actual and model predicted equilibrium moisture content of stored *pupuru* flour based on (a) GAB model (b) BET model (c) Oswin model (d) Smith model

Conclusion

This study was carried out to describe the sorption isotherm behaviour of the *pupuru* flour at different temperatures and water activity conditions. The sorption data were fitted into the GAB, BET, Oswin and Smith models for starchy foods. The model parameters were found to vary with an increase in the storage temperarture and the a_w of the product. The sorption parameters (M_o, C and K) were generally higher for the products stored at 10 °C and 0.10 a_w. The M_o was found to decrease with an increase in the temperature for GAB, BET, and Oswin models. On the contrary, the M_o estimated from the Smith model increases with an increase in the temperature, and this may mean high energy is required to process the product. The parameter C was found to increase with an increase in the storage temperature for the GAB model, but decrease for the BET model. However, the parameter K decreases with an increase in the temperature for the GAB and the Smith models, but remains inconsistent for the Oswin model. The high values of the R^2 and R^2_{adj}, and the MSE< 10% indicate the ability of the models to predict the sorption isortherm characteristics of the *pupuru* flour within the range of a_w and the storage temperatures considered. The GAB, BET and Oswin models are better estimators of the M_o, while the parameters C and K were better estimated using the GAB model.

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