

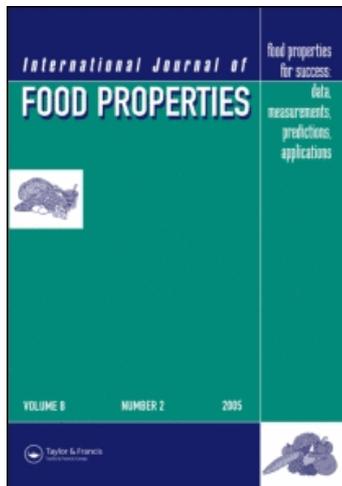
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## WATER SORPTION ISOTHERMS AND CRISPNESS OF FRIED YAM CHIPS IN THE TEMPERATURE RANGE FROM 293K TO 313K

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*Water sorption isotherms of fried yam chips were determined using a static gravimetric method with saturated salt solutions in the range of water activity between 0.22 and 0.85 at 293, 303 and 313K. Four sorption models namely GAB, Peleg, modified Mizrahi, and BET were fitted with the sorption data generated. The GAB model followed by Peleg and modified Mizrahi models were found to best represent the experimental data in the  $a_w$  range of 0.22–0.85. However, the BET model was more applicable between  $a_w$  range of 0.22–0.55. The adsorption isotherm of fried yam chips clearly showed the influence of temperature, decreasing the moisture content at a fixed water activity value with higher temperature. The net isosteric heats of sorption of water were estimated by applying the Clausius–clapeyron equation to the adsorption isotherms at different temperatures. The net isosteric heat of sorption was observed to be decreasing as moisture content increases. Samples stored in desiccators of 0.44 and 0.55  $a_w$  at 303 and 313K, respectively, were rated higher in terms of textural properties investigated.*

**Keywords:** *Fried yam chips, Equilibrium moisture content, Heat of sorption, Adsorption isotherm, Mathematical models, crispness.*

## INTRODUCTION

Frying, resulting in dehydration, is an established process of food preparation worldwide<sup>[1]</sup> and has long been a means of food preparation for achieving desired texture and flavour attributes of a variety of products.<sup>[2]</sup> Fried yam chips are prepared by frying yam slices at a high temperature,<sup>[3]</sup> which combines heat treatment at high moisture with dehydration and results in gelatinization of starch, softening of tuber tissues and at least a

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partial inactivation of enzymes.<sup>[4]</sup> Dehydration during frying leads to formation of a dry crust with its typical porous structure and crisp texture, and there is good evidence that this rapid drying is critical for ensuring favourable structural and textural properties of food products.<sup>[5]</sup> Dehydration in hot oil is characterized by very high drying rates.<sup>[4]</sup>

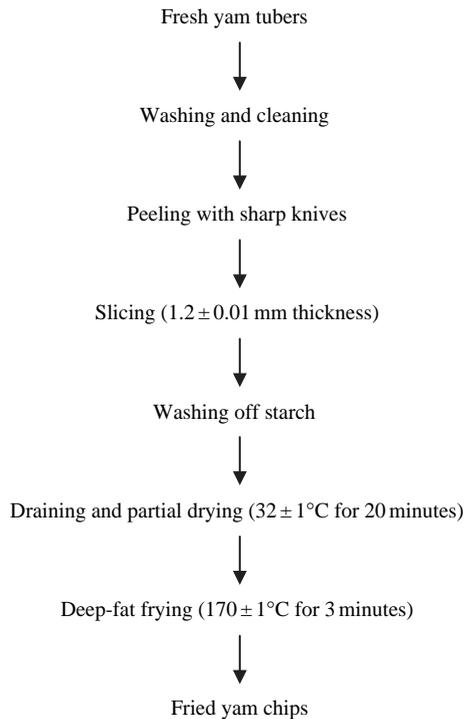
The graphical relationship between the amount of water adsorbed and the corresponding environmental humidity, expressed as water activity at constant temperature is called a sorption isotherm.<sup>[6]</sup> The relationship between water activity and equilibrium moisture content is fundamental to the design of dehydration operations (optimal moisture content and energy requirements) and also, to determine suitable storage and packaging conditions (to improve several characteristics such as texture or stability related to the quality)<sup>[7]</sup> of fried food material. Moisture sorption isotherms are useful thermodynamic tools for determining the interactions between water and the products dry surface and provide information useful for assessing food processing operation such as frying (drying), mixing, and storage.<sup>[8]</sup> Sorption isotherms can also be used to investigate structural features of a food product such as specific surface area and pore size distribution. Such data can be used for selecting appropriate storage conditions and packaging systems that optimize retention of aroma, flavour, colour, texture, nutrients and biological stability.<sup>[9]</sup>

It is well known that microbial and physiochemical stability of foods strongly depends on water activity ( $a_w$ ) of the food system<sup>[10]</sup> hence, it is important to determine the optimal storage conditions of fried yam chips in order to ensure its availability all year round while still retaining its quality attributes. A common procedure used to preserve the quality of a food material is reducing the water activity ( $a_w$ ) in order to inhibit microbial growth<sup>[11]</sup> and maintain crispness. Several models are available in the literature for predicting sorption isotherms of food materials. These include GAB, BET, Peleg, Modified Mizrahi, Kuhn, Oswin, Smith among others used for some agricultural products and foods like intermediate moisture meat products,<sup>[12]</sup> peanuts,<sup>[13]</sup> sweet potatoes,<sup>[14]</sup> lafun and soy flour,<sup>[15]</sup> fufu and tapioca,<sup>[16]</sup> cassava shrimp chips,<sup>[17]</sup> and cassava-based products.<sup>[18]</sup> However, applications of these models to predict sorption isotherms of fried yam chips have not been presented in the literature. Taking into account food materials with similar chemical composition but different physical characteristics may give different sorption isotherms; hence it is necessary to obtain experimental data for fried yam chips as well as fit these data to available mathematical models. Hence, the aims of the present study were to: (i) determine the adsorption isotherms at different temperatures; fit the data to mathematical models; correlate it with experimental data; (ii) evaluate the heats of sorption, which is a basic parameter for energy requirement determination; and (iii) examine the textural properties of fried yam chips during sorption study.

## MATERIALS AND METHODS

### Product Preparation Used for Adsorption Experiments

Fried yam chips were produced under laboratory condition as depicted in Figure 1 by modifying the method of.<sup>[3]</sup> Fresh yam tubers (*Dioscorea rotundata*), purchased from a local market were sorted and then washed to remove sand, dirt, and other adhering materials. The cleaned tubers were peeled using sharp knives and then sliced using a kitchen slicer (ART No: SF 923-1) to obtain a slice thickness of about  $1.2 \pm 0.01$ mm. This method was modified by washing off the starch surface followed by partial drying at  $32 \pm 1^\circ\text{C}$  for 20 min. Samples were then fried in a deep-fat fryer (Model DF5T, China) at  $170 \pm 1^\circ\text{C}$  for 3 min.



**Figure 1** Production of fried yam chips. Source: Modified from Enwere.<sup>[3]</sup>

### Experimental Procedure

The fried yam chips were immediately transferred into a glass desiccator containing phosphorus pentoxide ( $P_2O_5$ ) for about 3 days. The equilibrium isotherms of the samples fried at  $170 \pm 1^\circ C$  were determined by the static gravimetric method at 293, 303, and 313K in ventilated incubators (Model SG 93/06/369). Triplicates of the samples of known weight (about 2 g) were placed above saturated salt solutions (analytical grade, Merck),  $CH_3COOK$ : 0.220;  $MgCl_2$ : 0.330;  $Ca(NO_3)_2$ : 0.440;  $Mg(NO_3)_2$ : 0.550;  $NaBr$ : 0.591;  $CuCl_2$ : 0.680;  $NaCl$ : 0.755 and  $KCl$ : 0.845<sup>[19,20]</sup> with  $a_w$  in the range (0.220–0.845) in separate tightly closed glass jars of 12 cm diameter for 15–20 days until constant weight was reached. Samples weights were determined using a Mettler AJ150 balance. Crystalline thymol was placed inside the high relative humidity (> 65%) desiccators to protect samples from microbial spoilage.<sup>[11,21]</sup> The final moisture content at equilibration was determined by drying at  $105^\circ C$  for 8 h.<sup>[22]</sup> All moisture content was expressed as a percentage of non-fat dry weight because fat is known to exhibit no sorption of water below RH 90%.<sup>[23]</sup> The initial moisture content of the fried samples was found to be  $2.6 \pm 0.01\%$  (wb). The equilibrium moisture content at each water activity is the mean value of three replications.

### Mathematical Modeling

In order to solve a number of processing and /or storage problems using sorption isotherm data with computer techniques, there is a need to fit sorption data to mathematical models.<sup>[10]</sup> Such problems include: prediction of drying times to reach a desired  $a_w$

level, prediction of shelf-life of processed products under specified storage conditions, prediction of minimum packaging requirements and equilibrium product characteristics. In addition to such practical consideration, certain theoretical isotherm equations are also needed for evaluation of important thermodynamic parameters associated with water adsorption in heats, i.e., heat of sorption and monolayer moisture content.

A research of the literature showed that moisture sorption isotherms of food materials can be described by more than one sorption model.<sup>[24]</sup> The BET, GAB, Peleg and modified Mizrahi models are widely used for modeling of the sorption isotherms of food. Therefore, the four models, eqs. (1–4) were chosen to fit the experimental sorption data and the parameters of the models were estimated from the experimental results using the non-linear regression analysis of data fit version 6.1,<sup>[25]</sup> which minimizes the residual sum of squares.

GAB:<sup>[26]</sup>

$$M = \frac{M_o C K a_w}{[(1 - K a_w)(1 - k a_w + C K a_w)]} \quad (1)$$

BET:<sup>[27]</sup>

$$M = \frac{M_o C a_w}{[(1 - a_w) + (C - 1)(1 - a_w)a_w]} \quad (2)$$

Peleg:<sup>[28]</sup>

$$M = a(a_w)^b + c(a_w)^d \quad (3)$$

Modified Mizrahi:<sup>[29]</sup>

$$M = \frac{a + b a_w + c a_w^2}{(a_w - 1)} \quad (4)$$

where C, K, and a-d, are constants in sorption models,  $a_w$  water activity, M the equilibrium moisture content (dry basis), and  $M_o$  is the monolayer value (dry basis). The goodness of fit was determined using the coefficient of determination ( $R^2$ ), the average residual (A), percent average relative deviation (P %), and standard deviation (S).<sup>[30]</sup> Values of P below 10% are indications of a good fit.<sup>[28]</sup>

### Heat of Sorption

The location of the dynamic equilibrium between water vapour and the adsorb amount of water is affected by temperature. This influence was obtained at the investigated temperatures using the theoretical Clausius-Clapeyron Eq. (5) which relates temperature dependence to the water activity at constant water content.<sup>[31]</sup>

$$\ln \left( \frac{a_{w1}}{a_{w2}} \right) = \frac{-Q_s}{R} \left( \frac{1}{T_2} - \frac{1}{T_1} \right), \quad (5)$$

where,  $Q_s$  (kJ/mol) is the net isosteric heat of sorption;  $R$  is the gas constant (8.314 kJ/Kmol);  $T_2$  and  $T_1$  are absolute temperatures at  $a_{w2}$ ; and  $a_{w1}$ , respectively.

An empirical function describing the relationship between  $Q_s$  and the equilibrium moisture content was used:<sup>[32]</sup>

$$Q_s = Q_o \exp\left(\frac{-M_w}{M_o}\right), \quad (6)$$

where  $M_o$  is the inverse of the gradient produced by the linear fit of  $\ln(Q_s)$  vs.  $M_w$ . The slope indicates the change in binding energy with changes in water content, and  $Q_o$  is the isosteric heat of sorption for the strongest bound water molecules.

### Textural Analysis of Fried Yam Chips under Sorption Studies

The profiling method based on quantitative descriptive analysis was used. Ten trained testers were asked to rate by number the following descriptors of texture:<sup>[33]</sup> crispness intensity (0 = no crispness, flexible; and 100 = very crispy, breaking easily when bitten); crispness acceptance (0 = unacceptable; 100 = highly acceptable); hardness (0 = delicate or soft for samples lacking crispness; 100 = very hard); and texture acceptance (0 = unacceptable texture; 100 = highly acceptable texture). An unstructured linear scale was used that covered the range 0 to 100 points, described at both ends as shown above.<sup>[34]</sup> For each session, each tester judges eight samples with two each for samples kept at  $a_w$  between 0.22–0.55 at a particular temperature. Testing results were converted to numbers assuming the total scale to be 5 points.

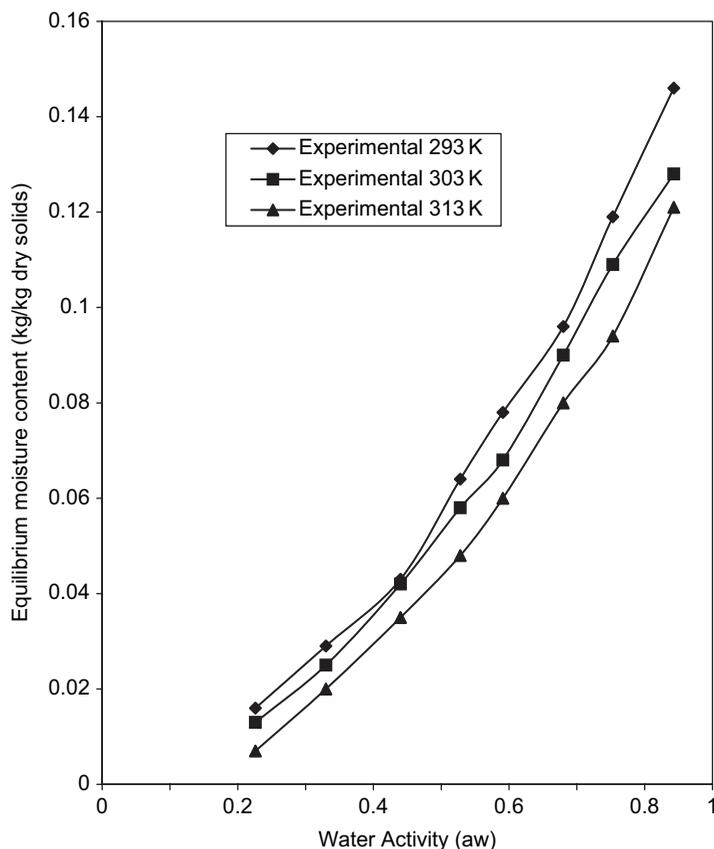
### Breaking Force Determination

The breaking force of the chips was determined using a universal testing machine (Model–M500, Testometric AX, Rochdale, England), equipped with a 50 kN load cell. Fried yam chips of uniform sizes were selected and then placed on a metal support with jaws at a distance of about 25 mm. They were pressed in the middle with a cylindrical flat-end plunger (70 mm diameter) at a speed of 2.5 mm/min. The measurement was recorded by a computer connected directly to the equipment. The breaking force (N) interpreted as chips hardness was obtained as the peak force from the force-deformation curve.<sup>[33]</sup>

## RESULTS AND DISCUSSIONS

### Effect of Temperature on Adsorption Isotherm

The moisture content of fresh yam slices was found to be  $75 \pm 2\%$  (wb), while the moisture content of fried chips was determined to be  $2.6 \pm 0.01\%$  (wb). The experimental adsorption isotherm of the samples at 293, 303, and 313K are shown in Figure 2. Similar trend showing effects of temperature have been reported for high starchy food products. These include cassava and cassava products like lafun;<sup>[15]</sup> fufu and tapioca;<sup>[16]</sup> cassava products;<sup>[17,18]</sup> potato;<sup>[35]</sup> and sweet potato slices.<sup>[14]</sup> From Figure 2, a significant temperature effect on the adsorption for the full range of  $a_w$  was observed for the product similar to the observations of some researchers.<sup>[36,37,11,21]</sup> From Figure 2, higher storage temperature



**Figure 2** Experimental equilibrium data of sorption isotherms for fried yam chips at different temperature.

resulted in progressively higher  $a_w$  values for a particular EMC. The practical implications of this phenomenon are especially important in the higher  $a_w$  range. For an EMC of 0.1 kg/kg dry solids;  $a_w$  shifts between 0.70 and 0.78 as storage temperature increased between 293 and 313K. As a result, an increased storage temperature could be detrimental to the microbial stability and sensory attributes of the fried yam chips. On the other hand, lower storage temperature results in lower  $a_w$  values for a given EMC thus improving stability of the products.

If  $a_w$  is kept constant, an increase in temperature causes a decrease in the amount of sorbed water. As earlier reported by<sup>[38]</sup> this is necessitated by the thermodynamic relationship,  $\Delta F = \Delta H - T\Delta S$  where  $\Delta F$ ,  $\Delta H$ , and  $\Delta S$  are the changes in free energy, enthalpy, and entropy (in J/kmol), respectively, and  $T$  the absolute temperature (K). Since  $\Delta F < 0$  (sorption is a spontaneous process) and  $\Delta S < 0$  (the sorbed water molecule has less freedom), hence  $\Delta H < 0$ . Therefore, an increase in temperature represents a condition unfavorable to water adsorption. Temperature shift can have an important practical effect on chemical and microbiological reactivity related to quality deterioration of a food material.<sup>[12]</sup> However, at lower temperature of storage, the product may become less dried to preserve it but most food materials are considered as dry foods when  $a_w < 0.6$ .<sup>[36]</sup> The quantity of sugars present in food materials plays a role in whether or not crossing of isotherms with temperature at

high water activities will take place.<sup>[39]</sup> From Figure 2, no crossing over was observed which may be attributed to very low sugar content of yam tubers (0.02–0.5%).<sup>[3]</sup> A similar result was observed for low sugar apples.<sup>[40]</sup>

### Fitting of Sorption Models to Experimental Data

The parameters for the sorption models for fried yam chips with the statistical results using a non linear regression package data fit version 6.1<sup>[25]</sup> are as shown on Table 1. A close examination of the results on Table 1 indicates that the GAB, Peleg, and Modified Mizrahi models adequately describes the experimental adsorption data for fried yam chips throughout the entire water activity range due to higher  $R^2$ . However, since  $R^2$  alone is not a reliable criterion for evaluation of EMC/ERH models, the P (%) values are a necessity.<sup>[28]</sup> Based on this the GAB and Peleg models could be said to best describe the sorption data of fried yam chips with the former having P values ranging from 0.40723% to 2.99689% and an average value of 1.2797%; compared with the latter having P values between 0.06784% to 3.1767% with an average of 1.28%. The mean relative percent deviation (P %) of these two models was observed to be less than 10% within the temperature of study (293 to 313K). Hence, the GAB model could be said to satisfactorily predict the equilibrium moisture content of fried yam chips. Similar results have been reported for

**Table 1** Model parameter and statistical data of water sorption isotherms of fried yam chips at different temperature.

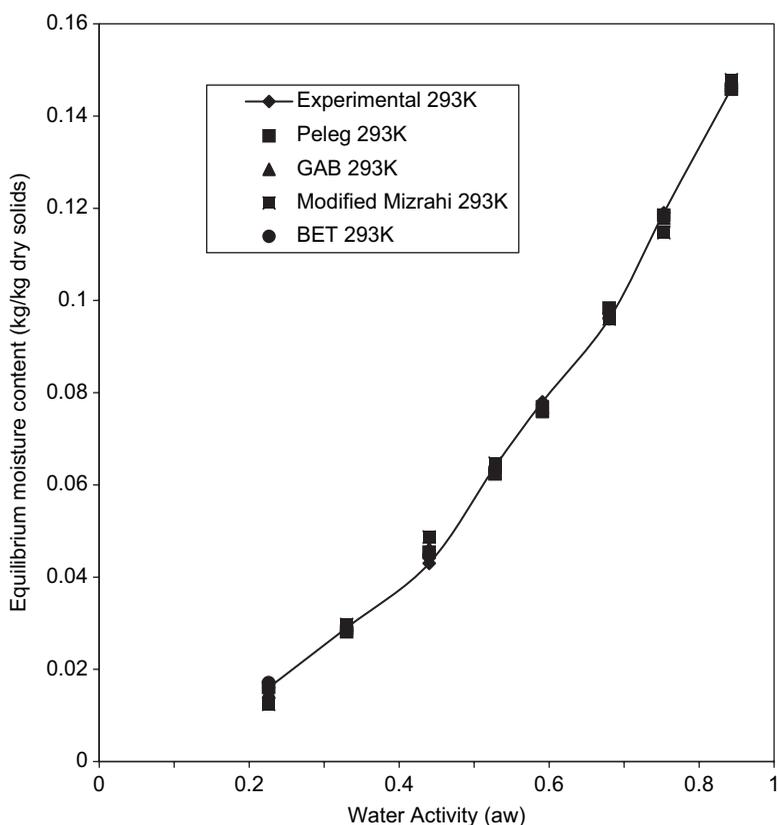
Model	Temperature (K)		
	293	303	313
<b>GAB</b>			
$M_0$	0.00921	0.08424	0.01037
C	1.00631	1.00611	1.00592
K	10.42021	7.58629	6.30553
P (%)	2.99689	0.43523	0.40723
$R^2$	0.99786	0.99819	0.99932
<b>Peleg</b>			
a	0.19653	0.08723	0.08373
b	1.90045	1.73786	1.94978
c	0.00371	0.08723	0.08373
d	-0.12579	1.73785	1.94979
P (%)	0.06784	0.59630	3.17670
$R^2$	0.99782	0.99793	0.99862
<b>Modified Mizrahi</b>			
a	-0.02392	0.02600	-0.02573
b	0.15835	0.15589	0.13554
c	-0.00846	-0.00549	-0.00736
P (%)	1.16714	0.19152	2.00090
$R^2$	0.99514	0.99435	0.99810
<b>BET</b>			
$M_0$	0.05445	0.04984	0.04044
C	1.00630	1.00601	1.00592
P (%)	1.67030	5.42079	21.12320
$R^2$	0.99569	0.99157	0.95773

BET is between 0.22–0.55 $a_w$ .

other starchy products like cassava—shrimp chips,<sup>[17]</sup> sweet potato slices,<sup>[14]</sup> fufu, tapioca, and lafun.<sup>[15,16]</sup>

The table also shows that the BET model gave a good fit for temperatures between 293 to 303K for  $a_w$  between 0.22 to 0.55. Since the P value within 0.22 to 0.55  $a_w$  at 313K is greater than 10% despite having high  $R^2$  (0.95773), it has a poor fit under this condition. However, the BET model is known to hold for  $a_w$  up to about 0.5.<sup>[38]</sup> Since the applicability of the BET model to fried yam chips is limited, it has to be admitted that prediction of data will be simple and reliable if a single equation fits the entire span of  $a_w$ .<sup>[28]</sup> Figures 3 to 5, shows the plots of experimental and predicted using the four models tested. The closeness of the experimental and predicted data obtained by GAB model confirms the suitability of this model in predicting the sorption isotherms of fried yam chips under the condition of investigation.

It can also be seen from the table that the monolayer value ( $M_o$ ) which is a measure of sorption capability of the material obtained by GAB model increases as temperature increases from 293 to 303K but falls when temperature gets to 313K. However, that of BET decreases as temperature increases from 293 to 313K within the  $a_w$  range of 0.22 to 0.55. These values ( $M_o$ ) obtained for this study was observed to be less than 0.1kg/kg dry basis which was the maximum value earlier reported for food materials.<sup>[26,36]</sup>



**Figure 3** Experimental equilibrium data and predicted sorption isotherms for fried yam chips at 293K.

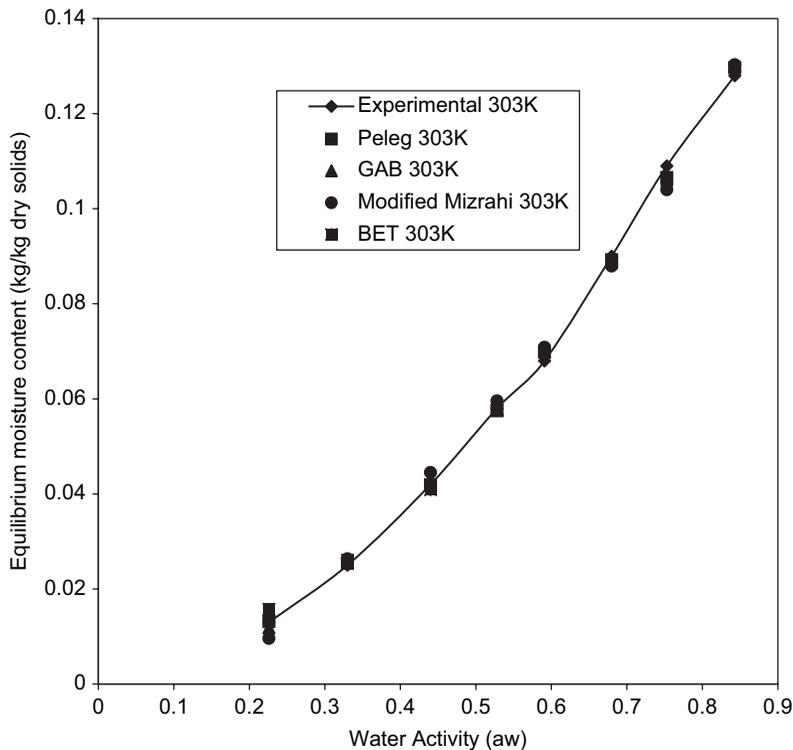
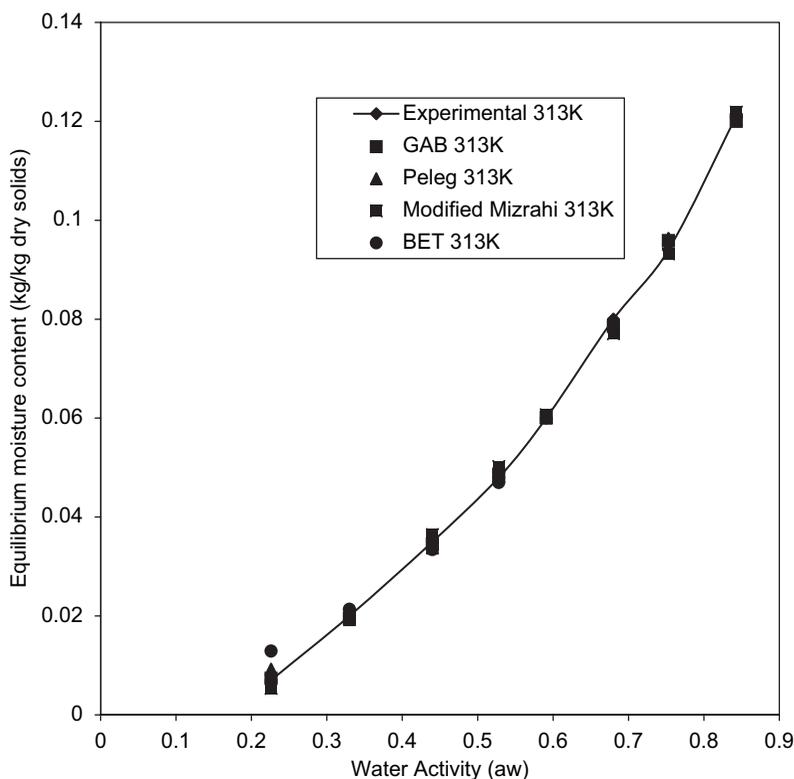


Figure 4 Experimental equilibrium data and predicted sorption isotherms for fried yam chips at 303K.

### Isosteric Heat of Sorption

The net isosteric heats of sorption or differential enthalpy shows the energy requirement for removing moisture from food material (water-solid binding strength) and has a practical use in complete drying calculations and modeling of energy.<sup>[41]</sup> Figure 6 shows the plots of  $\log a_w$  vs the reciprocal of the absolute temperature of fried yam chips at different moisture contents. The slope of the line ( $Q_{st}$  - heat of sorption) decreases as moisture content increases indicating a decrease in the binding energy for water molecules. From Figure 6, it was also observed that the calculated heats of sorption (slope of the graph), decreases with increasing temperature. This can be explained by the higher mobility and energy of water molecules with increasing temperature, with the energetic difference between sorption and no sorption being reduced.<sup>[36]</sup>

If  $Q_{st}$  for a particular food were known, it would be possible to predict the  $a_w$  of that food at any temperature. Since however, standard tables for  $Q_{st}$  for different foods do not exist, prediction of  $a_w$  requires the determination of moisture isotherms for at least two temperatures. In Figure 7, the calculated heats of sorption were plotted as a function of water content. At low moisture content, the heat of sorption is high indicating the highest binding energy for removal of water. These results indicate the high interaction energy between the water molecules and food matrix at low moisture content. These interactions reduces, lowering the heat of sorption, as the moisture behaves as free water. As may be expected, when starting with an absolutely dry material like fried yam chips, water first adsorbs preferentially on the most active sites with great interaction energy, and as these



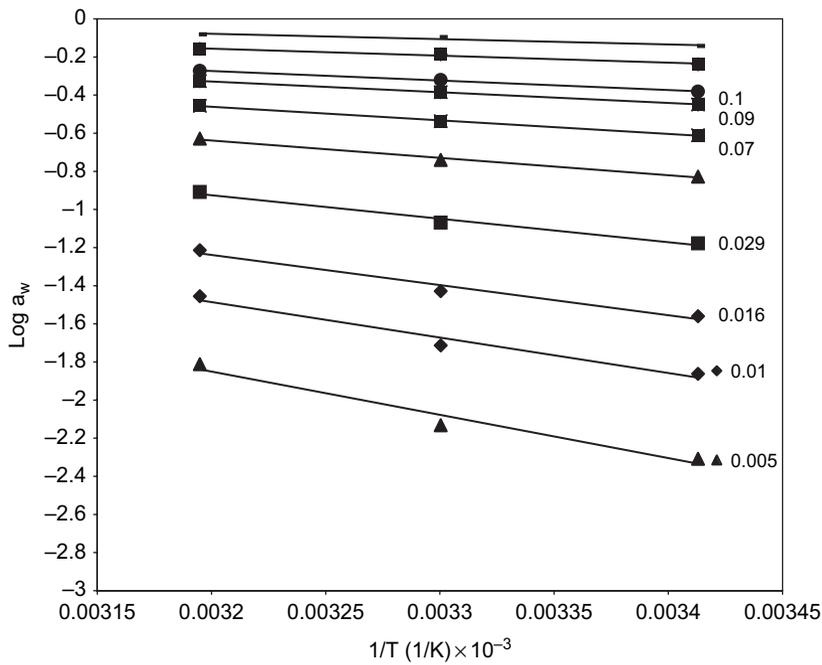
**Figure 5** Experimental equilibrium and predicted sorption isotherms for fried yam chips at 313K.

sites become predominantly occupied, further adsorption will take place on less active site with lower heats of adsorption. With increasing amounts of adsorbed water, the specific adsorption enthalpy decreases and attains ultimately the level of condensation of pure water.<sup>[23]</sup> A similar trend was observed by<sup>[11,16,21,41]</sup> in their work.

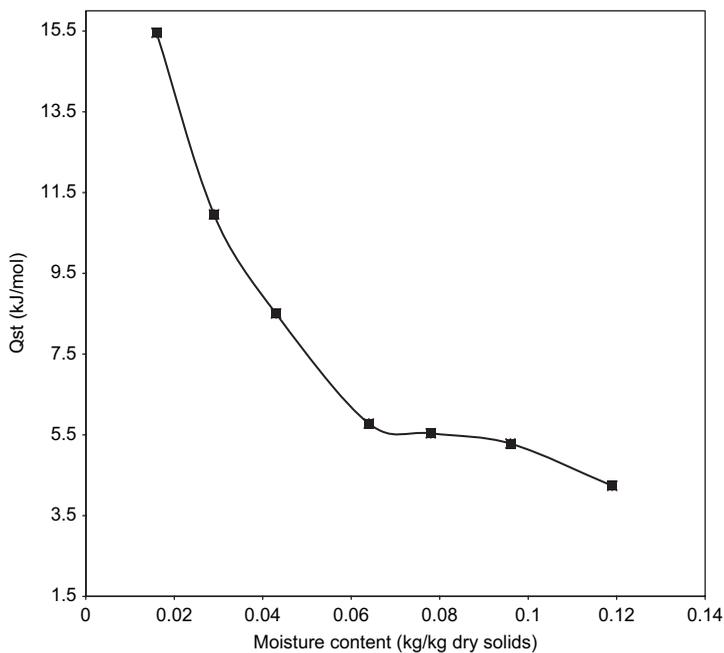
### Changes of Textural Properties

Figure 8 shows the results of sensory test of fried yam chips at various water activities and temperatures. Expectedly, the  $a_w$  affected texture (ANOVA not shown). From the figure, it was observed that samples under  $a_w$  0.44 at 313K and  $a_w$  0.55 at 303K were assessed similarly and significant differences between these samples were not observed. These chips were highly acceptable in terms of the textural properties investigated. However, samples kept in the  $a_w$  0.22 at various temperatures were judged significantly lower by the panelists. Similar results have been reported for apple chips.<sup>[34]</sup> From this study, we concluded that temperature seems to have a significant effect on textural properties. We expected that samples at lower  $a_w$  range will be more acceptable but this was not the case. Hence, it seems that storing fried yam chips at  $a_w$  range of 0.44–0.55 at temperatures 293K to 303K gave samples of high textural acceptance.

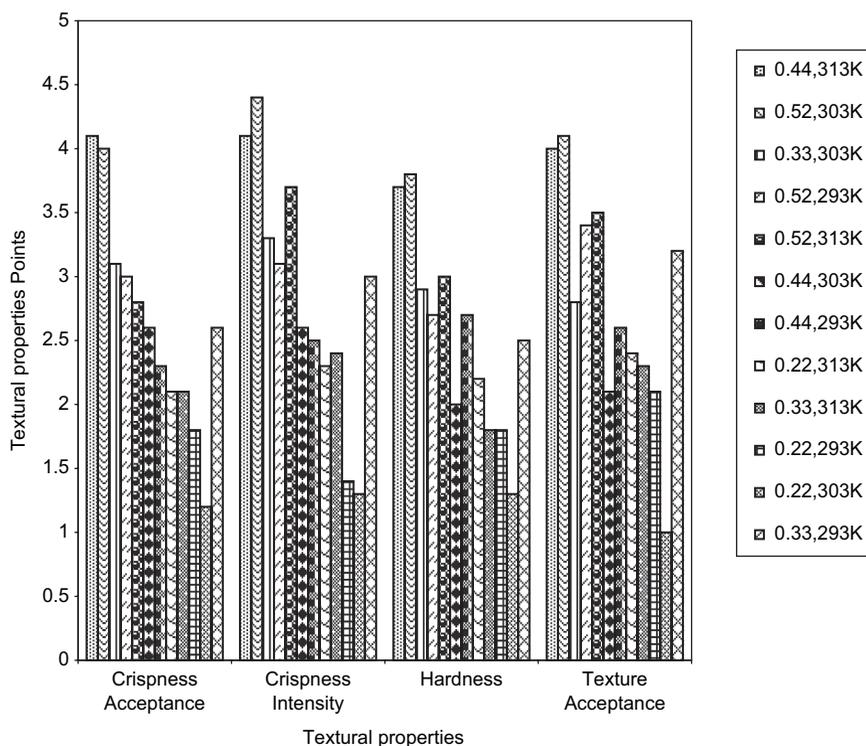
The breaking force which was interpreted as chips hardness also confirms sensory results as shown in Table 2. It was observed that the breaking force, i.e., peak force for



**Figure 6** Plots of  $\text{Log}_e a_w$  Vs the reciprocal of the absolute temperature of fried yam chips at various moisture contents (g H<sub>2</sub>O/100g solids).



**Figure 7** Isotheric heat of sorption of fried yam chips as a function of moisture content.



**Figure 8** Effect of water activity and temperature on textural properties of fried yam chips.

**Table 2** Mean values of breaking force of fried yam chips at different water activity and temperature of sorption study.

Water activity, temperature (K)	Breaking force (N)
0.44, 313	578.3 <sup>a</sup>
0.52, 303	593.5 <sup>a</sup>
0.33, 303	692.0 <sup>b</sup>
0.52, 293	687.3 <sup>b</sup>
0.52, 313	709.7 <sup>bc</sup>
0.44, 303	710.0 <sup>bc</sup>
0.44, 293	787.4 <sup>cd</sup>
0.33, 313	1347.9 <sup>e</sup>
0.33, 293	1324.8 <sup>e</sup>
0.22, 313	1693.5 <sup>f</sup>
0.22, 303	1676.3 <sup>f</sup>
0.22, 293	1702.9 <sup>g</sup>

Any two means not followed by the same letter vertically are significantly different ( $p < 0.05$ ).

rupture of chips in 0.44<sub>a<sub>w</sub></sub> at 313K has the lowest value followed closely by samples kept in 0.52<sub>a<sub>w</sub></sub> at 303K. Significant differences occurs between values for other conditions with samples kept in 0.22<sub>a<sub>w</sub></sub> at temperatures between 293K and 313k having the highest breaking

force hence low acceptance in terms of hardness. Similar results have been reported for carrot chips.<sup>[33]</sup> From the table, it could be observed that the breaking force was significantly affected by temperature of storage and water activity.

## CONCLUSION AND RECOMMENDATIONS

The equilibrium experimental data at 293, 303, and 313K and  $a_w$  range of 0.22 to 0.85 for fried yam chips were successfully fitted to four sorption models available in the literature with the GAB and Peleg showing a good fit for the range of water activities studied. The adsorption isotherm indicates that temperature affects the sorption characteristics of the samples. The EMC decreases with increasing temperature. The net isosteric heat of sorption decreases with increasing moisture content and at lower moisture content, a strong heat of sorption was evaluated. The heat of sorption also decreases with increase in temperature. The lowest breaking force and subsequently better textural properties was observed for samples kept between 0.44–0.55 $a_w$  range at 303 and 313K, while those kept in 0.22  $a_w$  at any temperature were rated poorly.

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