Adsorption isotherms and thermodynamics properties of water yam flour

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Short title: Isotherms of water yam flour
Received: 4 June 2015 / Accepted: 12 June 2016
Research Article

Summary

Adsorption isotherms of water yam flour were determined by static gravimetric method of saturated salt solutions in the range of water activities between 0.11 and 0.96 and at temperatures of 25, 35 and 45 °C. The experimental sorption data was fitted to five models: Guggenheim-Anderson-De Boer (GAB), Peleg, Iglesias-Chirife, Exponential and Oswin and the differential enthalpy and entropies were determined. Equilibrium moisture contents decreased with increase in temperature at all the water activities studied. Peleg model gave the best fit for the sorption isotherm of water yam flour. Monolayer moisture content values estimated from the GAB-model were found to decrease with increases in temperature. The differential enthalpy and entropy decreased with increases in moisture content. The isokinetic temperature was 371.32 K and the compensation theory was satisfied.

Keywords: water yam flour, water activity, differential enthalpy, differential entropy, compensation theory

1. Introduction

Yam (*Dioscorea spp*), an important staple, is cultivated in Nigeria and West Africa (Ekwu *et al.*, 2005). It is an important energy source for many people in Nigeria which, accounted for about 67.7% of world production in 2010 (http://faostat3.fao.org). Water yam (*Dioscorea alata*) is known for its high nutritive value compared to other yam species, with crude protein content of 7.4%, starch content of 75-84%, and vitamin C content ranging from 13.0 to 24.7 mg/100 g (Osagie, 1992). The proximate composition of yam comprises: 65-81% moisture; 1.4-3.5 g protein; 0.2-0.4 g fat; 16.4-31.8 g carbohydrate; 0.40-10.0 g fibre; and 0.6-1.7 g ash (Osunde, 2008). However, water yam is underutilised with little or no industrial applications. Also, its loose watery texture has been a limitation to its use. Hence, there is the need to process water yam tuber into a more shelf stable material it increase its potential for industrial food applications.

Fresh yams deteriorate within 2-3 month during storage (Afoakwa and Sefa-Dedeh, 2001). Olayemi *et al.* (2012) reported postharvest losses of yam in Nigeria to be about 37%. Hence, the need to process this important staple food commodity into product(s) of longer shelf life such as flour. Water yam tubers have been used as traditional food preparations in the home with little industrial applications. However the traditional uses are diverse and the crop has more utilisation potentials. Water yam flour is a novel product from yam, which is produced from wholesome fresh tubers. It should be odourless, crystal white and free from foreign or extraneous materials. It could find wide applications in the baking and confectionery industries. Most importantly, water yam flour can be easily stored for a longer period (12-18 months) compared to the fresh tubers.

Moisture sorption isotherms represent the relationship between water activity and equilibrium moisture content of food products at constant temperature. Knowledge of sorption isotherms is useful in designing drying equipment, appropriate storage and packaging of food products (Ahmed *et al.*, 2005). They are also useful in determining the thermodynamics of moisture sorption in food. Thermodynamic properties of food products provide means for understanding water properties and calculating energy requirements of heat and mass transfer in biological materials (Moreira *et al.*, 2008). Isosteric heat of sorption, an important thermodynamic parameter, is a measure of the binding energy of the forces between water vapour molecules and solid. Differential entropy of food materials is proportional to the number of available sorption sites at specific energy levels. Enthalpy-entropy compensation theory is useful in determining physical and chemical phenomena such as sorption. The theory states that compensation arises from the nature of interaction between solvent and solute and also a linear relationship exist between reaction enthalpy and entropy (Fasina, 2006).

Onayemi and Oluwamukomi (1987) reported moisture sorption isotherms of traditional yam flour (*elubo*) at 25 and 32 °C in the water activity range of 0.13-0.73. Oyelade *et al.* (2008) also reported moisture sorption isotherms of traditional yam flour (made from *Dioscorea rotundata*) at 27, 32, 37 and 40 °C in the water activity range 0.10-0.80. However, there is no published report on the moisture sorption isotherms of water yam flour. The objective of this study was fill that gap by presenting the sorption isotherms of water yam flour, fit the sorption isotherms to selected sorption models and use the best model to determine the thermodynamic properties.

2. Materials and methods

Preparation of water yam flour

Mature freshly harvested and wholesome water yam tubers were purchased from an open market in Osiele, Abeokuta, Ogun state, Nigeria. The yam tubers were cleaned, hand peeled (using a stainless steel knife), washed and sliced into chips (using a vegetable slicer). The yam slices were then pretreated in 0.28% potassium metabisulphite solution for 15 min. The pretreated slices were subsequently drained and dried in a cabinet dryer (DC500; Genlab, Widnes, UK) at 60 °C for 48 h. The dried chips were milled into flour using a laboratory hammer mill fitted with a 250 μm screen (Wahab *et al.*, 2015).

Determination of equilibrium moisture content

The equilibrium moisture content was determined by static gravimetric method (Pawar *et al.*, 1992) at different water activity levels (between 0.11 and 0.96). Nine saturated salt solutions, as shown in Table 1, were employed to maintain constant water activity within the desiccators. The determination of sorption isotherms was carried out at 25, 35 and 45 °C to simulate variation in temperature in different parts of Nigeria. Thymol was placed in desiccators in which water activity was 0.75 and above in order to inhibit microbial growth. Triplicates of 3 g flour samples were placed above saturated salt solutions in desiccators and kept in incubators at desired temperature maintained to an accuracy of ±1 °C. The samples were weighed daily and equilibrium was considered to be attained when three consecutive values were obtained (±0.001 g). The equilibrium was attained within 20-25 days. The equilibrium moisture content was calculated on a dry weight basis.

Table 1. Water activity of saturated sorption salts at selected temperatures.

Sorption salts	Temperature		
	25 °C	35 °C	45 °C
LiCl	0.11	0.11	0.11

CH_3COOK	0.23	0.22	0.21
$CaCl_2$	0.34	0.34	0.34
$Ca(NO_3)_2$	0.46	0.46	0.46
$Mg(NO3)_2$	0.53	0.50	0.47
$NaNO_3$	0.64	0.64	0.64
NaCl	0.75	0.75	0.75
$K_2Cr_2O_7$	0.86	0.86	0.86
K_2SO_4	0.97	0.97	0.96

Sorption isotherm modelling

Many models in the literature are available for predicting sorption isotherms of foods, five of which have been reported to give the best fit for the sorption data for root and tuber flour (Kuye and Ariri, 2005). The five models reported by Kuye and Ariri (2005) were employed in this study. They are: GAB, Peleg, Iglesias-Chirife, Oswin and Exponential (Table 2). The parameters of the models were estimated using non-linear regression procedure of SPSS version 21. The goodness of fit for each of the model was assessed by using statistical parameters such as coefficient of determination (R^2) and mean relative percentage deviation (P%) which is defined as:

(1)

Where, X_{exp} and X_{cal} are, respectively the calculated and experimental values of the equilibrium moisture content and n is the number of experimental values. The GAB model contains three parameters, X_m, C and K which are functions of temperature. M_o is the monolayer moisture content, C is a constant related to heat of sorption of the first layer and K is associated with heat of sorption of the multilayer. The Peleg model contains four parameters-A, B, C, and D, which are constants, while Exponential and Oswin models contain two parameters each, A and B, both also constants. The Iglesias-Chirife model contains three parameters which are $M_{0.5}$ (moisture content at the water activity of 0.5), A and B (both are constants).

Table 2. Sorption isotherm models fitted with experimental data.[1]

Models	Mathematical equation	
Guggenheim, Anderson and De Boer	$m = M_oCKa_w / (1 – Ka_w)(1 – Ka_w + CKa_w)$	Guggenheim, 1966; Anderson, 1946; De Boer, 1953
Peleg	$m = Aa^B_w + Ca^D_w$	Peleg, 1993
Exponential	$\ln m = A + \ln Ba_w$	Lazarides, 1990
Oswin	$\ln m = \ln A + B\ln(a_w/(1 – a_w))$	Oswin, 1946
Iglesias and Chirife	$\ln (m + (m^2 + m_{0.5})^{0.5}) = A + Ba_w$	Iglesias and Chirife, 1978

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Estimation of net isosteric heat of sorption and differential entropy

The net isosteric heat of sorption (Q_{st}) of the water yam flour, at any moisture content, was determined from the experimental sorption data using the Clasius-Clapeyron equation (Tsami, 1991):

(2)

Where, a_w is the water activity, R the ideal gas constant (8.314 J/mol K) and T the absolute temperature (K).

The Q_{st} of the flour was calculated from the slope of the plot of In a_w versus 1/T at constant moisture content. This approach was based on the assumption that: (1) there is no change in heat of vaporisation and excess heat of sorption with temperature; and (2) that moisture content of systems remains constant.

The differential entropy (S_d) of adsorption was calculated from the Gibbs-Helmholtz equation:

(3)

(4)

Where, G is the Gibbs free energy (kJ/mol).

Substituting Equation 4 in Equation 3, we have Equation 5:

(5)

By plotting In a_w against the inverse of temperature, at constant moisture content, the S_d was estimated from intercept (S_d/R).

Enthalpy-entropy compensation theory

The isokinetic relationship or enthalpy-entropy compensation theory proposed that a linear relationship exist between Q_{st} and S_d (Leffer and Grunwald, 1963).

(6)

From the plot of Q_{st} against S_d, the isokinetic temperature (T_1) and the Gibbs free energy (G) at T_1 were calculated using linear regression. T_1 is the temperature at which all reactions in the sorption sites proceed at the same rate.

A test of the compensation theory, as recommended by Krug *et al.* (1976) was carried out, which involves the evaluation of the isokinetic temperature with respect to the harmonic mean temperature T_{hm} defined as shown in Equation 7:

(7)

Where, n is the number of isotherms.

The theory can only be applied if $T_1 \neq T_{hm}$. If $T_1 > T_{hm}$ the process is enthalpy driven, if otherwise, the process is considered to be entropy driven.

3. Results and discussion

Effect of temperature on adsorption isotherms

Figure 1 depicts the sorption isotherms of water yam flour at 25, 35 and 45 °C, respectively. The isotherms indicate that the equilibrium moisture contents (EMC) of water yam flour increased as water activity increased, at constant temperature. The isotherms further indicated that at lower water activities, moisture increased linearly with water activity but at higher water activity levels, the moisture content increased rapidly with water activity for any given temperature. Moreira *et al.* (2008) reported similar trends. The isotherms for the water yam flour exhibited a type II sigmoid curve according to the Brunauer, Emmet and Teller classification (Brunauer *et al.*, 1938) and there was no inversion (crossover) of isotherms at higher water activities.

Figure 1. Sorption isotherms of water yam flour at 25, 35 and 45 °C (EMC = equilibrium moisture contents).

The EMC decreased as storage temperature increased for any given water activity. This implies that at any water activity, the water yam flour could be less hygroscopic as storage temperature increases, i.e. it could be expected to absorb more moisture at lower temperatures than it would at higher temperature. The decrease in EMC as temperature increased could be because at higher temperatures, the activation energy of the water molecules changes to higher energy levels, the bonds become less stable and break away from water-binding sites of the flour (Moreira *et al.*, 2008). Similar trends for the same temperature effects have been reported by Sanni *et al.* (1997), Onayemi and Oluwamukomi (1987) and Adebowale *et al.* (2007) for *fufu*, traditional yam flour (*elubo*) and tapioca grits, respectively. Higher water activity resulted in progressively higher EMC values at constant temperature. At any particular EMC value, lower storage temperature results in lower a_w values, which could be taken into consideration for the storage stability and the prevention of a potential microbial growth.

Fitting of sorption models to experimental data

The coefficients for the sorption models for water yam flour with the statistical parameters are presented in Table 3. From Table 3 it could be deduced that Peleg, GAB and Iglesias-Chirife models adequately described the experimental adsorption isotherms of water yam flour as shown by their higher R^2 values. However, the use of R^2 alone does not imply that that the models fit the experimental data accurately; hence, mean relative percentage deviation P (%) values are therefore necessary to make a conclusive judgment. Based on this, Peleg and GAB could be said to adequately describe the experimental adsorption data of water yam flour, with P values ranging from 0.0335 to 0.0873% and 0.1399 to 0.2197%, respectively. The mean relative percentage deviation (P%) of both models was observed to be below 10%, which is generally accepted to be a good fit (Simal *et al.*, 2007). Hence, Peleg satisfactorily predicts the sorption data of water yam flour.

Table 3. Model and statistical parameters of sorption isotherms of water yam flour.

Model terms	Temperature		
	25 °C	35 °C	45 °C
GAB			
M_o	10.739	9.360	8.309
C	71.347	271.527	3.973×10^6
K	0.703	0.688	0.701
P (%)	0.2197	0.1399	0.1551
R^2	0.992	0.997	0.992
Iglesias and Chirife			
A	-0.189	-0.54	-0.896
B	2.214	2.272	2.524
$M_{0.5}$	-3.466	-3.493	-3.497
P (%)	0.1195	0.0356	0.0635
R^2	0.993	0.997	0.993
Oswin			
A	16.571	4.264×10^6	13.292
B	-0.226	3.631	0.214
P (%)	1.2744	0.6587	0.5458

R^2	0.960	0.962	0.973
Exponential			
A	7.965	9.182	6.807
B	-0.955	3.045	1.324
P (%)	0.6579	0.6186	0.7136
R^2	0.985	0.990	0.974
Peleg			
A	19.434	16.473	15.772
B	3.073	2.766	2.878
C	15.538	12.537	11.2
D	0.194	0.115	0.097
P (%)	0.0873	0.0335	0.0594
R^2	0.994	0.997	0.993

Monolayer moisture content obtained from the GAB model was observed to vary between 8.309-10.73% (db) and it decreased with increases in storage temperature. Similar trends have been reported by Sanni *et al.* (1997) for *fufu* flour and Adebowale *et al.* (2007) for tapioca grits. This trend could be ascribed to the reduction in the number of active sorption sites of the flour materials due to chemical and physical damage caused by increased storage temperature. Monolayer moisture could be described as the sorption capability of the food materials; it is a measure of available active sorption sites for adsorption of water by food materials and as such it is an important parameter for accomplishing minimum quality loss in most foods over a long storage time (Moreira *et al.*, 2008; Quirijns *et al.*, 2005). Therefore, at any given temperature, the safest water activity of food material, in terms of microbiological safety, is that which corresponds to the monolayer moisture content.

A further study of GAB parameters C and K (Table 3) showed that they are temperature dependent. C, a constant defined as the ratio of the partition function of the first molecule adsorbed on a site and the partition function of molecules adsorbed beyond the first molecules, a measure of the strength of binding of water to the primary binding sites, increased with temperature. This indicates that sorption of water molecules is apparently characterised by a monolayer of molecules, which are strongly bound to the materials (high C) (Quirijns *et al.*, 2005). K, a constant defined as ratio of the partition function of molecules in bulk liquids and the partition function of molecules adsorbed in the multilayer, with values much lower than 1 (0.703, 0.688 and 0.701 at 25, 35 and 45 °C, respectively) showed little variation with temperature. This indicates that the water molecules adsorbed in the multilayer do differ considerably from the bulk liquid water (Quirijns *et al.*, 2005).

Thermodynamic properties

Net Isosteric heat of sorption and differential entropy

Q_{st} and the differential enthalpy are shown in Figure 2. There was an exponential decrease in Q_{st} of the flour as the moisture content increased. Sanni *et al.* (1997) have reported similar trends for *fufu* flour. Q_{st} measures the binding energy of the forces between water vapour molecules and the solids. The decrease in heat of sorption with increase in adsorbed water indicated that water-solid interactions are strongest at lower moisture contents (active polar sites). This could explain the high interaction energy between the water molecules (monomolecular layer) and the flour (Al Muhtaseb *et al.*, 2004). As a result, the energy required to remove these water molecules bounded to the flour is high. As the moisture content increased, the interaction reduced, lowering the net heat of sorption, and the water molecules behave as free water (available for microbial growth) (Quirijns *et al.*, 2005).

Figure 2. Effect of moisture content on the net isosteric heat of sorption (Q_{st}) for water yam flour.

S_d calculated from the intercept of the plot of In a_w versus 1/T at constant moisture content is shown in Figure 3. A strong relationship was found between S_d and moisture content which was similar with that of Q_{st}. S_d decreased with increase in moisture content. S_d is the degree of disorderliness in a food material. Sorption entropy of a food material is proportional to the number of available sorption sites at any specific energy level. Decreasing moisture content restricts the movement of water in the material as the degree of orderliness increases. Similar results have been reported for sweet potato flour by Fasina (2006).

Figure 3. Effect of moisture content on differential entropy (S_d) of water yam flour.

Enthalpy-entropy compensation theory

The plot of Q_{st} against $S_{d,}$ as shown in Figure 4, showed a linear relationship indicating that compensation exists.

$$Q_{st} = 371.32S_d - 719.06 \ (R^2=0.9995) \tag{8}$$

Figure 4. A plot of the net isosteric heat of sorption (Q_{st}) against the differential entropy (S_d).

This implies that the compensation theory could be applied in the range of moisture contents studied. The isokinetic temperature was determined from the slope as T_1 = 371.32 K, which is the temperature at which all the sorption reaction will take place at the same rate. The harmonic mean temperature was calculated as T_{hm} = 307.78 K; as this value significantly differs from T_1, thus confirming the suitability of the isokinetic theory. Also, as $T_1>T_{hm}$, the processes can be considered as enthalpy driven, which implies that only one mechanism of reaction is followed by all members of the reaction series. This further suggests that the microstructure of the flour was stable and does not undergo any changes during moisture adsorption (McMinn *et al.*, 2005). The result obtained agrees with that reported for sweet potato flour by Fasina (2006). The free energy ΔG is indicative of the affinity of sorbent for water, it provides a means of determining if the sorption process is spontaneous (-ΔG) or non-spontaneous (+ΔG). Examining the values of ΔG from Equation 8 above revealed that the sorption process was spontaneous. McMinn *et al.* (2005) had previously reported spontaneous sorption isotherms for starchy materials.

4. Conclusions

The adsorption isotherms of water yam flour at 25, 35 and 45 °C have been presented. A decrease in EMC was observed as the storage temperature increased. The Peleg model was found to be the best for predicting the sorption isotherms of water yam flour within the range of water activities studied. The monolayer moisture content decreased as temperature increased from 25 to 45 °C. The Q_{st} was high at low moisture content and decreased as moisture content increased. Such an analogous trend was observed for S_d. The enthalpy-entropy compensation theory was satisfied and the sorption process was enthalpy driven.

Acknowledgement

The authors wish to express their gratitude to the EU-FP7 for funding the research under the GRATITUDE project ‘Gains from losses of root and tubers’ (FP7-KBBE-2011-5).

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