Moisture Adsorption Characteristics of Bambara Groundnut (*Vigna subterranea*) Powders

J. S. Alakali[1] and A. A. Satimehin[2]

[1]Department of Food Science and Technology, University of Agriculture, Makurdi, Nigeria

[2]Department of Agricultural Engineering, University of Agriculture, Makurdi, Nigeria

adeadesat@yahoo.co.uk [A. A. Satimehin]

ABSTRACT

Adsorption equilibrium moisture content data of Bambara groundnut (V*igna subterranea*) powders were determined at 20, 30 and 40° C using the static gravimetric method. The equilibrium moisture content of the powders decreased with increasing storage temperature and increased with water activity. Four commonly used three-parameter moisture sorption isotherm models were evaluated for their ability to fit the experimental data using direct non-linear least squares regression techniques. The models were the modified Henderson, modified Chung-Pfost, modified Oswin and the modified Halsey models. The models were compared for their goodness of fit using their percent root mean square of error (%RMS) and standard error of estimate (SEE). The modified Chung-Pfost moisture sorption isotherm model gave the best fit to the experimental data (RMS = 8.321% and SEE = 0.051) The net isosteric heats of sorption of dehulled bambara groundnut powders (DBP) were generally higher than those of the undehulled (UBP) seeds and decreased with increase in moisture content. Similarly, the isosteric heat of adsorption of the first molecule of water (Q_o) was higher in the DBP than in the UBP. Conversely, the characteristics moisture content (M_o) of the DBP was lower than that of the UBP. The entropy of sorption of DBP was higher (-0.050 to -0.030 kJ K^{-1} mol^{-1}) than that of UBP (-0.055 to -0.039 kJ K^{-1} mol^{-1}) showing a greater degree of freedom of moisture in powders of dehulled than those of undehulled bambara groundnut.

Keywords: Bambara groundnut, V*igna subterranean*, moisture adsorption isotherm, isosteric heat, entropy, Nigeria.

1. INTRODUCTION

Bambara groundnut (V*igna subterranea*) is an indigenous African crop believed to have been domesticated in West Africa from its presumed wild ancestor (Fery, 2002; Heller *et al.,* 1997 and Linnemann 1990). It is extensively cultivated in West Africa where about 0.33 million tonnes are produced on 400,000 ha of land annually. The major producing countries are Nigeria and Ghana, where it is third in importance only to cowpeas and peanut (Lacroix *et al*., 2003; Fery, 2002).

Bambara groundnut is a major source of protein in the Sub-Saharan Africa (Brink *et al*., 2002; Adu-Dapaah and Sangwan 2004) and constitutes a major part of the local diet, culture and economy (Heller *et al*., 1997). The seed is regarded as a complete balanced food. On dry basis, it consists of 51 - 70% carbohydrate, 16 - 12% oil, 18.0 - 24.0% protein (with high lysine and meteomine content), 3.0 - 5.0% ash, 5.0 - 7.0% fat, and 5.0 - 12.0% fiber. Its mineral contents are 1144 - 1935 mg potassium, 2.9 - 12.0 mg sodium, 95.8 - 99.0 mg calcium and 4.9 - 48 mg iron per 100 g seed. Especially the iron content is high compared to other legumes. The energy content is 367 - 414 kJ per 100 g seed, a value that is higher than the energy content of several other pulses (Adu-Dapaah and Sangwan, 2004).

Bambara groundnut is consumed in several ways. The seeds are prepared into Bambara milk, which is often preferred to milk prepared from other pulses because of its flavour and colour. The seeds are eaten fresh or grilled while immature. In many countries of West Africa, fresh pods are boiled with salt and pepper and eaten as snack. In East Africa, the seeds are roasted, pulverized and used in preparing soup. Dry seeds are ground into powder, which is used for bread making or prepared into stiff porridge, a very popular semi-fluid food in some parts of Nigeria. Bambara groundnut is also used for livestock feeds (Goli, 1997; Heller e*t al*., 1997).

Like most agricultural products, Bambara groundnut powder is hygroscopic and the storage environment could adversely affect its quality. Consequently, the study of moisture sorption characteristics of Bambara groundnut under various environmental conditions is imperative. Knowledge of the moisture sorption characteristics is needed for shelf-life prediction and determination of critical moisture content for acceptability and storage of products. They also provide valuable information on the thermodynamics of moisture sorption (Gevaudan *et al*., 1989) and serves as useful tools for determining the interaction of water and the food substance.

Moisture is held in hygroscopic materials by physical and chemical forces (Cenkowski *et al*., 1992) and the mechanism of moisture binding is affected by sorption characteristics of the material. The heat of sorption therefore provides useful information on the heat and free energy changes during moisture sorption processes in foods (Sopade and Ajisegiri, 1994). It enables the determination of the level of moisture content at which isosteric heat of adsorption approaches the heat of vaporization of pure water. That is, it makes it possible to predict the moisture content below which additional energy will be required to remove bound water from the hygroscopic food substance.

The broad objective of this study was to determine the moisture adsorption characteristics of dehulled (DBP) and undehulled (UBP) Bambara groundnut powders. Specific objectives were to evaluate the moisture adsorption of Bambara groundnut powders at three temperatures, to evaluate the goodness of fit of four sorption models, to obtain the model that best fits the sorption data, to determine the isosteric heat and the entropy of moisture sorption of the powders, and to develop relationships for estimating the isosteric heat of Bambara groundnut powders at various levels of sorbed moisture.

2. MATERIALS AND METHOD

2.1 Materials

About three kilograms of Bambara groundnut (V*igna subterranea*) seeds were purchased from a local market in Makurdi, Nigeria. The seeds were divided into two lots. One lot was soaked in warm water for two hours and dehulled while the other lot was left undehulled. The dehulled seeds were dried in an electric oven (model T12H, Genlab, England) at 80° C for 48 hours to constant weight. Samples from each lot were, in turn, milled using a bench-top hammer mill (Model Brook Compton Series 2000, England) and sieved through a 500 μm mesh screen. The resulting powders were further desiccated over concentrated sulphuric acid for seven days to remove residual moisture. The samples were then packed in 10 g sachets using polythene bags (2 mm thick) and sealed with an impulse sealer (Model 210-SE).

2.2 Determination of Sorption Isotherms

Moisture adsorption was determined gravimetrically as described by Mok and Hettiarachchy (1990) in the temperature range of 20, 30 and 40° C. Constant relative humidity environments were created by means of various concentrations of sulphuric acid. Relative humidity data (37.1 to 93.9%) of the sulphuric acid solutions were obtained from literature (Weast and Astle, 1989). Samples weighing 0.5 g were placed in sample holders, which were in turn suspended over stainless steel gauze in the constant relative humidity environments and allowed to equilibrate at the selected temperatures in a Gallenkamp incubator (Model INF 600.010 R). The temperatures were monitored and controlled within ±1° C. The samples were weighed at 48 hours interval until three successive reading were each less than 0.5% of the previous one (Spiess and Wolf, 1986).

2.3 Moisture Sorption Isotherm Models

Henderson (1952), developed a sorption isotherm model (eqn. 1) to describe the temperature effect as:

$$M = \left[-\frac{\ln(1-a_w)}{AT} \right]^{\frac{1}{B}} \tag{1}$$

M = equilibrium moisture content of food substance (g H_2O/100 g solids), a_w = food water activity (same as the equilibrium relative humidity, in decimal fraction), A and B = constants, T = Temperature (° C).

Earlier, Oswin (1946) developed an empirical sorption isotherm model equation. The modified version of the Oswin equation, which accounts for the influence of temperature, is expressed as (eqn. 2):

$$M = (A + BT)\left[\frac{a_w}{1-a_w} \right]^{C} \tag{2}$$

The Oswin model has been found as a good fit model for sorption of various foods (Lomauro and Bakshi, 1985).

Two other sorption equations often employed in modeling moisture relationship of plant-based agricultural products are the modified Halsey equation (eqn. 3):

$$M = \left[\frac{\exp(A + BT)}{-\ln(a_w)} \right]^{\frac{1}{C}} \tag{3}$$

and Chung-Pfost equation (eqn. 4)

$$M = \frac{1}{C}\left[\ln\left(\frac{A}{T + B} \right) - \ln(-\ln(a_w)) \right] \tag{4}$$

that were given by Menkov and Durakova (2005).

2.4 Analysis of Moisture Sorption Data

Equilibrium moisture data obtained from the experiment were plotted against the water activity. The data were analyzed using the models of Henderson (eqn. 1), Oswin (eqn. 2), Halsey (eqn.3) and Chung-Pfost (Eq. 4). The parameters of the models were calculated by direct non-linear regression analysis, using SPSS for the Windows Release 6.0 (SPSS Inc. 1993). The accuracy of the models were compared by calculating their percent root mean square of error (%RMS) as prescribed by Mok and Hittiarachchy (1990) and their standard error of estimate (SEE) given in the following equations, respectively.

$$\%RMS = \sqrt{\frac{\sum \left[\frac{M_i - M_p}{M_i} \right]^2}{n}} x100\% \tag{5}$$

$$SEE = \sqrt{\frac{\sum (M_i - M_p)^2}{df}} \tag{6}$$

where M_i and M_p are experimental and predicted moisture values respectively; n = number of observations and df = degree of freedom.

2.5 Isosteric Heat of Sorption

Isosteric heat of sorption is the minimum amount of energy required to remove or add a given amount of water to a hygroscopic material. Chen and Lai (1990) described four methods of determining the heat of vaporization of agricultural products. These include the direct calorimetric measurement technique and calculation from the EMC data by using BET, Othmer and Clausius-Clapeyron equations. The Clausius-Clapeyron equation is widely used in sorption studies because of its theoretical basis (Iglesias and Chirife, 1976; Ezeike, 1988; Wang and Brennan, 1991; Cenkowski *et al*., 1992).

At a constant amount of sorbed water, the Clausius-Clapeyron equation is expressed by Wang and Brennan (1991) and Cenkowski *et al*. (1992) as follows:

$$\frac{d\ln P}{dT} = \frac{h^*_{fg}}{R_0 T_a^2} \tag{7}$$

For pure water, equation (7) becomes

$$\frac{d\ln P_o}{dT} = \frac{h_{fg}}{R_0 T_a^2} \tag{8}$$

Subtracting equation (8) from equation (7) yields

$$\frac{d\ln\left(\frac{P}{P_0}\right)}{dT} = \frac{d\ln(a_w)}{dT} = \frac{Q_{st}}{R_0 T_a^2} \tag{9}$$

where

$$Q_{st} = h^*_{fg} - h_{fg} \tag{10}$$

Integrating equation (9) yields

$$\ln(a_w) = \left(\frac{-Q_{st}}{R_0}\right)\frac{1}{T_a} + C_{st} \tag{11}$$

Where C_{st} is the constant of integration.

If Q_{st} is independent of temperature, at a constant amount of adsorbed water, its value can be estimated from the linear relation between $\ln(a_w)$ and $1/T_a$ by determining the slope, which equals $-Q_{st}/R_o$. A mathematical model, which expresses the heat of adsorption as a function of moisture, was developed by Gallagher (1951) and commonly cited in the literature (Ezeike, 1988 and Cenkowski *et al*., 1992). The equation is given as:

$$h^*_{fg} = h_{fg}\left[1 + a\exp(bM)\right] \tag{12}$$

Rearranging equation (12) leads to the equation proposed by Tsami *et al*. (1990):

$$Q_{st} = Q_o \exp\left[-\frac{M}{M_o}\right] \tag{13}$$

where Q_o is the isosteric heat of sorption of the first molecule of water in the food (kJ/mol), M_o is a characteristic moisture content (% dry basis) of the food material; it is the moisture content at which the net isosteric heat of adsorption has been reduced by 63% (Kiranoudis *et al*., 1993).

Equation (13) was fitted to the data for Q_{st} and moisture content (M) by least square non-linear analysis to obtain values of Q_o and M_o for DBP and UBP

2.6 Entropy of Sorption

The entropy of sorption was derived from the Gibbs free energy of sorption and related to temperature (Ariahu *et al*., 2006; Rizvi, 1995) as follows.

$$\Delta G^o = Q_{st} - T_a \Delta S^o \quad (14)$$

Where ΔG^o is Gibbs free energy (kJ/mol) and ΔS^o is isosteric entropy of sorption ($kJ/K^{-1}mol^{-1}$).
According to Rizvi (1995), Gibbs free energy is related to water activity as follows:

$$\Delta G^o = RT_a \ln(a_w) \quad (15)$$

From equations (14) and (15), it follows that:

$$\ln(a_w) = -\Delta S^o/R + Q_{st}/RT_a \quad (16)$$

The ΔS^o values were determined from the intercept coefficient derived from least square regression of the plot of $\ln(a_w)$ versus $1/T_a$ at constant moisture content.

3. RESULTS AND DISCUSSION

3.1 Effect of Temperature

From figure 1 and figure 2, the equilibrium moisture contents of the powders, at constant water activity (a_w), decreased with increasing temperature. At a_w = 0.744, the equilibrium moisture contents of DBP were 14.84, 13.83 and 10.84 g H_2O/100 g solid at 20, 30 and 40° C respectively. Similarly, the equilibrium moisture contents of UBP were 13.87, 12.77 and 8.94 g H_2O/100 g solid at 20, 30 and 40° C respectively. This implies that at any relative humidity, Bambara groundnut powders become less hygroscopic with an increase in temperature. Consequently, in an atmosphere of constant relative humidity, it may be expected to adsorb more moisture at lower temperatures than it would at a higher one. An increase in temperature at a fixed moisture content, tends to lower the isotherm curves, increase a_w and increase the product's susceptibility to microbial spoilage (Labuza *et al*., 1985).

From the kinetic molecular theory, it could be argued that as the temperature increased, sorbed molecules gain kinetic energy and high degree of freedom, which promotes escape of water from the sorbent surface. This causes the amount of sorbed water to decrease with increase in temperature (Karel, 1975 and Labuza *et al*., 1985). Several researchers observed an increase in a_w with an increase in temperature (Menkov and Durakova, 2005 for defatted pumpkin seed flour, Menkov *et al*., 2005 for bean powder, Palou *et al*., 1997 for cookies and corn flaxes, Diosady *et al*., 1996 for canola meals, and Wang and Brennan, 1991 for Irish potatoes). The authors postulated that as the temperature increased, the structure and constituent of the materials were affected resulting in surface plasticization and reduction in sorption sites; and hence the reduction in the equilibrium moisture contents. The implication is that at high storage temperatures, even though the equilibrium moisture content is constant, there would be a shift in water activity to values above the critical level for storage of the product. Hence the product will deteriorate at higher temperatures than it would at lower temperatures.

3.2 Effect of Water Activity

The effect of water activity on moisture adsorption of DBP and UBP is shown in figure 1 and figure 2. It was observed that the equilibrium moisture content (sorbed water) increased as water activity increased at constant temperature. The sorbed water corresponding to water activity below 0.70 was small for relatively large increase in water activity. For instance, at 20 °C and water activity range of 0.371 - 0.744, the sorbed water ranged from 10.64 to 14.84 g H_2O/100 g solid for DBP and 8.51 to 13.87 g H_2O/100 g solid for UBP. On the other hand, for relatively small rise in water activity (between 0.805 – 0.939), sorbed water increased significantly from 19.15 to 31.91 g H_2O/100 g solid for DBP and 16.62 to 27.66 g H_2O/100 g solid for UBP. These results indicate that at low water activities, less water was available for adsorption by the material.

From the above results, the critical equilibrium relative humidity (ERH) for DBP and UBP lies about 60 % for the temperature range of 20 - 40° C. Above this ERH, a marginal increase in water activity would cause a significant increase in the amount of water adsorbed. This in turn would accelerate deterioration. Loong *et al.* (1995) and Labuza (1968) observed similar trends in foods.

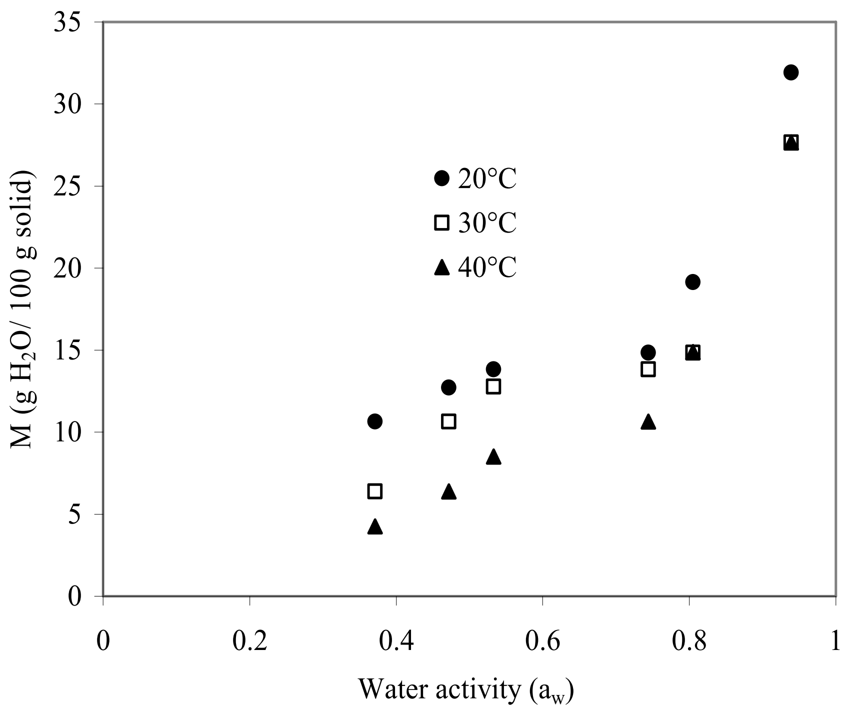

Figure 1. Equilibrium moisture isotherms for flours of dehulled Bambara groundnuts (DBP)

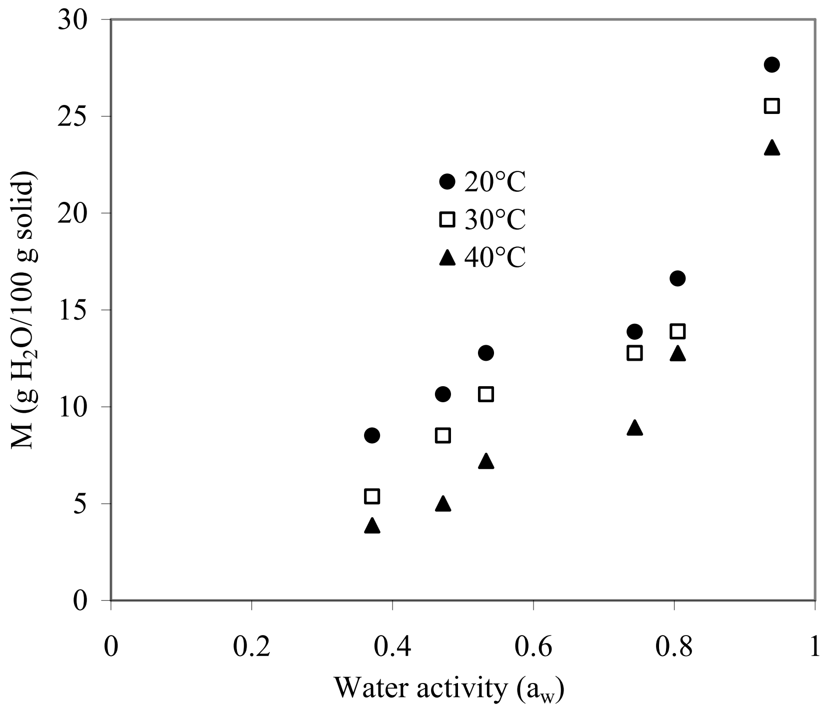

Figure 2. Equilibrium moisture isotherms for flours of undehulled Bambara groundnuts (UBP)

3.3 Effect of Processing Method

Figure 3 is a typical comparison between the equilibrium moisture isotherms of DBP and UBP at 40° C. The figure shows that the equilibrium moisture contents of the DBP were higher than those of the UBP at any temperature and water activity studied. For instance, at 40° C and water activity of 0.805, the equilibrium moisture content of DBP was 14.89 g H_2O/100 g solid whereas that of the UBP was 12.77 g H_2O/100 g solid. This indicates that DBPs were more hygroscopic than the UBPs. It would therefore seem that by removing the seed coat of Bambara groundnut, its powder would become more susceptible to moisture adsorption. The low hygroscopy of UBP might be due to the higher fiber content of the powder. Fibers are less hygroscopic than carbohydrates. Also, the presence of the coat in undehulled sample may have led to the reduction in sorption sites and further accounted for low moisture adsorption and equilibrium moisture contents of UBP. It follows, therefore, that at the same temperature and water activity, undehulled Bambara groundnut powder is expected to keep longer than that from dehulled seed, the former being less hygroscopic. Threshold for safe storage is a water activity below 0.7. At higher a_w micro-organisms will develop. Therefore, UBP has to be dried to lower moisture content than DBP to stay below a_w value of 0.7 during storage at the same temperature.

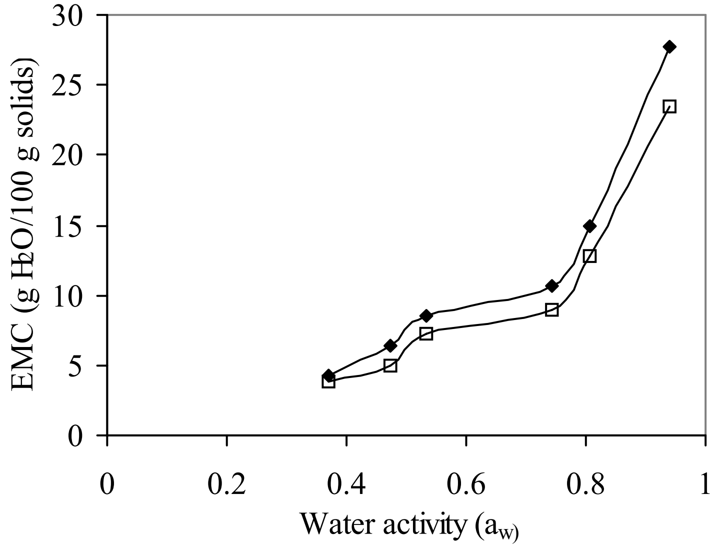

Figure 3. Typical comparison between the moisture isotherms
of DBP (□) and UBP(~) at 40° C

3.4 Evaluation of Sorption Models

The estimated parameters of the isotherm models and their percentage root mean square (%RMS) and standard error of estimate (SEE) are presented in Tables 1 and 2. The tables show that of all the four models tested, the modified Chung-Pfost model had the least %RMS of 10.292 % for the powders of dehulled and 8.321% for the undehulled seeds, respectively. Similarly the SEE values for the Chung-Pfost model were lowest at 0.051 and 0.062 for the dehulled and undehulled powders respectively. From the percentage RMS and SEE values, the Chung-Pfost moisture adsorption model gave the best fit to experimental data. This result, however, does not exactly support the view of Menkov *et al.* (2005) who reported that the modified model of Halsey gave the best fit to their data for the powder of another legume, namely common bean.

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Table1: Estimates of the parameters of different isotherm model equations for the moisture adsorption of dehulled Bambara groundnut at 20 – 40 °C

	Model parameters			Model accuracy		
Model	A	B	C	SEE	%RMS	R^2
Modified Henderson	0.00117	2.781	1.319	0.072	13.177	0.876
Modified Chung-Pfost	73.322	-4.261	0.152	0.062	10.292	0.910
Modified Oswin	13.505	-0.129	0.396	1.818	19.761	0.944
Modified Halsey	4.326	-0.045	1.530	0.081	14.976	0.844

Table 2. Estimates of the parameters of different isotherm model equations for the moisture adsorption of undehulled Bambara groundnut (UDP) at 20 – 40 °C

	Model parameters			Model accuracy		
Model	A	B	C	SEE	%RMS	R^2
Modified Henderson	0.00161	1.183	1.281	0.661	10.674	0.911
Modified Chung-Pfost	67.438	-4.085	0.166	0.051	8.321	0.939
Modified Oswin	11.997	-0.122	0.404	1.399	15.701	0.958
Modified Halsey	4.132	-0.047	1.527	0.069	12.188	0.888

3.5 Isosteric Heat of Adsorption

The net isosteric heat of adsorption (Q_{st}) of water was obtained from equation (11) using the adsorption isotherms of dehulled and undehulled bambara groundnut powers. The slopes of the plot of $\ln(a_w)$ versus $1/T_a$ (figure 4 and figure 5) gave the isosteric heats of sorption in the moisture content range of 4 - 12 g H_2O/100 g solid. A plot of the isosteric heat of adsorption of DBP and UBP as a function of moisture content (figure 6) shows that the heat of adsorption was considerably high when the product was dry but decreased as the moisture content increased. The values of isosteric heats of DBP and UBP were 19.941 and 21.681 kJ/mol respectively. The values of isosteric heats of adsorption are of the same order of magnitude as those of some other foods substances in the literature. Palou *et al*. (1997), Loong *et al*. (1995), Wang and Brennan (1991) explained this decrease quantitatively by considering that initially sorption occurs at most active available sorption sites giving rise to high interaction energy. As these sites become occupied, sorption occurs on the less active sites, giving rise to resulting in a lower heat of sorption. At low moisture, the high heat of adsorption is due in part to the greater resistant to moisture migration from the surface to the interior of a product (Cenkowski *et al*., 1992). It is also probably due to strong interaction between water molecules and the hydrophilic group of the food solid (Loong *et al*., 1995).

Wang and Brennan (1991) also indicated that heat of sorption at the lower moisture content could be attributed to the chemisorption on the polar sites and also to the strained hydrogen bonds in the food solid on dehydration. The value of the isosteric heat of the first molecule (Q_o) and the characteristic moisture contents (M_o) based on equation (8) are presented in Table 3.

Table 3. Regression parameters to calculate Q_{st} for dehulled (DBP) and undehulled (UBP) Bambara groundnut powder.

	Samples	
Regression Parameters	DBP	UBP
Q_o (kJ/mol)	19.941	21.582
M_o (g H_2O/100 g solid)	24.154	17.422
R^2	0.952	0.982

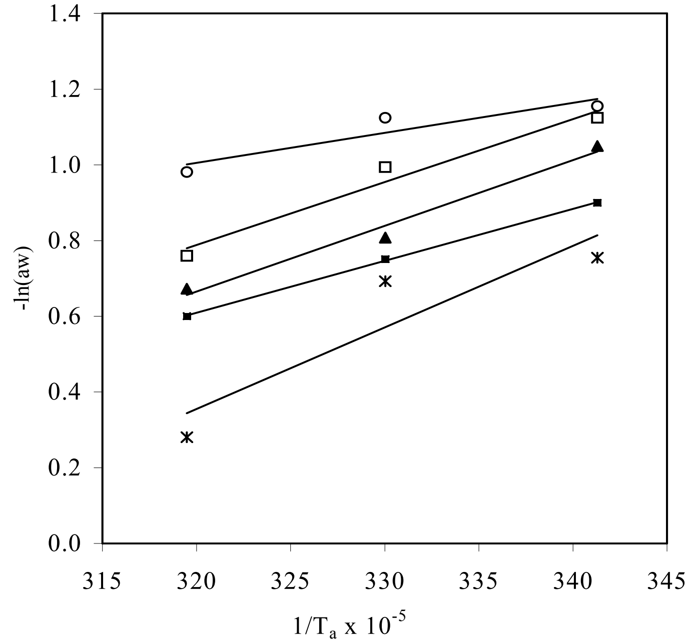

Fig 4: Plot of $-\ln(a_w)$ vs $1/T_a$ for DBP at 4% (o), 6% (□), 8% (▲), 10% (■), and 12% (∗) sorbed moisture

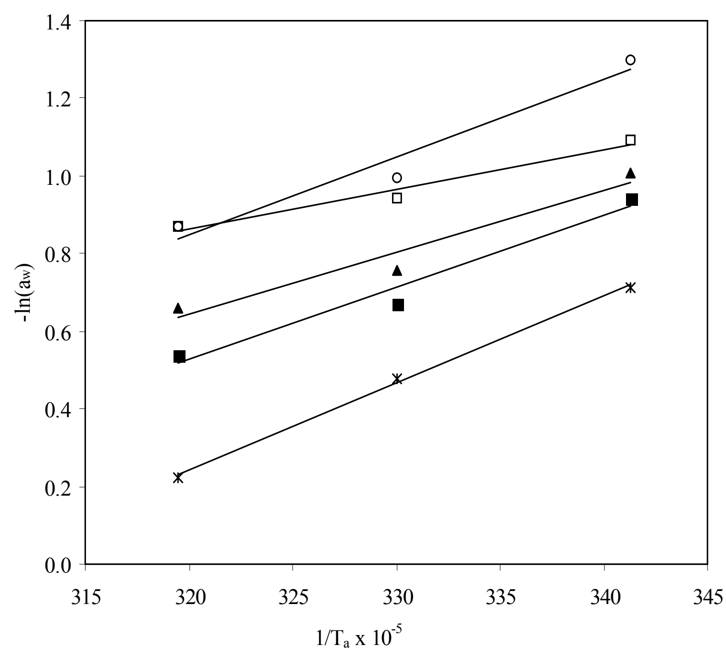

Fig 5: Plot of $-\ln(a_w)$ vs $1/T_a$ for UBP at 4% (o), 6% (□), 8% (▲), 10% (■), and 12% (∗) sorbed moisture

Values of Q_o on Table 3 were obtained from the intercept of the plot of $\ln(Q_{st})$ versus moisture content. From the table, Q_o for the DBP was lower than its corresponding value for UBP. This suggests that lesser energy would be required to dry DBP than would be required for the UBP. On the other hand, M_o was higher for DBP than UBP. This implies that the powders of dehulled bambara groundnut are expected to dry faster than those of undehulled seeds. This is because the seed coat in undehulled bambara groundnut presents an increased resistance to moisture movement and reduce the rate of drying. At higher M, i.e. in the case of drying, Q_{st} for UBP is lower than that of DBP. Therefore, the powder of dehulled seeds is expected to dry faster and with less energy input than is the case with undehulled seeds. This can be explained by the higher fibre content of the undehulled seeds, because fibre is less hygroscopic than carbohydrates.

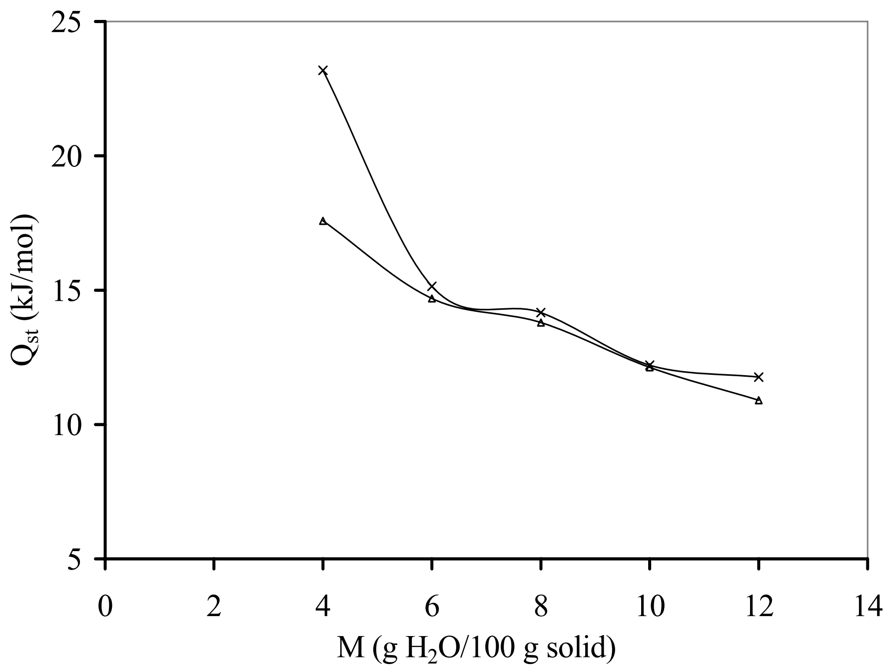

Figure 6. Isosteric heat versus moisture content of DBP (x) and UBP (Δ)

3.6 Entropy of Sorption

The entropy of sorption (ΔS^o) was determined based on equations (16), and found to increase with moisture content (Figure 7). For the DBP, the entropy of sorption was generally higher than that of UBP. This was expected because the EMCs of DBP were higher than those of UBP, thereby accounting for the higher entropy of sorption in DBP. In the moisture content range of 4 to 12 g H_2O/100 g solid, the entropy of sorption of DBP increased from -0.050 to -0.030 kJ K^{-1} mol^{-1}, and that of UBP increased from -0.055 to -0.032 kJ K^{-1} mol^{-1}. According to Bethelhiem *et al*. (1970), Loong *et al*. (1995) and Ariahu *et al*. (2006), at low moisture contents water adsorbs on the most accessible sites. As moisture content increases, the polymer swells, opening up new higher energy sites for water to bind. The water molecules also become more mobile with higher degree of freedom, leading to higher entropy of sorption. At low moisture content, however, the water molecules are tightly bound by physical and chemisorption forces resulting in loss of degree of freedom.

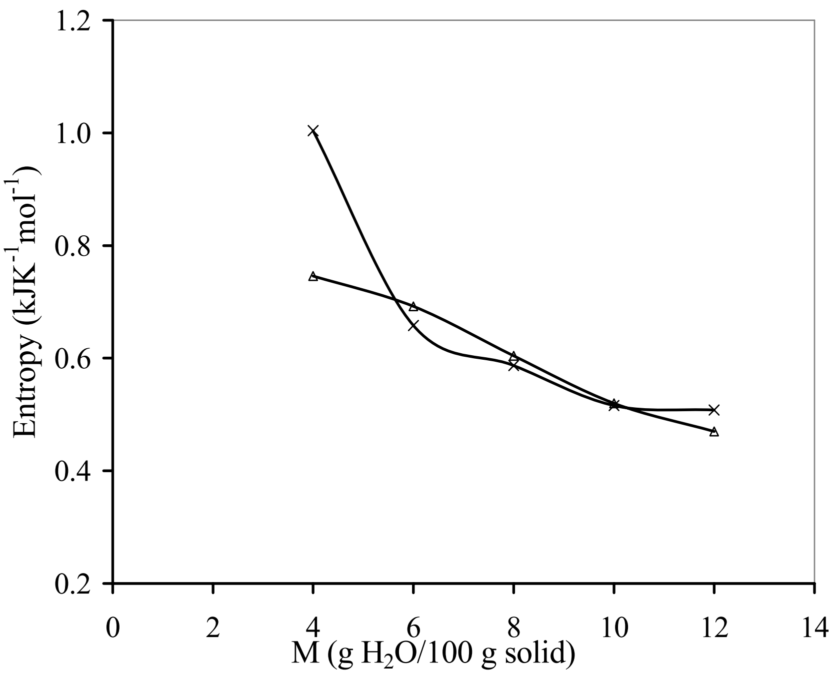

Figure 7 Entropy versus moisture content of DBP (x) and UBP (Δ)

4. CONCLUSION

The equilibrium moisture contents of Bambara groundnut powders increased with water activity and decreased with increasing temperature. Moisture adsorption isotherm data for the products can be more accurately predicted by Chung-Pfost sorption model. Powders of dehulled Bambara groundnut seeds adsorbed more moisture compared to those of undehulled seeds at all the temperatures and water activities studied. The net isosteric heat of sorption of dehulled Bambara groundnut was higher than that of the undehulled groundnut. Net isosteric heat of adsorption of both materials decreased as their moisture content increased. Generally, the entropy of sorption of dehulled bambara groundnut was higher than that of the undehulled material.

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