Adsorption Isotherm and Thermodynamic Characteristics Of Soy-Melon Enriched "Gari" Semolina From Cassava

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ABSTRACT

The adsorption equilibrium moisture contents of soy-melon and control gari semolina samples from cassava were determined using the static gravimetric method. Determinations were made within a range of water activities of 0.20-0.92 at three different temperatures of 20^o, 30^o and 40^{o}C. Results showed that the moisture sorption curves were sigmoidal in shape, which conformed to type II classification characteristic of most biological tissues. A thermodynamic approach was used to interpret the experimental adsorption isotherm data. The net isosteric heat of sorption was determined from the equilibrium adsorption data, using the Clausius–Clapeyron equation in the temperature range used in the experiment. Results indicated that the slopes of the sorption isosteres decreased as water content increased indicating a decrease in the binding energy for water molecules. The values of the isosteric heat of sorption for the two gari semolina samples were consistently higher at lower moisture content and decreased exponentially as moisture increased. Isosteric heat of sorption of soy-melon gari decreased from a value of 42 kJ/mol as the moisture content increased from 4.0% (db) while for the control (unsupplemented) gari, the heat of sorption decreased from a value of 57.5kJ/mole at a moisture content of 10%(db). At moisture content above 18% for the Control gari and 12% for the Soy-melon gari, the isosteric heats of sorption reached minimum values and remained practically constant. The net isosteric heat of sorption of contol gari was generally higher than that of soy-melon gari while control gari existed at higher moisture contents at the water activities used for this study (0.23-0.91). This further explains the ability of control gari to absorb more moisture than soy-melon gari at those water activity levels. The lower values of the thermodynamic functions of soy-melon gari were attributable to the higher amount of oil the soy-melon flour used for supplementing the gari semolina while its higher value than other similar products was due to the gelatinization of the starch in gari semolina as a result of its roasting.

Keywords*:* Net isosteric heat of sorption, adsorption isotherm, soy melon gari semolina, thermodynamic properties, Nigeria.

1. INTRODUCTION

Gari is a fermented, dewatered and toasted semolina of cassava, widely consumed all over West Africa and in Brazil, is the most popular cassava product consumed and the most important item in the diet of millions of Nigerians (IITA and Kordylas, 1990). It forms a significant part of the diet in many of these countries where it is called "Farinha de mandioca" (Lancaster *et al*., 1982).Traditionally, it is produced by pressing the juice out of peeled, grated cassava roots, allowing a natural lactic fermentation to take place for 2-5days. The fermented mash is then toasted in an open aluminum pan over open fire until the starch gelatinizes to 65% of its native form and the moisture content falls to less than 12% dry basis (Chuzel *et al*, 1986, Akinrele, 1967). However, cassava and its products are low in protein, deficient in essential amino acids and therefore, have poor qualitative and quantitative protein content (Oyenuga, 1968). Thus, continuous dependence on gari without supplementation with meat, fish and/or other protein-rich sources would result in protein deficiency. There is therefore, a need to search for cheaper but good quality protein sources that are readily available for the supplementation of gari. Some studies have been carried out to improve the nutrient content of cassava products, but not much in the area of packaging and distribution. The problem of poor keeping quality of many dehydrated foods in the tropics is related to their moisture uptake during merchandising because of poor packaging materials and the moisture levels at which they were prepared (Onayemi and Oluwamukomi, 1986). The relationship between water activity and moisture content of a food-stuff is important in predicting quality stability during drying, storage and the selection of appropriate packaging materials for retail purposes (Dan-Wen, 1999). It has also been used to predict microbial growth and extent of enzymatic reactions in foods (Labuza *et al*, 1970). Some studies have been carried out on the sorption isotherm of Cassava and its products such as for Gari (Chuzel and Zakhia, 1991, Ajibola, 1986, Fasina *et al*., 1999), Lafun (Onayemi and Oluwamukomi, 1986)), Instant cassava flour (Oluwamukomi, 1999), and Fufu (Sanni, *et al*, 1999), cassava and melon seed (Aviara and Ajibola, 2002). The design of effective drying and storage systems for any food product will require knowledge of energy requirements, state and mode of moisture sorption within them.

Thermodynamics is one of the three approaches used to understand the properties of water and calculate energy requirements associated with the transfer of heat and mass in biological systems. The thermodynamic properties of foods relate the concentration of water in the food to its partial pressure, which is crucial in analysis of the heat and mass transport phenomena during dehydration (Al-Mutaseb *et al*., 2004). They determine the end-point to which the food must be dehydrated in order to achieve a stable product with optimal moisture content, and the minimum amount of energy required to remove a given amount of water from the food (Aviara & Ajibola, 2002).They provide an insight into the microstructure associated within the food–water interface (Rizvi, 1995). Some thermodynamic functions used in analyzing sorption behavior of biological systems include free energy, differential heat of sorption, integral enthalpy and integral entropy.

These functions are needed in the analyses of energy requirements in the dehydration process and to predict kinetic parameters of the sorption phenomena (Tolaba *et al*. 1995).
Differential heat of sorption, often referred to as isosteric heat of sorption, Q_{st}, is used as an indicator of the state of water adsorbed by the solid particles (Fasina *et al*., 1997). The state of adsorbed water is a measure of the physical, chemical and microbial stability of biological materials under storage (Labuza, 1968). Isosteric heat of sorption is commonly estimated by applying the Clausius-Clapeyron equation to sorption isotherm data. The Clausius-Clapeyron equation as applied to vapor from free water is given by (Kapsalis 1987). Several methods have been employed by researchers. Gal, (1975) carried out thorough reviews of the methods of determining the moisture sorption isotherms of agricultural materials and pointed out that there are three basic techniques namely the manometric, the gravimetric and the special methods. Labuza *et al*. (1976) reported that the vapour pressure manometric method is one of the best methods as it measures directly the vapour pressure exerted by moisture in grain kernels. However, the gravimetric technique has been noted to be preferable for obtaining complete sorption isotherms, and has been recommended as the standard method (Gal,1981). Knowledge of the differential heat of sorption is of a great importance when designing equipment for dehydration processes. This is due to the fact that the heat of vaporization of sorbed water may increase to values above the heat of vaporization of pure water as food is dehydrated to low moisture levels (King, 1968).

The net isosteric heat of sorption (q_{st}) is defined as the total heat of sorption in the food minus the heat of vaporization of water, at the system temperature (Tsami *et al*., 1990).The heat of adsorption is a measure of the energy released on sorption, and the heat of desorption, the energy requirement to break the intermolecular forces between the molecules of water vapour and the surface of adsorbent (Rizvi, 1995). Thus, the heat of sorption is considered as indicative of the intermolecular attractive forces between the sorption sites and water vapour (Wang & Brennan, 1991).

Many studies have been carried out on thermodynamic properties of foods. Iglesias *et al*. (1976) studied the thermodynamics of water vapour sorption by sugar beet root and advanced hypotheses to explain the values and trends observed. Kiranoudis *et al*., (1993) evaluated the heat of sorption of some vegetables, reporting an exponential function with moisture content. Fasina and Sokhansanj (1993), Fasina *et al*., 1997 and Fasina *et al*., 1999 calculated the thermodynamic functions of moisture sorption in alfalfa pellets, winged been seed and gari, respectively from their moisture sorption isotherms. Tolaba *et al*., (1997) obtained an analytical expression to predict the isosteric heat of cereal grains as a function of moisture content using a three parameters equilibrium model. Viollaz and Rovedo (1999) determined the equilibrium moisture sorption isotherms of starch and gluten and proposed a new method of calculating the isosteric heat (differential heat of sorption) at different temperatures. Aviara and Ajibola (2002), studied the sorption characteristics of cassava and melon seed, they reported that the differential heat of cassava was higher than that of melon seed indicating that cassava has a higher affinity for moisture than melon seed. Ajibola, *et al*. (2003), studied the sorption isotherm of cowpea at five different temperatures and also determined their thermodynamic functions. Aviara, *et al*,

(2004) determined the desorption characteristics of soy bean seed and concluded that the net enthalpy and entropy reduced as the moisture content increased. Öztekin and Soysal (2000), compared the adsorption and desorption isosteric heats for some grains-wheat, corn, rice and rapeseed. The objectives of this study therefore are to determine the equilibrium moisture contents of soy-melon gari semolina stored at three different temperatures of 20, 30, and 40^oC and use this experimental data to determine its isosteric heat of sorption.

2. MATERIALS AND METHODS

A static gravimetric method was used for the experiment. About 3g of freshly prepared Soy-melon supplemented gari semolina samples were placed in petri dishes inside 6 desiccators containing saturated salt solutions ($KC_2H_3O_2$, MCl_2, K_2CO_3, NaBr, NaCl, $NaNO_3$) providing constant relative humidity environments ranging from 23 – 92% in desiccators as described by Onayemi and Oluwamukomi, (1987) and Rockland,(1960). The desiccators containing salt solutions and samples of soy-melon gari semolina were placed inside temperature controlled Gallenkamp DV 400 incubators, which were set at 20, 30 and 40^oC. The temperatures were monitored to within ±1·0°C. The samples were weighed daily using a Mettler PC 2000 electronic balance with an accuracy of 0·001 g. Equilibrium was considered to have been attained when three identical consecutive measurements were obtained. The dry matter content was produced by oven drying at 103°C for 72 h. The equilibrium moisture contents were calculated on dry basis from which the moisture sorption isotherm was determined. The thermodynamic characteristics of soy-melon gari semolina were analytically determined using the non-linear regression procedure in SPSS 10·0 for Windows.

3. RESULTS AND DISCUSSION

3.1 Moisture Adsorption Isotherm of Soy-melon Gari Semolina

The experimental equilibrium moisture contents of Soy melon enriched and control gari semolina at three temperatures of 20^o, 30^o and 40^oC are shown in Table 1. The equilibrium moisture content at each water activity (a_w) represents the mean of three determinations. The standard deviation of each experimental point ranged from 0.002 to 0.0035. The sorption isotherm curves (figs 1 & 2) showed the typical sigmoid shape confirming type II classification which is characteristic of biological material, which sorbs relatively small amount of water at lower water activities and large amounts at high relative humidities (Rockland, 1969, Arévalo-Pinedo *et al.*, 2004, Yoshida and Mengalli, 2000)

Table 1: Equilibrium moisture contents of Soy Melon enriched and control gari semolina samples at 20^o, 30^o and 40^oC

	Soy-melon gari semolina			Control gari semolina		
RH/ a_w	**20°C**	**30°C**	**40°C**	**20°C**	**30°C**	**40°C**
0.23	0.0379	0.0231	0.0217	0.0843	0.0657	0.0536
0.35	0.0611	0.035	0.0316	0.1070	0.0727	0.0665
0.45	0.0806	0.0451	0.0404	0.1304	0.0962	0.0862
0.61	0.0844	0.052	0.0414	0.1526	0.1091	0.1002
0.75	0.1155	0.1052	0.0795	0.1904	0.1481	0.1408
0.92	0.3182	0.1695	0.1448	0.3351	0.2305	0.2155

3.2 Isosteric Heat Of Sorption

The isosteric heat of sorption was determined from the data using the Clausius–Clapeyron equations:

$$q_{st} = -R\left[\frac{d(\ln a_w)}{d\left(1/T\right)}\right] \qquad 1$$

$$Q_{st} = q_{st} + \lambda_{vap} \qquad 2$$

Plotting the sorption isotherm in the form ln(a_w) versus 1/T , for a specific moisture content, the slope of the regression line provided a measure of the net isosteric heat of sorption, qst, with the heat of sorption Qst being obtained by applying Eq. (2). This procedure assumes that qst is invariant with temperature, with the application of the method requiring measurement of sorption isotherms at more than two temperatures. The latent heat of vaporization of pure water (λ) at 30^{o}C was utilized for subsequent analysis; this represents the average temperature within the investigative range 20–40^{o}C (Iglesias & Chirife, 1976). Plots of 1n (a_w) vs. (1/T) at different moisture contents were straight lines (figs 3 and 5) of slopes -Qs/R calculated by regression analysis; the linearity indicating consistency of the isotherms (Linvonen and Roos, 2003).

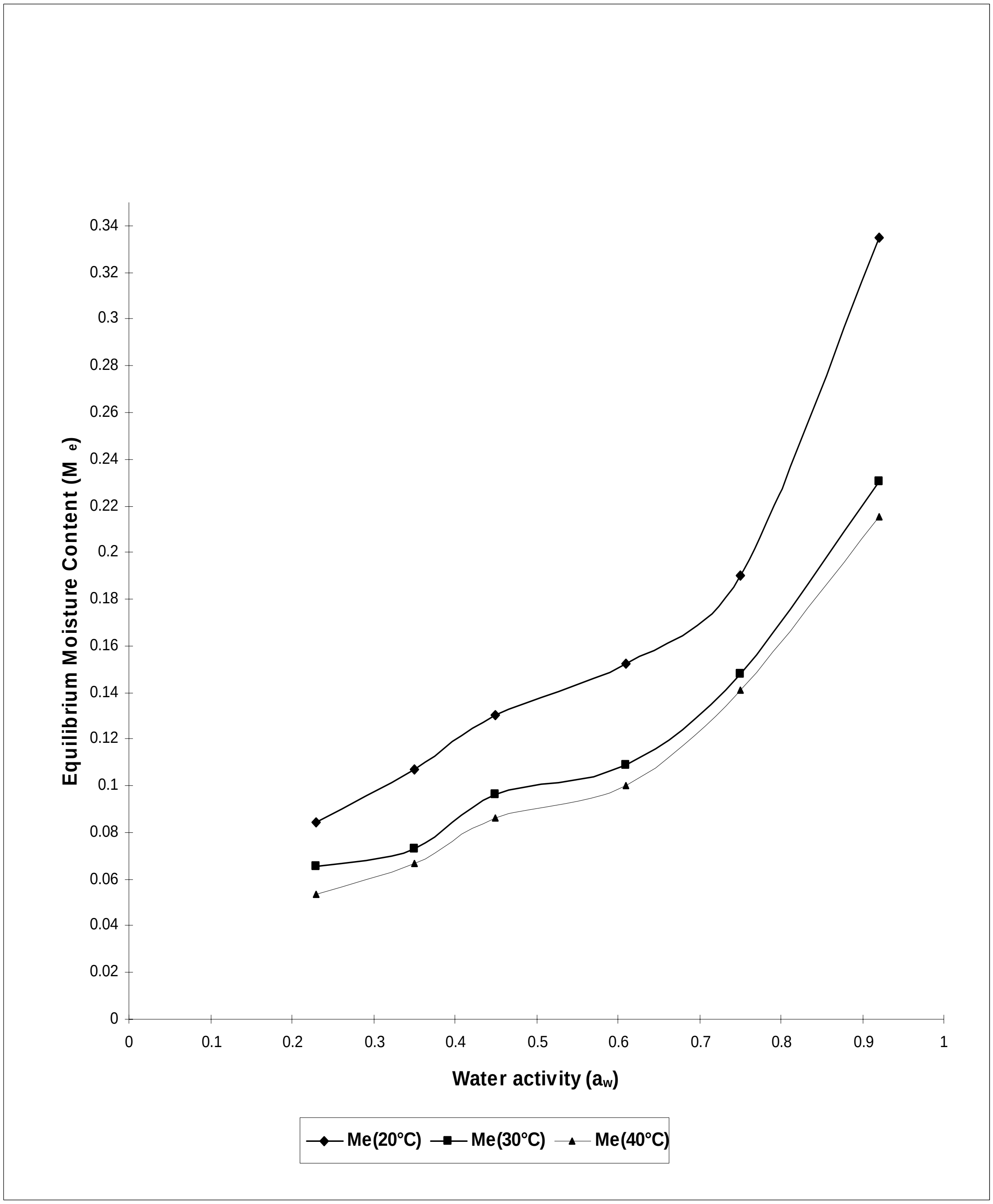

Figure 1: Adsorption isotherm of control gari semolina at 20°, 30° and 40°C

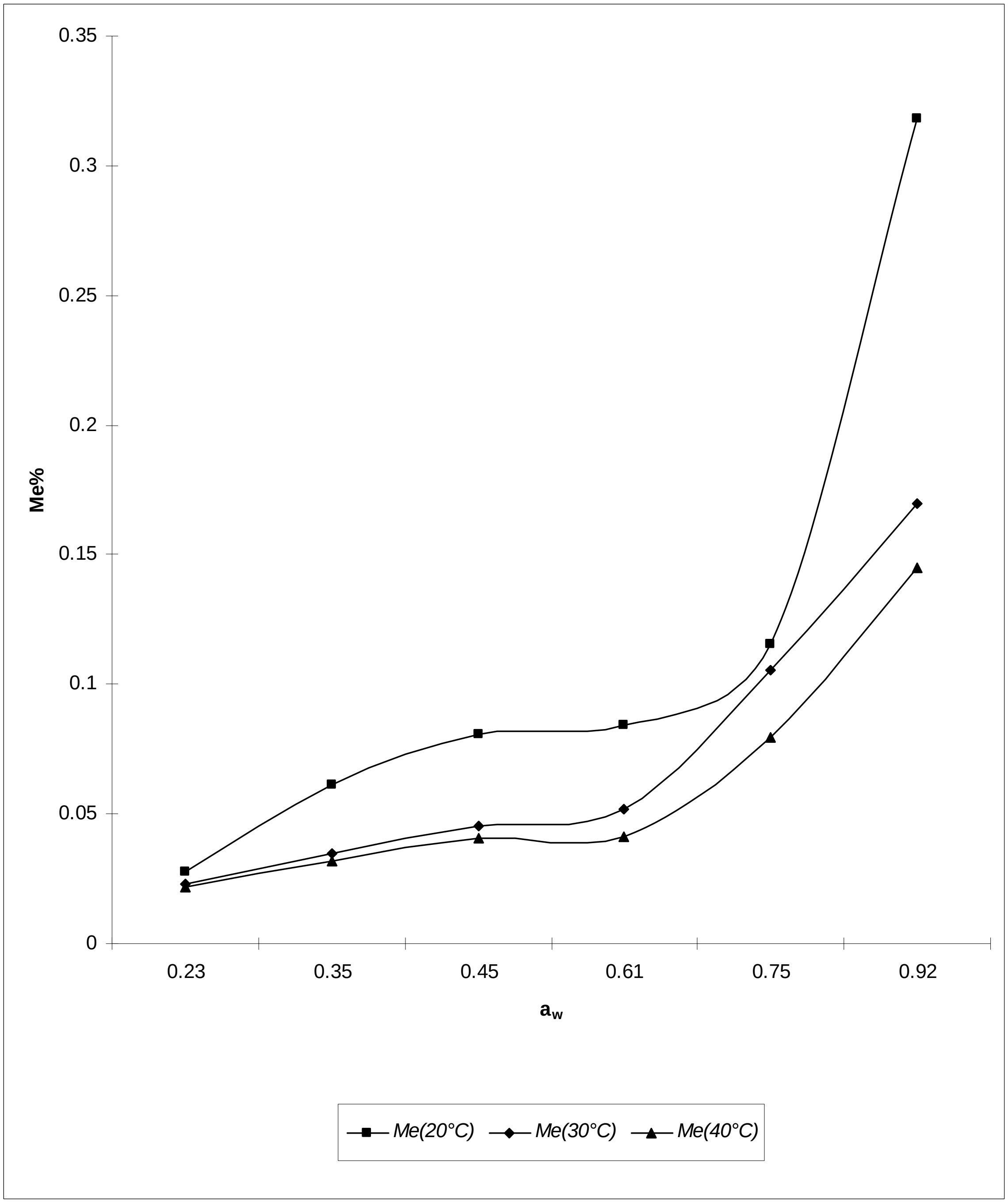

Figure 2: Adsorption Isotherm of soy-melon gari semolina at 20^o, 30^o and 40^oC.

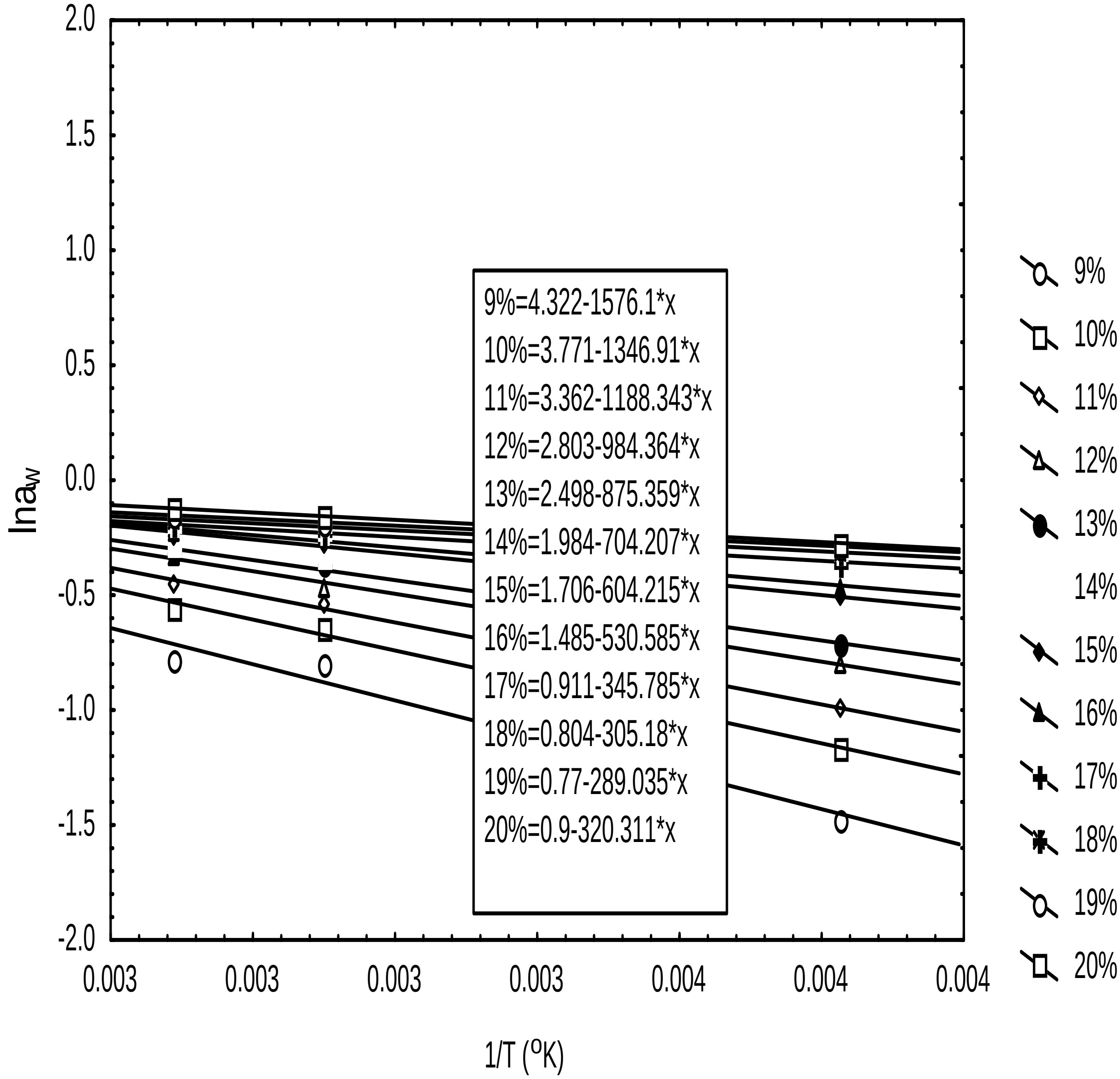

Figure 3: Sorption Isosteres of Control gari semolina

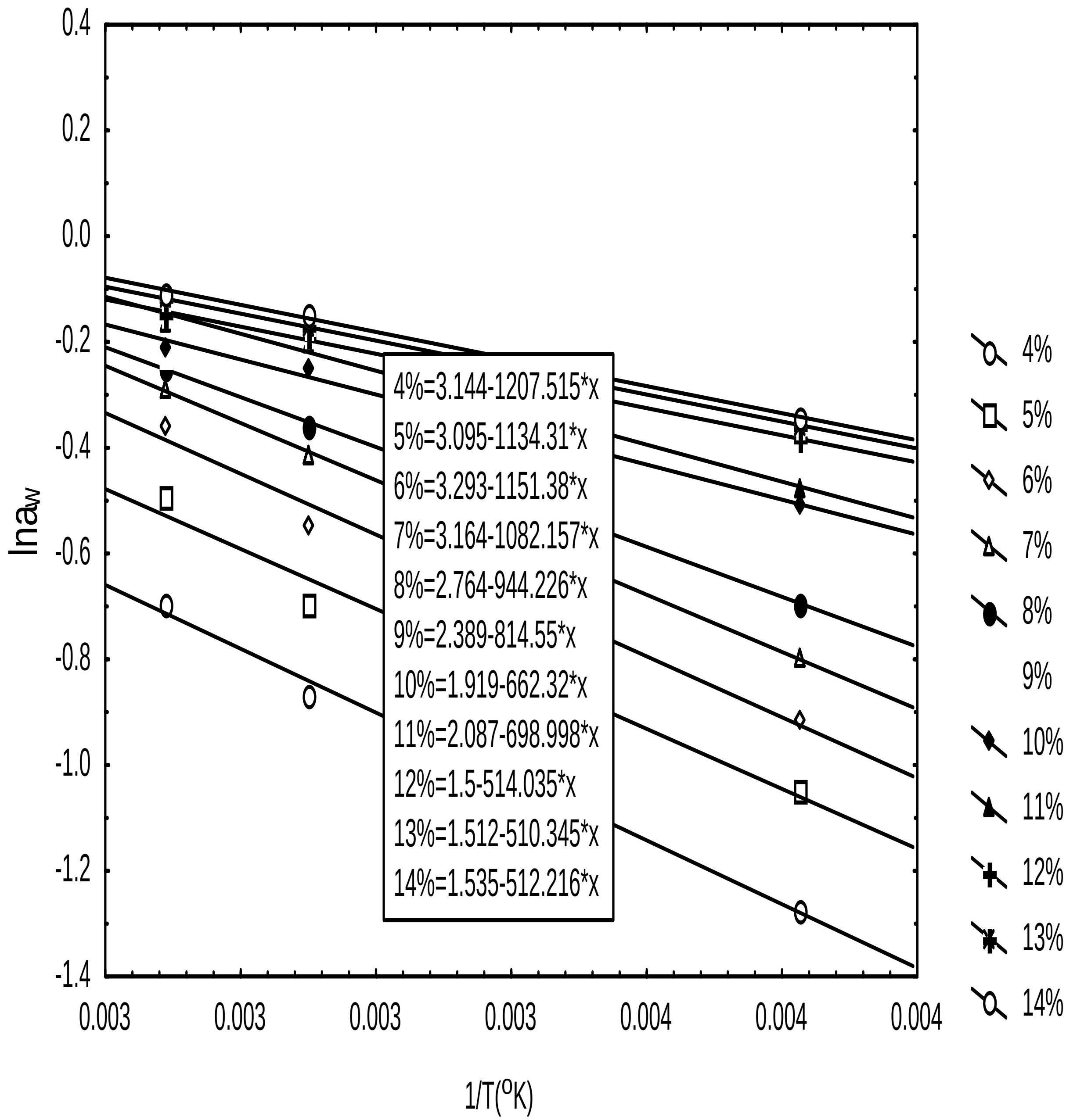

Figure 4: Sorption Isosteres of Soy-melon gari semolina

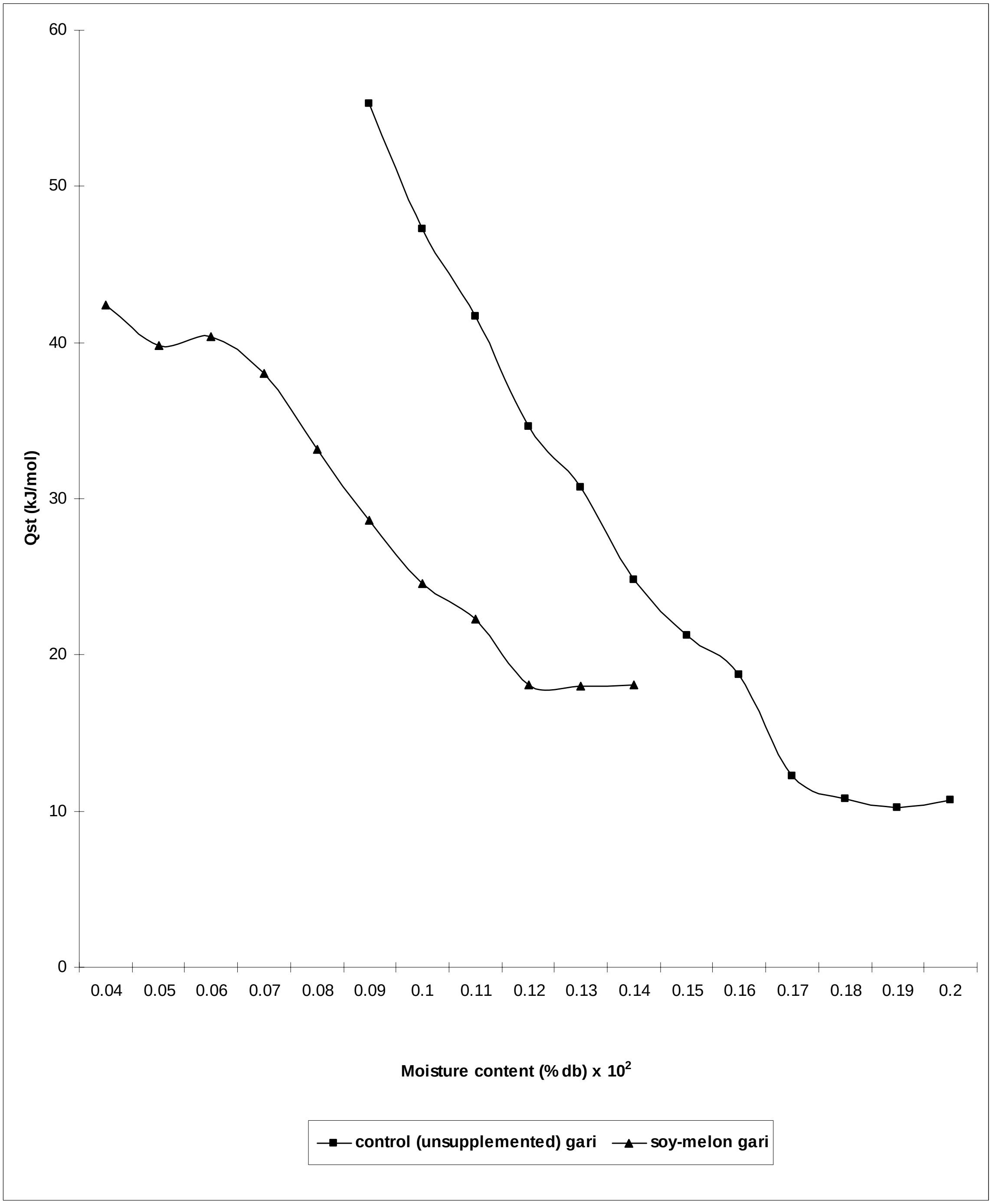

Figure 5: Isosteric Heat of Sorption of Control and soy-melon gari semolina

4. DISCUSSION

4.1 Moisture Adsorption Isotherm of Soy-melon Gari Semolina

There was a general increase in equilibrium moisture content (EMC) with increasing water activity (a_w) (Iglesias and Chirife, 1976, Taylor, 1961). The EMC increased up to the water activity of about 0.45 when it remained slightly constant until it shot up again after the water activity of 0.6. The gradual slope of the isotherm at water activity below 0.60 is characteristic of such products with high protein and fat content. Rockland (1969) found that products with such chemical composition have been found to exhibit gradual sloping isotherm at low water activity. It has also been found that sorption characteristics of foods have been shown to be influenced by their chemical compositions (Crapstice and Rostein, 1982) and their processing treatments to which they have been subjected to (Strolle and Cording1965)

4.1.1 Effect of Water Activity

There was a shift to higher water activity as the temperature increased at given moisture contents. At lower levels of water activity of less than 0.45 representing the region of adsorbed bond water, there was a gentle upward slope. With small increases in a_w, there were slight increases in moisture content of the gari semolina samples at all the temperatures, suggesting that fluctuations in temperatures and low environmental relative humidity will have minimal effects on the storage stability of gari semolina samples. However at higher water activity greater than 0.60, slopes of the isotherms were more steeply, thus producing large increases in EMC per unit a_w, suggesting that fluctuations in temperature and environmental RH will greatly have significant effects on the storage stability of the soy-melon supplemented gari semolina. The effect of increasing temperature is to lower the EMC over a wide range of a_w studied. At constant a_w, samples adsorb more moisture at lower temperatures than at higher temperatures. This is in agreement with results obtained by many workers (Leiras and Iglesias, 1991, Lievonen and Roos, 2002).

4.1.2 Effect of Temperature

The water sorption was found to be temperature dependent (Kapsalis, 2000). The higher the temperature, the lower the Equilibrium moisture content (EMC) at constant water activity ($a_{w)}$. This is explained by the higher excitation statue of water molecules at higher temperature decreasing the attractive forces between them. The gari semolina became less hygroscopic at higher temperatures (Risvi, 1993). The temperature influence at low and intermediate water activities ($a_w < 0.55$), has the normal tendency predicted by theory of physical adsorption, the equilibrium moisture content of gari semolina decreased with increasing temperature. With increasing temperature, the sample adsorbed less water at constant RH% (Ezeike, 1988, Oluwamukomi *et al.*, 2005)

4.2 Isosteric Heat of Sorption

The slopes of the regression lines (Figures 3 and 4) decreased as water content increased indicating a decrease in the binding energy for water molecules (Kapsalis, 1987). The linearity of the isosteres denoted by the r^2 were good enough but showed there were some variations in the values and were based on the assumption that the heat of sorption was not temperature dependent (Labuza *et al*., 1985). When the isosteric heat of sorption was examined as a function of water content, the heat of sorption for the soy-melon were found to show a steeper water content dependence than the corresponding value for control gari but in practice most food isosteres diverge from linearity due to irreversible changes at high temperatures (Iglesias *et al*, 1989).
The variation of isosteric heat of sorption with the moisture content for soy-melon and control gari is shown in Fig. 5. The values of the isosteric heat of sorption for the two gari samples were consistently higher at lower moisture content and decreased exponentially as moisture increased. Wang and Brennan (1991) explained this decrease that initially sorption occurs on the most "active available" site giving rise to high interaction energy. As these sites become occupied, sorption occurs on the less active sites giving rise to lower heats of sorption. At low moisture content the higher heat of sorption could be due in part to strong interaction between water molecules and the hydrophilic groups of the food solid (Lim *et al* 1995) or it could also be due to the chemisorptions on the polar site and also to strong hydrogen bonds in the food solid on drying (Satimehin & Ezekiel 2002)

Isosteric heat of sorption of soy-melon gari decreased from a value of 42 kJ/mol as the moisture content increased from 4.0% (db). The trend seemed to become asymptotic at an isosteric heat of sorption value of 18.0kJ/mol as the moisture content of 12% was approached. Similar trends have been reported for the enthalpy of sugar beet root, its insoluble fraction and sucrose (Iglesias et al., 1976), grain sorghum (Rizvi & Benado, 1983), melon (Aviara and Ajibola, 2002), wing bean seed and gari (Fasina *et al*., 1999). For soy-melon gari the net isosteric heat of sorption decreased with increase in moisture content up to 12%(db) then. The net isosteric heat of sorption of contol gari were generally higher than that of soy-melon gari while control gari existed at higher moisture contents at the water activities used for this study (0.23-0.92). This further explains the ability of control gari to absorb more moisture than control gari at those water activity levels.

The variation of isosteric heat of sorption of soy-melon gari with moisture content was similar in trend to that of the control gari. For control gari, the heat of sorption, decreased from a value of 57.5kJ/mole at a moisture cont of 10 %(db) and tends towards an asymptotic value as the moisture content increased to 18%. This is the point at which the heat of sorption approaches that of heat of vaporization of water. Iglesias and Chirife, (1976) explained that the level of moisture content at which the heat of sorption approaches the heat of vaporization of water can be a sign of water existing in free form in a food product. Akanbi *et al*, (2006), attributed this behavior to differences in the hydrogen bonding as moisture is absorbed probably due to differences in attractive forces between water molecules and sorption sites; and between water molecules themselves. Above this moisture level the absorbed water vapor in the product behaves like pure water. Below this the isosteric heat of sorption was higher than the latent heat of pure water.

When the isosteric heat value is still greater than the heat of vaporization of pure water it means the energy or interaction between the sorbed and the sorption site is greater than the energy that hold the sorbed molecule together in the liquid state (Iglesias & Chirife, 1976). There was a change in the slope of the linear graph as the moisture content changes from 9% to 10% for soy-melon gari and 13-14% for the control gari. At moisture content above 18% for the Control gari and 12% for the Soy-melon gari, the isosteric heat of sorption reached the minimum value and remains practically constant. This asymptotic response may be due to the fact that as the moisture content of the product increased, the sorption sites get saturated and water molecules become held to the particles more by surface tension than by chemical adsorption.

The surface tensional forces being weaker are then easily broken and this results in lower isosteric heat of sorption (Ajibola *et al*, 2004). This behavior was reported by many workers including Akanbi, (2006), for tomato slices, Satimehin and Ezeike (2003) for gelatinized white yam, Kuye and Sanni (2002) for lafun and soy flour; Oluwamukomi *et al*., (2001) for elekute flour, Chung & Pfost, (1967) for corn and corn product, and Igbeka *et al*., (1975) for cassava. Similar trends have also been reported for alfalfa pellets (Fasina *et al*. 1997), winged bean seed and gari (Fasina *et al*., 1999), rice (Benado ad Risvi, 1985), melon seed (Aviara and Ajibola, 2002), cowpea (Ajibola *et al*., 2003).

When the values of the isosteric heat of sorption in this study were compared with values from similar studies, they were found to be similar 42kJ/mol and 58kJ/mol for soy melon and control gari respectively at the low moisture content (10%) while it was 14 and 25 kJ/mol at the medium moisture content of 14%. It was found out from previous studies to be 52kJ/mol for fufu flour (Sanni *et al*, 1999), 30.2kJ/mol for soy flour and 15.1kJ/mol for lafun flour (Kuye and Sanni (2002), 36.0kJ/mole for shelled egusi (Ezeike, 1988). It could be observed that the control gari was highest followed by fufu, soy-melon gari, soy flour and lafun. The lower values of the thermodynamic functions of soy-melon gari might have been due to the higher amount of oil the soy-melon flour used for supplementing the gari semolina which contributes to lowering sorption capacity in agricultural products (Iglesias and Chirife, 1982), while its higher value than 'fufu' and other similar products might have been due to the gelatinization of the starch in the gari semolina as a result of its roasting.

5. CONCLUSIONS

The following conclusions can be drawn from the study of the isosteric heat of sorption:
(i)The Clausius–Clapeyron equation allows satisfactory determination of the isosteric heats of sorption, which were found to increase with decreasing moisture content.
(ii)An exponential relation was found to adequately describe the dependence of net isosteric heat of sorption on the equilibrium moisture content.

(iii) The net equilibrium heat (isosteric heat of sorption) decreased with increasing moisture content and approached the latent heat of vaporization of pure water at a moisture content between 10 and 14.0% (dry basis) for soy-melon gari and 18.0 to 21.0% for control gari
(iv) At lower moisture levels, the net isosteric heat of sorption of control gari was higher than that of soy-melon gari, indicating that control gari has a higher affinity for moisture than soy-melon gari above a moisture content of 10 %(db)
(v) The lower values of the thermodynamic functions of soy-melon gari might have been due to the higher amount of oil the soy-melon flour used for supplementing the gari semolina while its higher value than 'fufu' and other similar products might have been due to the gelatinization of the starch in the gari semolina as a result of its roasting.

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Nomenclature

R	universal gas constant (8.314 J mol^{-1} K^{-1})
a_w	water activity
T	temperature (K)
Qst	isosteric heat of sorption (kJ mol^{-1})
λvap	latent heat of vaporization of pure water
qst	net isosteric heat of sorption (kJ mol^{-1})