International Journal of Food Engineering and Technology
2017; 1(1): 1-8
http://www.sciencepublishinggroup.com/j/ijfet
doi: 10.11648/j.ijfet.20170101.11

Moisture Adsorption and Thermodynamic Properties of Sorghum-Based Complementary Foods

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To cite this article:

Sengev Iorfa Abraham, Ariahu Chukwuma Charles, Abu Joseph Oneh, Gernah Dickson Iorwuese. Moisture Adsorption and Thermodynamic Properties of Sorghum-Based Complementary Foods. *International Journal of Food Engineering and Technology*. Vol. 1, No. 1, 2017, pp. 1-8. doi: 10.11648/j.ijfet.20170101.11

Received: July 13, 2016; **Accepted**: November 28, 2016; **Published**: December 30, 2016

Abstract: The moisture adsorption and thermodynamic properties of sorghum-based complementary foods were investigated. Non-fermented and fermented sorghum, crayfish, Mango mesocarp and fluted pumpkin leaf powders were blended in the ratios of 91.06% non-fermented sorghum: 0.17% mango mesocarp: 8.77% fish (NFSMC), 91.06% fermented sorghum: 0.17% mango mesocarp: 8.77% fish (FSMC), 91.04% non-fermented sorghum: 0.19% fluted pumpkin: 8.77% fish (NFSPC) and 91.04% fermented sorghum: 0.19% fluted pumpkin: 8.77% fish (FSPC). The sample formulations were done based on 16% protein using material balance. Established procedures were used for sample preparation and analyses. The equilibrium moisture contents (EMCs) generated through static gravimetric method was fitted with Guggenheim-Anderson-deBoer (GAB) model by polynomial regression analysis. The moisture adsorption isotherms of the samples exhibited sigmoidal shape (Type II). The enthalpy of monolayer ranged from 50.34 to 60.75kJ/mol, multilayer ranged from 43.83 to 45.89kJ/mol and bulk water ranged from 42.98 to 44.20kJ/mol. The isosteric heat of sorption decreased with increase in moisture content, the entropy of adsorption of NFSMC, FSMC and FSPC decreased as the moisture content increased. The isokinetic temperature ranged from 326.51 to 603.33K while the harmonic mean temperature was 297.78K. The adsorption process was enthalpy driven. Therefore, NFSMC, FSMC and NFSPC are recommended for their relatively lower moisture content.

Keywords: Sorghum, Fermentation, Crayfish, Isosteric Heat, Entropy, Water Activity

1. Introduction

Sorghum-based foods were formulated using sorghum, crayfish, mango mesocarp and fluted pumpkin flours for complementary food application. The need for combination of available local food materials for the production of complementary foods for improved protein and micronutrients to address the problem of malnutrition has been advocated by early researchers [1. 2]. The use of fermentation as an adaptable technology in the processing of some staple foods to improve on their nutritional and functional properties has been reported elsewhere in literature by several workers [3, 4, 5].

Moisture uptake has been implicated in the poor keeping quality of many dehydrated foods in the tropics due to poor packaging materials and the moisture levels at which they were prepared [6]. The shelf life of packaged food materials has been shown to be influenced by the temperature, relative humidity and moisture content and thus the water activity (a_w) of the material [7]. Moisture sorption isotherms, important tools for predicting interactions between the water and the food components, describe the relationship between water activity and equilibrium moisture content of a product at a given temperature. It depends on several factors, such as physical structure, chemical composition and water affinity. Therefore, determination of sorption isotherms for each material is necessary and their knowledge is important to various food processes, since they are used to estimate drying time, to predict ingredients behavior upon mixing, to select packaging, to model moisture changes that occur during storage and to estimate shelf life stability [8, 9]. Several mathematical models for description of moisture sorption are available in literature. They are based on theories on the sorption mechanism or are purely empirical or semi-empirical.

Thermodynamic properties (net isosteric heat, differential enthalpy, differential entropy, integral entropy and integral enthalpy) of food could be calculated from sorption isotherms and provide information about properties of water and permit estimation of energy requirement of drying process. The net isosteric heat of sorption or enthalpy of sorption (q_{st}) is used as an indicator of the state of adsorbed water by solid particles and is defined as the difference between the total heat of sorption (Q_{st}) and heat of vaporization of water (H_L) [8, 10]. Therefore, the knowledge and understanding of the moisture uptake (adsorption) and thermodynamic properties of the products is desirable to avoid their quality deterioration during storage. This study was aimed to evaluate the moisture adsorption characteristics and thermodynamic properties of complementary foods produced from blends of sorghum, crayfish, mango mesocarp and fluted pumpkin leaf powders.

2. Materials and Methods

2.1. Sample Procurement

About 10kg of red sorghum grains [*Sorghum bicolor*, (L) Moench] and 5kg of semi ripe mango fruits (a local variety) (*Mangifera indica*) popularly known as *Wua nyian* and *Chul kpev* in Tiv respectively, 1kg of crayfish (*Procambarus clarkii*) and fluted pumpkin leaves *(Telferia accidentalis)* each were sourced from a local market in Makurdi, Benue State. These materials were transported to the Department of Food Science and Technology, University of Agriculture, Makurdi for processing prior to product formulation and subsequent analysis.

2.2. Preparation of Sorghum Flour

About 10kg of sorghum was dehulled using rice huller (Model: Navin, Madras) and washed with tap water and sun dried for 12h at average relative humidity of 65%. The dehulled grains were milled using single disc attrition mill (Model: Asiko AII). The flour was sieved using a laboratory test sieve of 0.5mm aperture.

2.3. Solid State Fermented of Sorghum Flour

Fermentation of sorghum flour was carried out using the method of Sengev *et al.* [11] with modification. The milled sorghum flour was divided into two equal parts. One part was mixed with tap water in the ratio of 2:1w/v and allowed to ferment for 48h at 30 ± 1°C, relative humidity of 65% in none air-tight covered plastic tray. At the end of the fermentation, a pH of 3.80 was recorded. The fermented sorghum was dried in hot air oven at 70°C for 12h and milled into fine flour of 0.5mm.

2.4. Preparation of Mango Mesocarp Powder

The method described by Sengev *et al.* [11] was adopted with modification. Five kilograms (5kg) of partially ripe mango fruits, *Chul kpev* (a local variety) (pH=3.8, Brix=7.0, Refractive Index=1.34) were sorted, washed, peeled and the mesocarp was manually sliced to an average thickness of 2.5mm. The slices were spread on a tray covered with aluminum foil and oven-dried at 70 ± 1°C for 24h to a moisture content of about 10%. The slices were milled using a single disc attrition mill (Model: AII Asiko, Nigeria) and sieved through a 0.5mm sieve to obtain mango mesocarp powder (M).

2.5. Preparation of Crayfish Powder

The method of Onuorah and Akinjede [12] was adopted with modification. About 1kg of crayfish was washed to remove extraneous materials, sundried for 12h at relative humidity of 65%. The sample was milled using hammer mill (Model: Brook Crompton Series 2000, England) and sieved through 0.5mm laboratory test sieve instead of 0.6mm to obtain crayfish powder (C).

2.6. Preparation of Fluted Pumpkin Leaf Powder

Fluted pumpkin powder was prepared as described by Uboh *et al.* [13]. The fluted pumpkin leaves were washed with tap water, steam blanched for 3 sec. and dried under the shade to constant weight. The dried leaves were milled using a single disc attrition mill (Model: AII Asiko, Nigeria) and screen through a 0.5mm sieve to obtain fluted pumpkin leaf powder (P).

2.7. Blend Formulation

The formulation was carried out according to Sengev *et al* [14]. In this method, the blends were formulated based on protein and mass balance equations. Sorghum (fermented and non-fermented), crayfish, mango mesocarp and fluted pumpkin leaf powders were blended based on 16% protein level as shown in Table 1.

Table 1. *Formulation of Blends.*

Product	Ingredient mix (g/100g)			
	Sorghum Flour	Mango Mesocarp Powder	Fluted Pumpkin Leaf Powder	Crayfish Powder
NFSMC	91.06	0.17	-	8.77
FSMC	91.06	0.17	-	8.77
NFSPC	91.04	-	0.19	8.77
FSPC	91.04	-	0.19	8.77

Key: NFSMC=Non-Fermented Sorghum+Mango Mesocarp+Crayfish, NFSPC=Non-Fermented Sorghum+Fluted Pumpkin Leaf+Crayfish, FSMC=Fermented Sorghum+Mango Mesocarp+Crayfish, FSPC=Fermented Sorghum+Fluted Pumpkin Leaf+Crayfish

2.8. Measurement of Equilibrium Moisture Content and Water Activity

Equilibrium moisture content was determined gravimetrically by exposing the samples to atmospheres of known relative humidities following the method described by Ariahu *et al*. [15] with some modifications. Sulphuric acid (H_2SO_4) solutions of 10, 20, 30, 40, 50 and 60% were used to provide water activities ranging from 0.15 to 0.96 as described by Ruegg [16]. A thermostatically controlled biochemistry incubator (Model: SPX-80-II, Searchtech Instruments) and 500 mL plastic containers were used for temperature and humidity controls respectively. The solutions made from the acid (200mL each) were carefully introduced into the plastic containers. A screen made of wire gauze was arranged in the plastic containers above the acid solutions to provide a platform for the samples to rest.

2.8.1. Calculation of Equilibrium Moisture Content

The equilibrium moisture contents were determined by material balance from the initial moisture content using the equation below.

$$EMC = \frac{MW_1 + 100\left(W_3 - W_2\right)}{W_1 + \left(W_3 - W_2\right)} \tag{1}$$

M=Initial moisture content of the sample, W_1=Weight of sample used during sorption, W_2=Initial weight of sample and crown cork, W_3=Final weight of sample and crown cork at equilibrium and EMC=Equilibrium Moisture Content.

2.8.2. Modeling and Analysis of Sorption Isotherm Data

The relation between the equilibrium moisture content (%db) and the water activity of the samples was predicted using GAB model. This model was chosen due to its reported simplicity, versatility and physical application to foods [15]. The GAB model parameters were obtained using Microsoft Excel (2007) through polynomial regression method. The GAB model is presented thus:

$$\frac{M}{M_o} = \frac{GKa_w}{(1-Ka_w)(1-Ka_w+GKa_w)} \tag{2}$$

where G and K are constants related to the energies of interaction between the first and distant sorbed molecules at the individual sorption sites. Theoretically they are related to sorption enthalpies as follows:

$$G = G_o \exp\left[(\overline{H}_o - \overline{H}_m)/RT\right] = G_o \exp\left(\Delta\overline{H}_c\right)\Big/RT \tag{3}$$

$$K = k_o \exp\left[(\overline{H}_m - \overline{H}_l)/RT\right] = k_o \exp\left(\Delta\overline{H}_k\right)\Big/RT \tag{4}$$

where G_o and k_o are entropic accommodation factors; $\overline{H}_o$, $\overline{H}_m$ and $\overline{H}_l$ are the molar sorption enthalpies of the monolayer, the multiplayer and the bulk liquid, respectively. When K is unity, the GAB equation reduces to the BET equation

The goodness of fit of GAB model was evaluated using percent root mean square of error (%RMS) between experimental (M_{obs}) and predicted (M_{est}) moisture contents as described by Wang and Brennan [17].

$$\%RME = \sqrt{\frac{\sum\left[\frac{M_{obs} - M_{est}}{M_{obs}}\right]^2}{N}}\, x100 \tag{5}$$

Where N=number of experimental data.

2.9. Heat of Sorption

The net isosteric heat of sorption was calculated by applying Clausius-Clapeyron equation to the isosters obtained at constant moisture content following the procedure reported by Ariahu *et al*. [15]. By plotting ln (a_w) versus 1/T for a specific moisture content, ΔH_{st} was evaluated from the slope ($-\Delta H_{st}/R$). The differential entropy of sorption (ΔS^o) was also obtained from the intercept coefficient ($\Delta S^o/R$) of the same plot. Applying this at different moisture content allowed the dependence of ΔH_{st} and ΔS^o with moisture to be determined. The isokinetic temperature (T_o) and the Gibbs free energy (ΔG^o), were obtained from the slope and intercept respectively of the plot of ΔH_{st} versus ΔS^o.

$$\ln a_w = \frac{\Delta H_{st}}{RT} - \frac{\Delta S^O}{R} \tag{6}$$

Where a_w=Water activity, R=Universal gas constant (0.008314 kJ/mol K)

$$\Delta H_{st} = T_o \Delta S + \Delta G^o \tag{7}$$

Where T_o=Isokinetic temperature (K), ΔG^o=Gibbs free energy (J/mol), ΔS^o=Net isosteric entropy of sorption (KJ/mol)=S_s–S_1 with S_s and S_1 as entropy of sorption of the species and pure water respectively. In order to corroborate the compensation theory a statistical analysis test was carried out as proposed by Krug *et al.* [18]. The harmonic mean temperature (T_{hm}) was given as follows:

$$T_{hm} = \frac{n}{\sum_{i=l}^{n}\left(\frac{1}{T}\right)} \tag{8}$$

3. Results

3.1. Moisture Sorption Isotherms

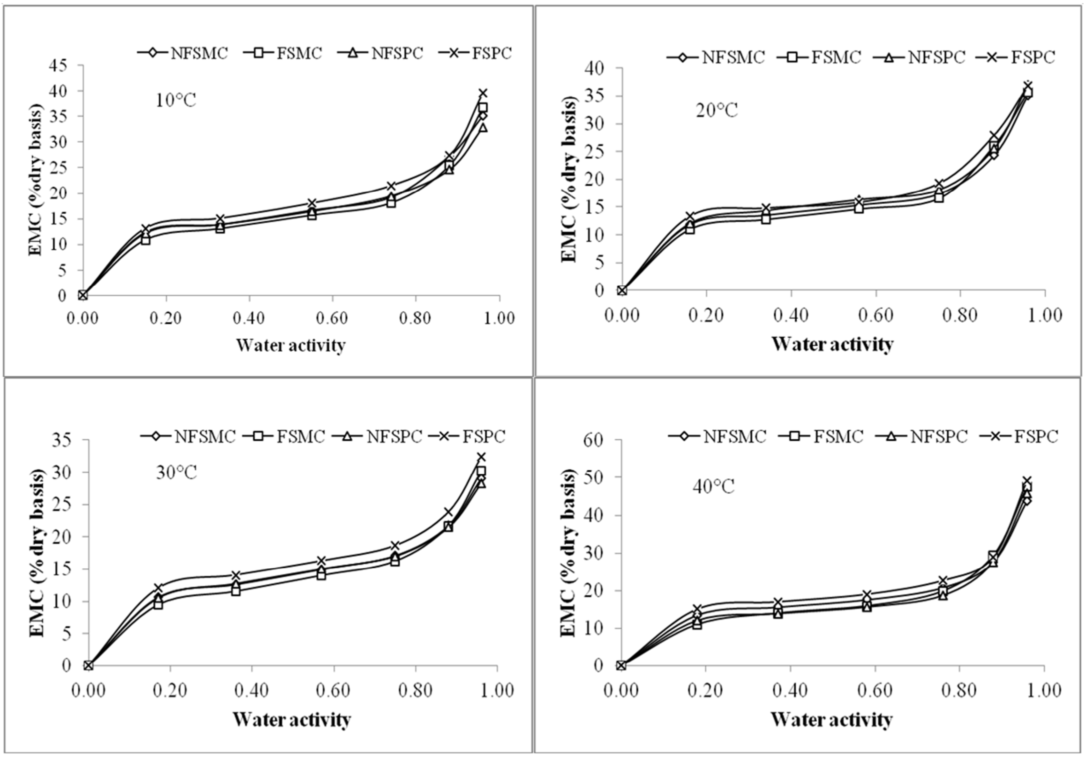
The moisture adsorption isotherms of NFSMC, FSMC, NFSPC, and FSPC are presented in Figure 1. The moisture sorption isotherms were sigmoidal corresponding to the type II isotherms. It was observed that the equilibrium moisture content (EMC) decreased with increase in water activity (a_w) at constant temperature. It was also observed that water activity decreased slightly with increase in temperature form 10–30°C and increased slightly at 40°C. The fermented sample containing fluted pumpkin leaves had higher EMCs while the fermented sample with mango mesocarp had the least EMCs between the a_w range of 0.15 to 0.76.

3.2. Goodness of Fit of GAB Model

The percent root mean square (%RMS) of GAB model tested in this study at temperature range of 10-40°C for moisture adsorption for NFSMC ranged from 6.11 to 8.86, NFSPC ranged from 5.90 to 8.95, FSMC ranged from 5.87 to 9.92 and FSPC ranged from 6.90 to 8.68.

3.3. GAB Adsorption Energetics

The results of GAB adsorption energetics of sorghum-crayfish-based complementary foods are presented in Table 2. The molar enthalpy of monolayer ($\overline{H}_o$) for NFSMC ranged from 52.81 to 54.03 kJ/mol, 59.53 to 60.75kJ/mol for NFSPC, 51.56 to 50.34 kJ/mol for FSMC and 60.15 to 61.37 kJ/mol for FSPC. The molar enthalpy of multilayer ($\overline{H}_m$) for NFSMC ranged from 43.79 to 45.01 kJ/mol, 44.67 to 45.89 kJ/mol for NFSPF, 44.30 to 45.52 kJ/mol for FSMC and 43.83 to 45.05 kJ/mol for FSPC. The molar enthalpy of vaporization of liquid ($\overline{H}_l$) water ranged from 42.98 to 44.20 kJ/mol for all the samples and the molar enthalpies for all the samples decreased with increase in temperature.



EMC=Equilibrium moisture content.

Figure 1. *Moisture adsorption isotherms of sorghum-based complementary foods.*

Table 2. *GAB Adsorption Enthalpy (kJ/mol) of Sorghum-based Complementary Foods.*

Sample	Temperature (°C)	$\overline{H}_o$	$\overline{H}_m$	$\overline{H}_l$
NFSMC	10	54.03	45.01	44.20
	20	53.66	44.64	43.83
	30	53.25	44.23	43.42
	40	52.81	43.79	42.98
FSMC	10	51.56	45.52	44.20
	20	51.19	45.15	43.83
	30	50.78	44.74	43.42
	40	50.34	44.30	42.98
NFSPC	10	60.75	45.89	44.20
	20	60.38	45.52	43.83
	30	59.97	45.11	43.42
	40	59.53	44.67	42.98
FSPC	10	61.37	45.05	44.20
	20	61.00	44.68	43.83
	30	60.59	44.27	43.42
	40	60.15	43.83	42.98

Key: $\overline{H}_o$, $\overline{H}_m$ and $\overline{H}_l$ are the molar sorption enthalpies of the monolayer, the multiplayer and the bulk liquid, respectively.

3.4. Evaluation of Adsorption Isosteric Heat

The Clausius-Clapeyron relationships between water activity (a_w) and absolute temperature produced isosters at constant equilibrium moisture content (EMC). The net isosteric heat of sorption generated from the isosters is presented in Figure 2 within the EMC range of 10-26%. The net isosteric heat ranged from 0.94 to-0.01kJ/mol for NFSMC, 3.58 to-1.86 kJ/mol for NFSPC, 8.00 to-2.38kJ/mol for FSMC and 0.57 to-1.50kJ/mol for FSPC.

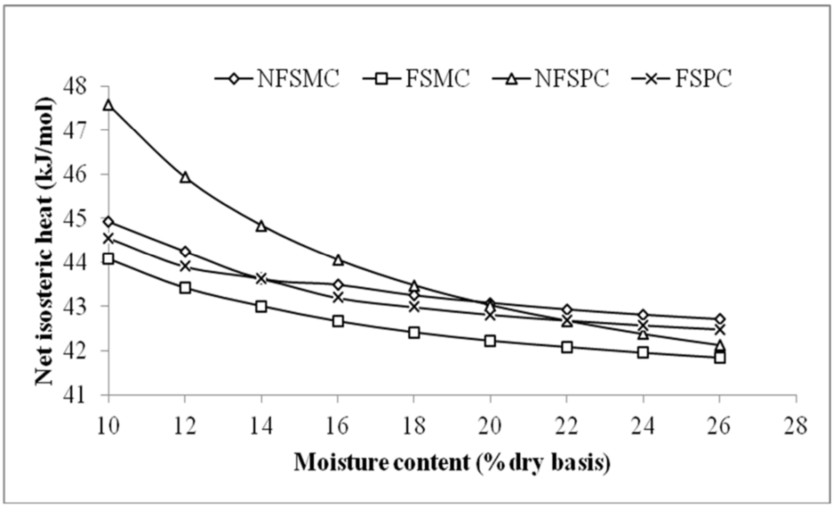


Figure 2. *Evaluation of isosteric heat of sorption of sorghum-based complementary foods.*

3.5. Evaluation of the Entropy of Adsorption

The evaluation of entropy of sorption with moisture content is presented in Figure 3. The entropies of NFSMC, FSMC and FSPC on their decreasing path almost converged at 14% moisture content and decreased gradually as the moisture content increased. The entropy of adsorption of NFSPC increased smoothly as the equilibrium moisture content (EMC) increased.

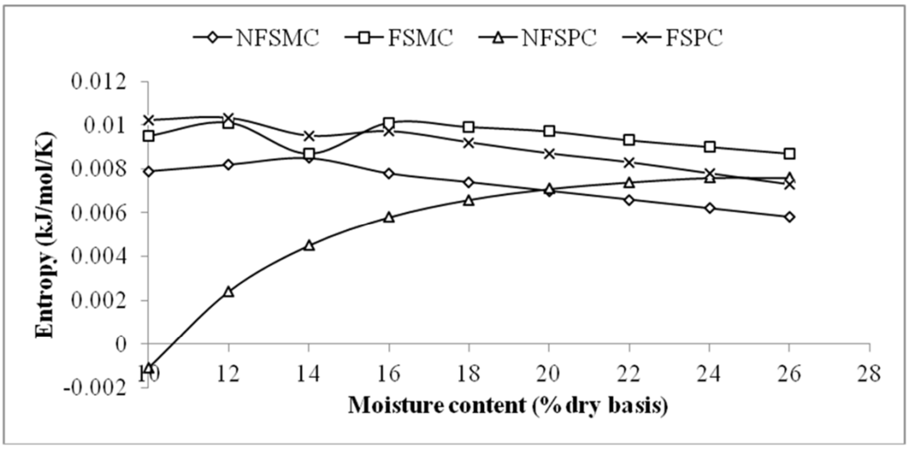


Figure 3. *Evaluation of the entropy of adsorption of sorghum-based complementary foods.*

3.6. Enthalpy-Entropy Compensation Parameters

The enthalpy-entropy compensation parameters of sorghum-based complementary foods are presented in Table 3. The isokinetic temperatures for NFSMC, NFSPC, FSMC and FSPC were 603.33, 602.76, 326.51 and 582.97K respectively while the gibbs free energy for adsorption were-4.92, 3.23,-4.39 and-6.03 kJ/mol for NFSMC, NFSPC, FSMC and NFSPC respectively. The harmonic mean temperature for the sorption process at 10, 20, 30 and 40°C was 297.78K. The coefficient of determination (r^2) ranged from 0.05 to 0.97.

Table 3. *Enthalpy-Entropy Compensation Parameters for Sorghum-Based Complementary Foods.*

Sorption Mode	Sample	T_β (K)	T_{hm} (K)	ΔG (kJ/mol)	r^2
Adsorption	NFSMC	603.33	297.78	-4.92	0.59
	FSMC	326.51	297.78	-4.39	0.05
	NFSPC	602.76	297.78	3.23	0.97
	FSPC	582.97	297.78	-6.03	0.77

Key: T_β=Isokinetic temperature (K), ΔG=Gibbs free energy, T_{hm}=Harmonic mean temperature (K), r^2=Coefficient of determination

4. Discussion

4.1. Effect of Water Activity and Temperature on the Moisture Sorption Isotherms

It was clearly observed from Figure 1 that for all isotherms, the EMC increased as a_w increased been more evident at a_w above 0.55. This is a common phenomenon in sorption and this trend is in agreement with the findings of Igbabul *et al.* [19] and Al-Mahasneh *et al.* [20] who reported an increase in EMC with a_w. All the adsorption isotherms of the samples exhibited sigmoidal shape for all the temperatures translating to the type II classification of sorption isotherms. The sigmoidal shape of the sorption isotherms has been reported numerous times for food materials in the literature [16]. The type II isotherm observed suggested that sorption occurred according to a multilayer mechanism throughout the equilibrium relative humidity range.

The equilibrium moisture content (EMC) of the blends decrease slightly as the temperature increased from 10 to 30°C. This trend is very common and may be explained by considering excitation states of molecules. As the temperature increases, the kinetic energy of water vapor molecules increase and this discourages their binding on the active sorption sites available on substrate [21]. On the contrary, the EMCs of the samples increased as temperature increased to 40°C. This could be due to product composition and faster dissolution of the food constituents at higher temperatures. Increased water binding at higher temperature has also been reported elsewhere for foods particularly rich in soluble solids and susceptible to structural orientations [22]. It was also reported that foods high in protein and sugar contents absorbed more water to overcome the negative effect of temperature at higher hydration levels [23]. Chowdhury and Das [24] also reported such an intersection or crossover behavior of edible films of blends of starch, amylose and methylcellulose. The EMC shift by temperature was mainly due to the change in water binding, dissociation of water or increase in solubility of solute in water, which is in close agreement with results quoted by Rahman [25]. This implies that at higher temperatures, sorghum-based complementary foods would become more hygroscopic and therefore spoilage becomes eminent.

The higher EMCs observed in FSPC isotherms under the temperatures investigated could be attributed to the addition of fluted pumpkin leaf powder. Earlier studies by Kajihausa *et al.* [26] and Bally [27] indicated that fluted pumpkin leafand mango fruit pulp contained 44.56% and 17.0% carbohydrate respectively. Hence, addition of flute pumpkin leaf powder would contribute more carbohydrate thereby increasing the sorptive sites of FSPC resulting to higher EMC even though fermentation has been reported [28] to decrease sorptive sites.

4.2. Goodness of Fitness of GAB Adsorption Model

The mean values of %RMS for GAB model were <10%. The lower the %RMS values, the better the adequacy of fit of the model. The RMS values of higher than 10% indicates a poor fitting ability. According to Wang and Brennan [16], percent root mean square of error (% RMS) values of ≤ 10% indicate a reasonably good fit for practical purposes.

4.3. GAB Adsorption Energetics

The enthalpies of adsorption of sorghum-based complementary foods revealed higher enthalpy at monolayer than multilayer. This is expected since monolayer moisture is strongly attached to the food matrix and therefore, the energy requirement of monolayer > multilayer > pure water. Kinsella and Fox [29] and Mohsenin [30] remarked that slightly higher enthalpy of vaporization was needed to remove the water at monolayer region. The enthalpies decreased with increase in temperature. This could be due to increase in the kinetics of water molecules at higher temperature.

4.4. Evaluation of Isosteric Heat of Adsorption

The net isosteric heat is defined as the heat in excess of the latent heat of vaporization of pure water. The sorption heat was considerably more at lower moisture content, and it decreased gently to approach the latent heat of vaporization of pure water (q_{st}=0) as the moisture content increased. The heat evolved during sorption of the first layer of water molecules was substantially more than the heat of condensation of free water. This could be due to strong interactions between these water molecules and the hydrophilic groups of the food solid. According to Wang and Brennan [17], the decrease in the isosteric heat with higher amounts of sorbed water can be quantitatively explained by considering that initially sorption occurs on the most active available sites, giving rise to high interaction energy. As these sites become occupied, sorption occurs on the less active ones, resulting in lower heats of sorption. At high water content, it tilts towards the heat of condensation of pure water [31]. In addition, the variation in heat of sorption with moisture content provides valuable data for energy consumption calculations and subsequent design of drying equipment, and knowledge of the extent of the water–solid versus water–water interactions [32].

4.5. Evaluation of the Entropy of Adsorption

The relationship between entropy and moisture content of NFSMC, FSMC and FSPC decreased as the moisture content increased, although the samples showed very low degree of disorderliness. This unconventional behavior could be ascribed to the presence of polymers which increased the affinity for water molecules and reduction in their degree of freedom. Thys *et al.* [33] reported that the entropy of sorption decreased with increased moisture content. This is contrary to observation of Ariahu *et al* [15] who remarked that the entropy of sorption increased as the moisture content increased. For NFSPC, the entropy of sorption increased gently and smoothly as the moisture content increased. This observation agreed with the findings of Ariahu *et al* [15].

4.6. Enthalpy-Entropy Compensation Parameters

The plot of net isosteric heat versus entropy of sorghum-based complementary foods exhibited a linear relationship for adsorption. The relationship gave r^2 values of NFSMC, NFSPC and FSPC within the acceptable range. Santhi *et al*. [34] and Van Liew *et al*. [35] reported that r^2 ranges from 0 to 1, with higher values indicating less error variance, and typically values greater than 0.5 are considered acceptable. The r^2 value of 0.05 is an indication of poor linearity and high error variance for FSMC.

The enthalpy-entropy compensation parameters revealed that the moisture sorption process was enthalpy driven since the isokinetic temperature was greater than the harmonic mean temperature ($T_\beta > T_{hm}$). The isokinetic temperature has an important physical meaning as it represents the temperature at which all reactions in the series proceed at the same rate [36]. The isokinetic temperature values reported in this study were higher than that reported by McMinn *et al* [37] who submitted that isokinetic of starch materials was in the range of 363.4 to 427.7k. The higher values could be attributed to the nature and composition of the food products.

From the concept of thermodynamics, the free energy (ΔG) is an indicative of the affinity of the sorbent water. It also provides a condition as to whether the sorption process will be spontaneous ($-\Delta G$) or non spontaneous ($+\Delta G$). Therefore, considering the values of ΔG presented in this study, it could be deduced that NFSMC, FSMC and FSPC were spontaneous while NFSPC was non spontaneous.

5. Conclusion

Moisture adsorption studies conducted on sorghum-based complementary foods indicated that the sorption isotherm were sigmoidal (Type II), equilibrium moisture content increased with increase in water activity and decreased with temperature. Enthalpies at monolayer regions were higher followed by multilayer and bulk water. The isosteric heat of adsorption of the samples decreased with increase in equilibrium moisture content. The entropy of adsorption of all the samples decreased as the equilibrium moisture content increased except for NFSPC. The enthalpy-entropy compensation revealed that the adsorption process was enthalpy driven. The EMCs of NFSMC, FSMC and NFSPC are relatively lower compared to FSPC and are therefore recommended since lower moisture content ensures shelf stability.

Acknowledgements

The authors deeply acknowledged the contributions of Mr. Julius Amove of the Department of Food Science and Technology, University of Agriculture, Makurdi for his contributions to the success of this work.

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