Moisture Adsorption Studies on Soy - Mumu Supplemented with Moringa Leaf Powder

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Abstract: Moisture adsorption Studies was carried out on Soy-mumu, a Nigerian cereal-based food product supplemented with 0, 10, 15 and 20% Moringa leaf powder at four different temperatures (20°C, 30°C, 40°C and 50°C) using the standard static gravimetric method. The different blends of Soy-mumu products were subjected to water activities of 0.1 – 0.957 for each of the four temperatures (20°C, 30°C, 40°C and 50°C) and data obtained were analysed using Oswin, Henderson and GAB models while thermodynamics of moisture transfer were estimated using Clausius-Clapeyron and kinetic compensation equations. The moisture adsorption isotherms were sigmoid in shape (type II). The equilibrium moisture content (EMC) of the Soy-mumu products increased with increase in water activity at constant temperature and were lower as the temperature increased at constant water activity. The sorptive capacity of Soy-mumu products increased with increasing levels of Moringa leaf powder supplementation and exhibited higher isotherms. Using the percent root mean square of error (% RMS), Oswin model gave the best fit for describing the adsorption isotherms of the Soy-mumu products while GAB and Henderson models fitted poorly (<10% RMS). Monolayer moisture content (Mo) and surface area (So) of adsorption decreased with increase in temperature. The GAB monolayer moisture contents were higher than BET monolayer moisture contents. The isosteric heat of sorption generally decreased as the moisture content increased. The maximum isosteric heat (ΔHst) was 14.1928; 14.1928; 37.0272 and 57.9120kj/mol for samples that were supplemented with 0, 10, 15 and 20% Moringa leaf powder respectively. The enthalpy-entropy compensation theory is suitable for describing the moisture adsorption phenomenon of the Soy-Mumu products.

Keywords: Soy-Mumu, Moisture Adsorption, Moringa Leaf Powder

1. Introduction

Mumu is a Nigerian cereal-based food product processed from roasted sorghum, millet or maize and consumed particularly by Tiv people (adults and children). Soy-mumu is a protein enriched mumu processed by blending cereal with soybeans. Blending of maize with soybean results to a product of higher nutritional value in terms of protein but low in micronutrient content [1]. This may lead to malnutrition problems. The product has gained a lot of attention among the over four million Tiv people and therefore, there is need to supplement it with locally available micronutrient sources, optimization of its processing and storage requirements to enhance quality, hence, the need for sorption studies.

A moisture sorption isotherm describes the relationship between the water activity (aw) and the equilibrium moisture content (EMC) of a food product at constant pressure and temperature. The knowledge and understanding of moisture sorption isotherms is of great importance in Food Science and Technology to solve many problems such as the design of equipment, optimisation of processing, assessing packaging problems, for modelling moisture changes which occur during drying, for predicting shelf life stability, for ingredient mixing predictions etc. [2]. The knowledge of moisture adsorption isotherms of soy-mumu supplemented with Moringa leaf powder is therefore needed for various processing applications.
Knowledge of the monolayer moisture content ($M_0$) of the soy-mumu which is regarded as moisture content for maximum stability of a dehydrated product in addition to Isopteric heat of sorption which is defined as the total heat of sorption of water from the material minus the heat of vaporisation of water [3] are essential for estimating the drying time and energy requirements for dehydration. Such information would also be helpful in the design of drying equipment. For effective packaging of the soy-mumu products supplemented with Moringa leaf powder, the critical equilibrium moisture content and relative humidity are needed and such information can be obtained from moisture sorption isotherms.

Various mathematical models have been proposed in literature to describe sorption isotherms [4]. Some were developed with a theoretical basis to describe adsorption mechanisms (e.g., GAB and BET) [5] whereas others are just empirical or a simplification of more elaborate models. Chirife at al reviewed 23 theoretical and experimental isotherms equations, and they are used for fitting sorption isotherms of food products [6]. None of these equations described accurately the sorption isotherm over the whole range of relative humidity and for different types of food materials. Labuza noted that no sorption isotherms model could fit data over the entire range of relative humidity because water is associated with food matrix by different mechanisms in different activity regions [7].

The objectives of this work were; to obtain adsorption isotherms at four temperatures (20°, 30°, 40° and 50°C) for Soy-mumu supplemented with Moringa leaf powder, to determine the effect of temperature, water activity and Moringa leaf powder supplementation on the sorption isotherms of the soy-mumu products, determine the goodness of fit of GAB, Henderson and Oswin sorption models to the experimental data and calculate the isosteric heat of sorption of the soy-mumu products.

2. Materials and methods

2.1. Sources of Raw Materials

White Maize (Zea mays) and light yellow variety of soybeans (Glycinemax) were purchased from North Bank Market, Makurdi. Matured Moringa oleifera leaves were obtained from Moringa trees on University of Agriculture, Makurdi farms.

2.2. Sample Preparation

2.2.1. Preparation of Roasted Maize Flour

Roasted maize flour was prepared according to the method described by [1] with slight modification (without fermentation of the grains). Maize grains were sorted and winnowed to remove grain stalk, sticks and remaining cob parts. The grains were further subjected to visual screening to remove foreign particles such as stones. This was followed by washing with water to remove dust, soil particles and any over floats. Damaged, diseased or discoloured grains as well as immature or sprouted grains were discarded. Cleaned maize grains were cooked in two times water (1:2 w/v) for 45min, allowed to cool and drained of excess water for 15min. prepared maize grains were oven roasted at 150°C for 60min. The roasted grains were kept under silica gel to avoid moisture absorption until when required for milling and mixing for formulation of blends. A hammer mill was used to mill the roasted grains and a sieve of 0.5mm was attached to collect the milled product.

2.2.2. Preparation of Roasted Soybean Flour

Soybeans for supplementation were prepared by the methods described by [1]. Dry whole soy-beans were soaked overnight in tap water solution of 0.5% NaHCO$_3$ (1:3 w/v) after sorting and washing. After soaking, the beans were dehulled, tied loosely in bags of fine muslin and blanched at 100°C for 30 minutes in fresh tap water solution of 0.5% NaHCO$_3$ (1:3 bean: solution ratio). The blanched beans were drained and further rinsed with tap water and held for 1 hour in order to allow for moisture equilibration within the beans. This was followed by drying at 60°C for 6 hours in accordance with the method described by [1] for the production of roasted soybeans. Dry roasting was achieved by use of air-oven instead of coffee roasters as reported by [1]. The soy beans were browned at 150°C for 30 minutes. The roasted soybeans were cooled and stored under silica gel in a desicator (to avoid moisture absorption) until when required for milling and mixing. A hammer mill was used to mill the roasted soy-beans and a sieve of 0.5mm was attached to collect the milled product.

2.2.3. Moringa Leaf Powder Preparation

The Moringa Leaf powder was prepared according to the methods described by [8]. About 1 kg of Matured and fresh Moringa leaves were plucked from the Moringa tree. Diseased and damaged leaves were discarded manually just after collection of the fresh leaves. Collected leaves were washed in trough using clean potable water to remove dirt. Washed leaves were soaked in 1% Saline solution (Sodium Chloride) for 5 minutes to remove microbes and finally rinsed in clean potable water. The washed leaves were drained of excess water by spreading them on perforated trays for 15 minutes before taking them for drying. The washed and drained Moringa leaves were dried by spreading them on sterile clean net mesh in a well- ventilated room (at an ambient temperature of 30°C) for a drying period of 16 days with frequent turning with hand to ensure uniform drying. Crushing of the dried Moringa leaves was carried out using mortar and pestle and sieved using 0.5mm sieve screen. The sieved powder was then packaged in air-tight plastic containers and stored at ambient temperature (30°C) until when needed for blending.

2.3. Formulation of Blends

Four different blends, A, B, C and D were formulated using different ratios: sample A was comprising 85% roasted maize flour, 15% roasted soy-bean flour and 0% Moringa
leaf powder which served as the control; sample B comprising 75% roasted maize flour, 15% roasted soy-bean flour and 10% Moringa leaf powder; sample C comprising 70% roasted maize flour, 15% soy-bean flour and 15% Moringa leaf powder and sample D comprising 65% roasted maize flour, 15% roasted soy-bean flour and 20% Moringa leaf powder.

2.4. Determination of Adsorption Isotherms

Equilibrium Moisture Contents (EMCs) were determined using the standard static gravimetric method as described by [3]. Sulphuric acid solutions of 10%, 20%, 30%, 40%, 50% and 60% were used to provide water activities ranging from 0.08 - 0.957 as described by [9]. About 200ml of each acid solution was introduced into 500mL airtight plastic containers. In each plastic container, wire gauze was forced into place over the sulphuric acid solution to form support for the samples.

Duplicate samples (0.5g each) of dried product were weighed in crown corks and placed on the wire gauze above the solution for adsorption. The containers were covered tightly and placed in the incubator at selected temperatures of 20°C, 30°C, 40°C and 50°C. Small glass bottles (bijl bottles) containing toluene were placed in the containers to prevent mould growth at relative humidity above 50% [10].

The samples were removed and weighed every 2 days using electronic balance (ADAM AAA 160I) until difference between consecutive readings were <0.5% of sample weight.

The total time for removal and putting back in the airtight containers was about 2-5 minutes as recommended by the cooperative project cost 90 on physical properties of foods [2].

The equilibrium moisture contents were determined according to [12] by material balance from the initial moisture content (9.340, 9.400, 9.760 and 10.400 for sample A, B, C and D respectively).

2.5. Moisture Sorption Models

The equilibrium moisture data were fitted using the BET, GAB, Henderson and Oswin models. These models were chosen for their versatility, relatively simple mathematical computations and their reported fits for starchy foods and vegetables [3]. Monolayer moisture contents were evaluated using the BET ($a_w$ up to 0.5) and GAB ($a_w$ up to 0.9) models.

The Brunauer-Emmett-Teller (BET) equation is the most important popular due to its thermodynamic base [13]. It fits experimental sorption data in the range of water activities of approximately 0.05-0.45 [14].

The model is presented in the form:

$$\frac{aw}{(1-aw)M} = \frac{1}{M_o} + \frac{(C-1)aw}{M_o C}$$

Where,

- $M$ = equilibrium moisture content (gH$_2$O/100g solids),
- $M_o$ = monolayer moisture content (gH$_2$O/100g solid),
- $C = a$ constant related to heat of sorption for monolayer adsorption and

$a_w =$ water activity

The Guggenhein-Anderson-De Boer (GAB) model is a three parameter equation derived independently by [15], [16], [17] based on the BET theory. The GAB isotherm was used to fit sorption isotherm up to water activities of 0.9 in many cases [14]. The GAB equation has been applied successfully to various foods and has been recommended by the European project cost 90 on physical properties of foods [2].

The model is mathematically expressed as:

$$M = \frac{MoGka_w}{(1-ka_w)(1-ka_w + Gka_w)}$$

Where $G$ and $k$ are constants related to the energies of interaction between the first and distant sorbed molecules at individual sorption sites. The constant $G$ and $k$ are temperature dependent and are represented in Arrhenius type relationship as follows:

$$G = G^o \exp \left( \frac{H_m - H_n}{RT} \right)$$

$$K = k^o \exp \left( \frac{H_L - H_n}{RT} \right)$$

Where $G^o$, $k^o$ = entropic accommodation factors; $H_m$ = molar sorption enthalpy of the monolayer, $H_n$ = molar sorption enthalpy of the multilayer on top of the monolayer, $H_L$ = molar sorption enthalpy of the bulk liquid; $T = $ absolute temperature; and $R = $ universal gas constant.

The Henderson equation is one of the earliest models describing the effect of temperature on moisture sorption isotherms [18].

The Henderson equation is expressed as follows:

$$\ln (1-a_w) = -ATM^B$$

Where $A$ and $B$ are constants, $T = $ temperature ($^\circ$K)

Oswin [19] developed an empirical sorption isotherm model equation based on Pearson’s expansion for sigmoid curves applied to type II isotherms.

The Oswin equation is given as:

$$M = A \left[ \frac{a_w}{1-a_w} \right]^B$$

Where $M = $ equilibrium moisture content, $A$ and $B$ are constants $a_w =$ water activity.

The Oswin equation has been found to be a good fit model for the sorption of various food products and gave the best fit for non-proteinous, starchy and starchy related foods [20, 21]

2.6. Sorption Data Analysis

From the data generated, equilibrium moisture content versus water activities for each temperature was plotted. The acquired sorption data were analyzed using BET, GAB, Henderson and Oswin models. The parameters of BET,
Henderson and Oswin were obtained by least square linear regression from their respective equations. GAB parameters were obtained using Least Square non-linear regression using water analyzer series version 97.2 software. The goodness of fit of the different models used were evaluated using percentage root mean square of error (%RMS) between the experimental (\(M_{\text{obs}}\)) and predicted (\(M_{\text{est}}\)) moisture content as described by [22, 23] using the equations:

\[
%\text{RMS} = \sqrt{\frac{1}{N} \sum_{i=1}^{N} \left( \frac{M_{\text{obs}} - M_{\text{est}}}{M_{\text{obs}}} \right)^2} \times 100
\]  

(7)

Where %RMS = percentage root mean square of error, \(M_{\text{obs}}\) = observed (experimental) moisture content, \(M_{\text{est}}\) = estimated (predicted) moisture content, \(N\) = number of experimental data.

The BET monolayer values (Branauer et al., 1938) were calculated from the regression equation of the BET plot using the moisture sorption data up to 0.5 \(a_w\) and GAB monolayer moisture values were calculated from the data in the \(a_w\) range of 0.1 to 0.9.

3. Results and discussion

3.1. Moisture Adsorption Isotherms

The adsorption isotherms of soy-mumu products supplemented with Moringa leaf powder at 20, 30, 40 and 50 °C are shown in figure 1. The moisture adsorption isotherms were sigmoid in shape and conformed to type II classification which is typical of most food materials[24]. The type II isotherm is divided into three different parts; the first part (\(a_w = 0-0.22\)) is the monolayer region in which the water is bounded by hydrophilic water ion and is the most strongly adsorbed immobile and depends on the chemical composition of the products.

The second region (\(a_w = 0.22-0.73\)) is the linear portion and represents the water held by the material in the matrix.

The third is the concave shape (\(a_w = 0.73-1.0\)) and represents the least strongly bound water and the most mobile and is called bulk phase water [25, 26]. Figure 1 showed that at constant temperature, equilibrium moisture content increased with increase in water activity and decreased with increasing temperature at constant water activity. This can be explained by the higher active state of water molecules at higher temperatures thereby decreasing the attractive forces between them [4]. It can also be observed that at any given moisture content there was a consistent shift of equilibrium relative humidity to higher values with increase in temperature. This implies that at any given RH, Moringa-Soy-mumu products become less hygroscopic with increase in temperature. This means that in the storage of the products in an atmosphere of constant RH, the products can absorb more moisture at lower temperature than higher temperatures.

The water activity shift of food isotherms at constant moisture content with respect to temperature changes has been shown to be directly related to the rates of food deteriorative reactions [27-28]. Similar trends were observed for Tacca Inolucrata tuber products by [3].

3.2. Effect of Temperature on Moisture Adsorption Isotherms of Soy-Mumu Supplemented with Moringa Leaf Powder

Products with higher levels of Moringa leaf powder showed higher MSIs indicating higher EMCs. This observation indicates that the blending of soy-mumu with Moringa leaf powder could have affected the sorptive capacities of the powder (flour). This implies that the change in composition has effect on the moisture sorption isotherms as documented by [29].
Fig. 1. Moisture adsorption isotherms of soy-mumu products at 20°C, 30°C, 40°C and 50°C.

KEY: A = 85% Roasted maize flour: 15% Roasted Soy-bean flour: 0% Moringa leaf powder, B = 75% Roasted maize flour: 15% Roasted Soy-bean flour: 10% Moringa leaf powder, C = 70% roasted Maize flour: 15% Roasted Soy-bean flour: 15% Moringa leaf powder and D = 65% Roasted Maize flour: 15% Roasted Soy-bean flour: 20% Moringa leaf powder.

3.4. Goodness of Fit of the Sorption Models

The goodness of fit of the various Soy-Mumu products was determined using the Root Mean Square of error (%RMS). A good fit of an isotherm is assumed when the %RMS values is less than 10%.

The %RMS of the various models and their goodness of fit is presented in Table 1. The three models yielded different %RMS. Oswin best described the product with 10% Moringa leaf powder (sample B) 15% (sample C) and 20% (sample D) Moringa leaf powder supplementation. The result of this study therefore agrees with [3], who suggested that Oswin model might be better for non-proteinaceous materials and best for starchy and starchy related foods. From Table 1, the values of %RMS for GAB and Henderson were far above 10% and so considered poor fit.

<table>
<thead>
<tr>
<th>Sample</th>
<th>Equation</th>
<th>%RMS</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>20°C</td>
<td>30°C</td>
</tr>
<tr>
<td>GAB</td>
<td>25.07</td>
<td>27.13</td>
</tr>
<tr>
<td>GAB</td>
<td>8.22</td>
<td>11.30</td>
</tr>
<tr>
<td>GAB</td>
<td>22.62</td>
<td>21.38</td>
</tr>
<tr>
<td>GAB</td>
<td>11.50</td>
<td>8.65</td>
</tr>
<tr>
<td>GAB</td>
<td>6.11</td>
<td>3.97</td>
</tr>
<tr>
<td>GAB</td>
<td>22.30</td>
<td>26.27</td>
</tr>
<tr>
<td>GAB</td>
<td>7.77</td>
<td>15.39</td>
</tr>
<tr>
<td>GAB</td>
<td>4.29</td>
<td>8.58</td>
</tr>
<tr>
<td>GAB</td>
<td>22.16</td>
<td>24.15</td>
</tr>
<tr>
<td>GAB</td>
<td>8.11</td>
<td>14.23</td>
</tr>
<tr>
<td>GAB</td>
<td>4.02</td>
<td>6.64</td>
</tr>
</tbody>
</table>

Table 1. Goodness of Fit of soy-mumu products to the sorption Models.

3.5. Monolayer Moisture Content

The BET and GAB models were used to estimate the monolayer moisture contents as shown in Table 2. The BET equation gave lower monolayer moisture contents values than those obtained from GAB model. It was observed that in both BET and GAB models, monolayer moisture contents decreased with increase in temperature for all the soy-mumu products except for sample A at 50°C (BET model) and sample D at 40°C (for GAB model). The general trend of decrease in \( M_0 \) with increase in temperature has been reported by several workers [24] and [3]. The decrease in monolayer moisture content as observed in the study for soy-mumu products may be due to a reduction in the total number of active sites for water binding as a result of physical and chemical changes in the product induced by temperature [23]. It could also be that with increase in temperature the water molecules got activated because of increase in their energy level thereby causing them to become less stable and to break away from the water binding sites of the food material thus resulting to decrease in the monolayer moisture contents.

Table 2. GAB and BET Monolayer Moisture contents.

<table>
<thead>
<tr>
<th>Temperature (°C)</th>
<th>Sample</th>
<th>GAB (M_0) (gH(_2)O/100g solids)</th>
<th>BET (M_0) (gH(_2)O/100g solids)</th>
</tr>
</thead>
<tbody>
<tr>
<td>20</td>
<td>A</td>
<td>7.0542</td>
<td>5.2577</td>
</tr>
<tr>
<td></td>
<td>B</td>
<td>8.7881</td>
<td>6.6344</td>
</tr>
<tr>
<td></td>
<td>C</td>
<td>10.0537</td>
<td>7.2821</td>
</tr>
<tr>
<td></td>
<td>D</td>
<td>12.5888</td>
<td>8.7009</td>
</tr>
<tr>
<td>30</td>
<td>A</td>
<td>4.8402</td>
<td>4.1702</td>
</tr>
<tr>
<td></td>
<td>B</td>
<td>7.9357</td>
<td>5.6900</td>
</tr>
<tr>
<td></td>
<td>C</td>
<td>6.0687</td>
<td>4.6957</td>
</tr>
<tr>
<td></td>
<td>D</td>
<td>7.3895</td>
<td>5.6071</td>
</tr>
<tr>
<td>40</td>
<td>A</td>
<td>5.7950</td>
<td>3.6887</td>
</tr>
<tr>
<td></td>
<td>B</td>
<td>7.3977</td>
<td>5.1391</td>
</tr>
</tbody>
</table>
**3.6. Net Isosteric Heats**

The net isosteric heat of sorption (Table 3) obtained in the moisture range of 6.0–20.0 g H₂O/100 g solids for the adsorption process, decreased rapidly with increasing moisture, passed through maxima and gradually decreased to adsorption process, decreased rapidly with increasing moisture range of 6.0–20.0g H₂O/100 g solids except for sample B with 10 percent *Moringa* leaf powder which did not follow the trend strictly.

Hossain [13] also reported decrease in isosteric heat with amount of water sorbed, can be qualitatively explained considering the fact that initially, sorption occurred on the most active available sites giving rise to greatest energy interaction.

As these sites became occupied, sorption occurred on the less active site resulting to lower heats of sorption [23]. Water sorption in foods is such a complex phenomenon; the main water sorption constituents of foods are various polymers such as proteins, starch, cellulose, hemicellulose, chitin etc. The different polar groups of the polymers provide energetically different preferential sites for water. Besides as a polymer sorbs water, it undergoes changes in constitution, dimensions and properties [3].

<table>
<thead>
<tr>
<th>Sample</th>
<th>Regression Parameters</th>
<th>Moisture Content (g H₂O/100g solids)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>6.0</td>
</tr>
<tr>
<td>A</td>
<td>N</td>
<td></td>
</tr>
<tr>
<td></td>
<td>R²</td>
<td>0.9503</td>
</tr>
<tr>
<td></td>
<td>ΔS</td>
<td>0.0334</td>
</tr>
<tr>
<td>B</td>
<td>R²</td>
<td>0.3280</td>
</tr>
<tr>
<td></td>
<td>ΔS</td>
<td>-0.0106</td>
</tr>
<tr>
<td>C</td>
<td>R²</td>
<td>0.7324</td>
</tr>
<tr>
<td></td>
<td>A</td>
<td>12.460</td>
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<td></td>
<td>B</td>
<td>-4453.6</td>
</tr>
<tr>
<td>D</td>
<td>R²</td>
<td>0.1036</td>
</tr>
<tr>
<td></td>
<td>B</td>
<td>-6965.6</td>
</tr>
<tr>
<td></td>
<td>ΔS</td>
<td>0.1697</td>
</tr>
</tbody>
</table>

KEY: A =85% Roasted maize flour: 15% Roasted Soy-bean flour: 0%Moringa leaf powder, B =75% Roasted maize flour: 15% Roasted Soy-bean flour: 10% *Moringa* leaf powder, C =70% roasted Maize flour: 15% Roasted Soy-bean flour: 15%Moringa leaf powder and D=65% Roasted Maize flour: 15% Roasted Soy-bean flour: 20% *Moringa* leaf powder, N= number of experimental data, R²= coefficient of determination, ΔHₘₑₜ= net isosteric heat of adsorption (KJ/mol), ΔS = entropy of adsorption (KJ/mol).

**4. Conclusion**

1. The moisture adsorption Isotherms of the Soy-mumu products was sigmoid in shape, conforming to type II isotherm pattern which is typical of most food materials with equilibrium moisture contents decreasing with increasing temperatures at constant water activity and increasing with increase in water activity at constant temperature.

2. Monolayer moisture contents determined from BET and GAB models and surface area of adsorption determined from the BET model were found to decrease with increasing temperatures.
(3) Using the percent root mean square of error (%RMS). Oswin model gave the best fit for describing the adsorption isotherms of the Soy-mumu products while GAB and Henderson models fitted poorly.

(4) Sorptive capacity of Soy-Mumu products increased with increasing levels of Moringa leaf powder supplementation.

(5) The enthalpy-entropy compensation theory is suitable for describing the moisture adsorption phenomenon of the Soy-Mumu products.

(6) The heats of sorption decreased with increase in moisture content.

References


