Water vapor adsorption characteristics of starch—albumen powder and rheological behavior of its paste

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Available online 28 May 2015

Abstract

This paper concerns the water vapor adsorption properties of starch-albumen powder (SAP), as a new product with many potential food processing applications. The adsorption data were generated at some practical storage temperatures (27, 35 and 40 °C) and water activities, \(a_w\) (0.11–0.86) using gravimetric method. The data were fitted to some isotherm models (GAB, Peleg, DLP and BET). The values of some thermodynamic parameters based on the Clausius–Clapeyron equation were also calculated. The rheological behavior of SAP paste was also studied at 20, 30, 45 and 60 °C. SAP was found to have type II isotherm shape and is highly hygroscopic. The adsorption data were better fitted by Peleg and DLP models \((r^2 = 0.987–0.999)\). Monolayer moisture capacity ranged between 4.9 and 6.8 g/100 g solid. The water vapor sorption process in SAP is thermodynamically non-spontaneous and enthalpy-driven. SAP paste showed a characteristic shear-thinning behavior. Activation energy of flow \((E_a)\) was found to be 31.63 kJ/mol.

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Keywords: Starch-albumen powder

1. Introduction

Development of new and modified food ingredients requires certain pragmatic approaches to ensure low cost of production and optimal functionality of the ingredient in the prospected food products. Starch–albumen powder (SAP) is a new composite food powder developed by our research group as an additional way of preserving liquid egg white. Addition of starch to liquid egg white was done to increase the total solid content of the mixture prior air drying of the mixture in order to reduce drying time and energy cost. It was then hypothesized that SAP would combine the high hydration capacity of egg white proteins and high swelling capacity of starch in a single product to make a novel functional ingredient.

Thus, the functional properties of SAP as affected by drying temperature and starch content were studied and reported in a previous paper (Shittu et al., 2008). Increased starch content of the SAP significantly reduced the foaming, water absorption, and emulsifying capacities of SAP whereas the gelation capacity was increased. On the other hand, increasing air drying temperature from 40 to 60 °C resulted in SAP with higher foaming capacity. The study further established that it is possible to preserve egg white in powdered form using a simple and low-cost convective air drying technology compared with the conventional spray drying technology. Also, the product’s characteristics indicate that it may be useful as functional ingredient in diverse food product applications such a breading material in fried food, as basal weaning food and as ingredient in some baked products.

Food powders experience gradual or rapid water vapor adsorption when packaged and stored under highly humid conditions. This further brings about various deteriorative changes such as microbial degradation, reduced flowability (or caking) and dissolution depending on their physical property and composition. These changes greatly affect the acceptability of the products by their consumers and end users.

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Peer review under responsibility of Association of Vice-Chancellors of Nigerian Universities.

http://dx.doi.org/10.1016/j.nifoj.2015.04.014
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Water vapor sorption isotherms are graphical plots of the equilibrium moisture content achievable by food materials when kept under a constant temperature but different relative humidity condition of food material. Sorption isotherms have been used as tools to determine the shelf stability of food materials. Therefore, to design an appropriate packaging and storage facility for food powders, their water vapor adsorption characteristics must be understood.

The water vapor adsorption by solid materials is due to binding of water molecules to the hydrophilic, charged and polar groups present in them. The hydroxyl groups in the glucose units of starch molecules serve as the binding sites for the hydrophilic, charged and polar groups present in them. The hydroxyl groups in the starch molecule. The ratio of the linear (amylose) to the branched (amylopectin) components of starch also affect its crystallinity which also influence its water vapor adsorption characteristics (Chaudhary and Adhikari, 2013). According to Lewicki (1997), when biopolymers (starch, sodium caseinate, and cellulose) were mechanically mixed together, the water adsorption characteristics would therefore depend on the number of free adsorptive sites present in the starch molecule. The ratio of the linear (amylose) to the branched (amylopectin) components of starch also affect its crystallinity which also influence its water vapor adsorption characteristics (Chaudhary and Adhikari, 2013). According to Lewicki (1997), when biopolymers (starch, sodium caseinate, and cellulose) were mechanically mixed together, the water adsorption characteristics of their mixtures followed additive law at lower a_w values. However, at higher a_w values, the adsorption mechanism became more complex and ceased to follow additive law. The complexity of water adsorption mechanism could possibly be due to anyone of molecular matrix swelling, conformational changes, polymer–polymer interactions, crosslinking, co-operative binding and multiple hydrogen bond formation or their combination.

Starch powders have been reported to exhibit type II isotherm curves (Al-Muhtaseb et al., 2004; Peng et al., 2007) while pure protein powders mainly exhibit type III isotherm curves (Ayranci and Duman, 2005; Jovanovich et al., 2003) according to BET classification (Brunauer et al., 1938). The actual sorption behavior of starch–albumen powder (SAP), which is a conjugate product but not a mechanical mixture of the two biopolymers, is however unknown. It would therefore be of practical interest to determine the water vapor adsorption behavior of this new product to assist in designing appropriate storage and packaging systems useful to extend its shelf life. A previous study had also shown that conjugation of egg white proteins with some carbohydrate polymers had varied influence on its foam forming properties (Ptaszek et al., 2014).

This study was basically conducted to generate water vapor adsorption data of SAP at three temperatures (20, 30, and 40 °C). Three mathematical models (GAB, Peleg and BET) were also tested for fitting the sorption data. Some thermodynamic properties of the product were also determined using well established thermodynamic functions. Some basic rheological properties of SAP paste were also reported.

2. Materials and methods

2.1. Production of starch–albumen powder

The albumen was obtained from freshly laid poultry eggs. The yolk was carefully separated from the albumen and then the albumen was mixed gently without foaming with native cassava starch at a weight ratio of 15/85 (w/w) for cassava starch/albumen. The mixing was continued for about 60 s in a Kenwood laboratory size blender, poured on a flat tray to spread thinly and dried in a natural convective oven preset at 40 °C. The flakes obtained were then milled into powder.

2.2. Moisture adsorption characteristics

The gravimetric method under different temperature (20–40 °C) and relative humidity conditions (a_w ≈ 0.11–0.86) was used in generating the adsorption data. Saturated solutions of lithium chloride, potassium acetate, magnesium chloride, calcium nitrate, magnesium nitrate, sodium nitrite, sodium chloride and potassium chromate were used to maintain the respective relative humidity in the desiccators. Duplicates of each sample were presented for analyses. The temperature regulation was achieved using an incubator at accuracy of ± 1 °C. About 2 g of starch substituted egg white powder samples (dried at different temperature) were accurately weighed into the petri dishes and left above the saturated salt solution. After equilibrium weight was reached, each sample was dried at constant temperature of 105 °C for 4 h to determine the equilibrium moisture content.

2.3. Modeling of adsorption isotherms

Data obtained from the above were fitted into four isotherm models, namely, Peleg (Peleg, 1993), Double Log Polynomial (DLP) (Nurtama and Lin, 2010), Brunauer – Emmett – Teller (BET) (Brunauer et al., 1938) and Guggenheim-Anderson-de Boer (GAB) (Van den Berg and Bruin, 1981). Parameters k_1, k_2, n_1, and n_2 are constants in Peleg model (Eq. (1)) while b_1, b_2, b_3 and b_4 are constants in DLP model (Eq. (2)). Parameter M_0 in BET (Eq. (3)) and GAB model (Eq. (4)) is the monolayer moisture content, C and k are constants akin to the temperature effect on adsorption process. Model’s predictive accuracy was evaluated using two statistics, namely, deviation modulus (E %) and the root mean square error (RMSE) as shown respectively in Eq. (4) and (5). Residual plots of the equilibrium moisture content were also used as additional diagnostic for testing the degree of fit of the two long range isotherm models used. M_e and M_p are the experimental and predicted values of equilibrium moisture content at the set temperature and water activity conditions.

Peleg Model : \[ M = k_1a_w^{n_1} + k_2a_w^{n_2} \] (1)

DLP Model : \[ M = b_0 + b_1\ln(-\ln a_w) + b_2\ln(-\ln a_w)^2 + b_3\ln(-\ln a_w)^3 \] (2)

BET Model : \[ M = \frac{M_0Ca_w}{((1-a_w)(1+(C-1)a_w))} \] (3)

GAB Model : \[ M = \frac{M_0CKa_w}{((1-Ka_w)(1-Ka_w+CKa_w))} \] (4)

2.4. Application of thermodynamic models the adsorption data for starch–albumen powder

Clausius–Clapeyron equation (Eq. (6)) was used to calculate the net isosteric heat of sorption \( q_{st} \) with the following assumptions: (i) moisture content of the system remains constant and (ii) heat of vaporisation of pure water and excess heat of sorption do not change with temperature (Al-Muhtaseb et al., 2004).

\[
\frac{\partial \ln(a_w)}{\partial (1/T)} \bigg|_M = - \frac{q_{st}}{R}
\]

\( q_{st} = Q_{st} - h_{fg} \)

\( Q_{st} \) is the total heat of sorption, \( h_{fg} \) is the latent heat of vaporization, \( T \) is the absolute temperature (K), \( R \) is the universal gas constant (kJ/mol/K).

The change in molar differential entropy was calculated from Gibbs–Helmholtz equation (Telis et al., 2000):

\[
q_{st} = \Delta G - T S_d
\]

\( \Delta G = RT \ln a_w \)

\( \Delta G \) is the free energy (kJ mol\(^{-1}\)) and \( S_d \) is the differential entropy (kJ mol\(^{-1}\) K\(^{-1}\)). Combining Eqs. (8) and (9) gives a final relationship:

\[
\ln a_w = \frac{q_{st}}{RT} - \frac{S_d}{R}
\]

The values of \( q_{st} \) and \( S_d \) were calculated from the slope of the line when \( \ln(a_w) \) was plotted against \( 1/T \) at different constant moisture contents, \( (q_{st}/R) \) and the intercept \( (-S_d/R) \), respectively (McMinn and Magee, 2003). The enthalpy−entropy compensation theory assumes that a linear relationship exists between enthalpy and entropy as follows:

\[
q_{st} = T_{fi} S_d + \alpha
\]

The value of \( T_{fi} \), which is the isokinetic temperature (K) and \( \alpha \), which is the free energy at the isokinetic temperature were calculated from linear regression of \( q_{st} \) against \( S_d \) values. The isokinetic temperature \( T_{fi} \) represents the temperature at which all reactions in the series proceed at the same rate, and \( \alpha \) is a criterion to determine whether water vapor sorption process for material is a spontaneous \((-\Delta G\)) or non-spontaneous process \((+\Delta G)\).

The validity of the compensation theory was tested by comparing the isokinetic temperature \( (T_{fi}) \) with the harmonic mean temperature \( T_{hm} \) which is defined as

\[
T_{hm} = \frac{n}{\sum_{i=1}^{n} (1/T_i)}
\]

where \( n \) is the total number of isotherm.

2.5. Rheological property of SAP paste

The flow property of SAP paste determined using Brookfield Viscometer (Model DV-E, Brookfield Engineering Laboratory, UK). The paste was made by dispersing about 30 g of the SAP in a 600 ml of hot distilled water at 80 °C. The dispersion was stirred until homogenous paste of about 6.2% total solid was formed. The beaker containing the paste was dipped in a cold water bath set at about 4 °C and manually stirred medium to enhance its cooling rate to target temperatures 20, 30, 45 and 60 ± 2 °C. The viscometer spindle (No. 2) was immediately dipped into the paste sample which was stirred at shear rates of 10, 30, 50, 60 and 100 rpm. The apparent viscosity of the paste samples were then read from the viscometer when the reading became constant.

The rheological data were then modeled using Power law equation of the form:

\[
\eta = K T^{n-1}
\]

\( \eta \) is the apparent viscosity (Pa), \( K \) is the consistency index (in Pa·s\(^n\)), while \( n \) is the flow behavior index.

The temperature effect on the consistency index \( (K) \) was modeled with the Arrhenius type equation:

\[
K = K_o \exp \left( \frac{E_o}{RT} \right)
\]

\( K_o \) is a constant (in Pa·s\(^n\)), \( E_o \) is the flow activation energy (in J/mol), \( R \) is the universal gas constant (in J/mol/K), \( T \) is the absolute temperature (in K).

3. Results and discussion

3.1. Water vapor adsorption properties of SAP

The typical sorption behavior of food material is depicted by its isotherm curve generated experimentally. The shape of the isotherm curve indicates the changing water vapor adsorption capacities of the product under different relative humidity of the surrounding air. In addition, other information that could indicate its shelf life (or shelf stability) and thermodynamic changes accompany the water vapor sorption process in the product may be derived from the curves. Based on shape, five types of isotherm curves have been identified for different materials according to Brunauer et al. (1940). The moisture adsorption isotherms of SAP at various temperatures are shown in Fig. 1. The EMC decreased with sorption temperature. The shape of the isotherms indicates rapid increment in EMC with increased \( a_w \) implying that SAP is highly hygroscopic. From the shapes, it is evident that SAP exhibits type II. Similar curves were reported for several raw and processed...
starch containing food materials such as potatoes (McMinn and Magee, 1999), rice flour (Durakova and Menkov, 2004; Brett et al., 2009), and Jasmine rice crackers (Stripatrawan and Jantawat, 2006). Thus, SAP requires careful handling and proper packaging environment to make it stable over long period of storage. The EMC also decreased with increased temperature. The water vapor adsorptive capacity of SAP at the same temperature (35°C) and \(a_w\) (0.90) is about 5 – 6 times of that reported for capuassu chocolate powders, and about 1.5 – 2 times that reported for capuassu powder (Medeiros et al., 2006). The sorptive capacity by of SAP is similar with that reported Tencil (1999) for skim milk powder at the same conditions. Due to reduced binding energy between water molecules at the sorptive sites of SAP, the EMC reduced with increased temperature. This trend is commonly reported for many food items. However, in oat and rice flours an unusual trend was observed (Brett et al., 2009). There was reduction in EMC with increased temperature at \(a_w\) below 0.75. Above this value, the EMC increased with increased temperature. The inverted behavior observed was attributed to a greater exposure of active sites or hydrophilic groups at high \(a_w\) or water contents as the temperature increased.

Survey of literature has shown that the GAB and Peleg models give very reliable approximation of the full range water vapor sorption data for many raw and processed food materials. Table 1 shows coefficients of Peleg, DLP and BET (Brunauer et al., 1938) models used to fit the sorption data and their predictive capacity as reflected by the coefficients of determination (\(R^2\) values). In this study, DLP model gave better fit to the SAP sorption data (\(r^2\) ranged between 98.3% and 99.9%) than the other models as similarly reported for Taro flour (Nurtama and Lin, 2010). For an acceptable model the value of \(E\%\) should be less than 10% as indicator of good fit for a model (Boquet et al., 1979). The average \(E\%\) value for DLP (5.50) is lower than that of Peleg (8.23) and GAB (11.74) (Table 1). All the models however gave some random distribution of the moisture residuals (Fig. 2) indicating the errors of prediction accruing to each model is natural and not systematic. Therefore it can be inferred that Peleg model gave better fit to the adsorption data. The main distinction between them is that the coefficients of GAB models have thermodynamic meanings while those of Peleg model are rather empirical i.e. they do not have established physical meanings.

The value of the monolayer moisture content (\(M_o\)) is of particular interest since it indicates the amount of water that is strongly adsorbed to specific sites at substrate surface and is considered as the optimum value to assure stability of substrate material. Therefore, \(M_o\) is recognized as the moisture content

<table>
<thead>
<tr>
<th>Model</th>
<th>Parameter</th>
<th>Temperature (°C)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>27</td>
</tr>
<tr>
<td>GAB</td>
<td>(M_o)</td>
<td>0.049</td>
</tr>
<tr>
<td></td>
<td>(C)</td>
<td>(-5.345 \times 10^4)</td>
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<tr>
<td></td>
<td>(K)</td>
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<td></td>
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<tr>
<td></td>
<td>(k_2)</td>
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<td></td>
<td>(n_2)</td>
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<td>(E%)</td>
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<tr>
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<td>RMSE</td>
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<tr>
<td></td>
<td>(r^2)</td>
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<td>DLP</td>
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<td></td>
<td>(b_2)</td>
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</tr>
<tr>
<td></td>
<td>(b_3)</td>
<td>(-0.004)</td>
</tr>
<tr>
<td></td>
<td>(E%)</td>
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<td></td>
<td>(r^2)</td>
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<tr>
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<tr>
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<td>(r^2)</td>
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affording the longest time period with minimum quality loss at a given temperature. Below it, rate of deteriorative changes is kept at minimum. The monolayer moisture contents \((M_o)\) of SAP estimated from the GAB model ranged between 4.90% and 6.80% (dry basis) (Table 1). This corresponds to \(a_w\) value that ranged from 0.22 to 0.48. These values are similar with those reported for some fermented cassava powdered products (Sanni et al., 1997), some legume fortified cassava products (Sanni and Kuye, 2000), mango kernel flour (Arogba, 2001) but lower than that of pineapple pulp powder (Gabas et al., 2007). As observed in previous studies, the values obtained from BET were lower than those of GAB model. The monolayer values obtained in this work were not significantly affected by temperature. Some other studies have however reported decreased monolayer values with temperature due to the reduction in the number of total active sites for water binding in the respective materials. An unusual reduction in the monolayer value of oolong tea as the temperature increased from 5 to 15 °C was reported by Chen and Weng (2010). The monolayer values then increased with temperature above 15 °C.

The GAB parameter \(C\) is a measure of the binding strength of water molecule to primary binding site. The larger the value of \(C\) the larger is the enthalpic difference between the monolayer and multilayer molecules. The values of \(C\) reduced with increased temperature. The values of \(C\) could be fairly modeled by the Arrhenius type of equation:

\[
C = C_0 \exp \left(\frac{\Delta H_c}{\mathcal{R}T}\right)
\]

\(\mathcal{R}\) is the gas constant for water vapor (J/mol). This gave \(\Delta H_c\) value of 4.1 kJ/mol and \(C_0\) value of \(1.24 \times 10^{-12}\) kJ/mol. The value \(\Delta H_c\) gives the enthalpic difference between the monolayer and multilayer water molecule in the material. This value is much less than that obtained for corn.

Parameter \(k\) is defined as the ratio of the partition function of molecule in the bulk liquid phase and the partition function of molecules sorbed in the multilayer. It measures the entropic contribution of the multilayer water molecules to the whole system. The values of \(k\) for SAP appeared to be fairly constant with respect to the sorption temperature as similarly observed for texturized soy protein (Cassini et al., 2006) and lean meat (Trujillo et al., 2003). The value of \(k\) for protein is generally expected to be between 0.78 and 0.85 (≈ 0.8) while that of starch is between 0.65 and 0.75 (≈ 0.7). Considering the mean values of \(k\) obtained here (≈ 0.78), it could be confirmed that SAP has a sorptive energy constant value that resembles that of protein-rich solid matrix probably due to its higher protein content (≈ 81.3% dry basis).

Fig. 3 shows the plot natural log of \(a_w\) versus \(1/T\) at constant moisture content to calculate the values of the differential enthalpy and entropy. The slope which is a measure of differential heat of sorption gradually reduced to zero as the moisture content of product increased. The critical moisture content at which sorptive heat becomes is approximately 16%. This implies the water molecules removed from the surface of SAP at moisture level above this value will essential behave like free water during dehydration. The variation of \(q_{st}\) with

\[
q_{st} = 99.26 + 16.26M + 0.95M^2 + 0.02M^3 \quad (r^2 = 0.999)
\]

The enthalpy–entropy plot shown in Fig. 5 indicates that perfect linear relationships exists between the two thermodynamic parameters \((r^2 = 1.0)\). The isokinetic \((T_\beta)\) and the harmonic mean \((T_{hm})\) temperature values obtained for SAP are 330.7 and 306.9 K, respectively. This \(T_\beta\) value is lower than those reported for quinoa grains (361 K) (Tolaba et al., 2004), oatmeal biscuit and oat flakes (430.9 and 443.4 K, respectively) (McMinn et al., 2007) but within the range reported for
garlic (348 K) (Madamba et al., 1996) and sucuk (323.4 K) (Polatoğlu et al., 2011). The free energy constant ($\alpha$) for SAP is 318.8 J/mol, which firstly indicates the water vapor sorption process by SAP is a non-spontaneous process. Since $T \beta \not= T_{hm}$, then the enthalpy–entropy compensation theory holds for the water vapor adsorption process in SAP. In addition, the process is enthalpy-driven due to the fact that $T \beta > T_{hm}$.

3.2. Rheological behavior of SAP paste

Previous authors (Cardinaels et al., 2013; Németh et al., 2010) have shown that albumen at temperature below 60 °C has a shear thinning flow behavior with flow behavior index ($n$) value > 0.6. Starch paste regardless of concentration and biological origin generally has pseudoplastic behavior with flow index ($n$) values < 0.6 (Sopade and Kiaka, 2001; Adebowale and Sanni, 2013). SAP paste showed no sign of time-dependent flow behavior (Fig. 6 shows the typical flow behavior of SAP. The reduced viscosity with increased shear rate indicates that the SAP paste (at 6.2% solid content) is a shear thinning fluid. The rheological data fitted very well into the power law model at all temperatures ($r^2=0.96–1.00$). The consistency index reduced with increased temperature (Table 2). The flow index ($n$) values of SAP ranged between 0.50 and 0.68 implying that the pseudoplastic behavior of both starch and albumen was an intermediate of the two natural hydrocolloids composed in SAP. The reduction of K and increase in $n$ value with increased product temperature agrees with the observation of Rafe and Masood (2014). The consistency index ($k$) of the paste decreased with increased temperature. The variation of $k$ with temperature was found to obey Arrhenius law ($r^2=0.954$) with activation energy ($E_a$) value of 31.632 kJ/mol.

4. Conclusions

The study revealed that SAP is a highly hygroscopic material that exhibit Type II isotherm shape. Its sorption behavior is close to other protein rich food powder. The monolayer moisture content ranged between 4.9 and 6.8g/100 g solid. The adsorption data were better fitted by DLP and Peleg models. Thermodynamic analyses indicate that water adsorption process of SAP is non-spontaneous and obeys the compensation theory. The 6.2% aqueous paste of SAP
displayed a pseudoplastic behavior that was not affected by temperature changes.

References


