



MOISTURE SORPTION ISOTHERM CHARACTERISTICS OF AWARA FOOD

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ABSTRACT

Moisture sorption and storage stability of Awara were evaluated using gravimetric method. Guggenheim-Anderson-de boer (GAB) and Brunauer Emmett and Teller (BET) were used in evaluating sorption and desorption isotherm at temperature range of 30, 40 and 50°C in the range of water activity (0.1-0.9). Sigmoid moisture isotherm shapes were obtained at various temperatures. Lowest level EMC was obtained at highest temperature (50°C). A non-linear regression analysis was used to fit both BET and GAB. GAB was found to generate higher coefficient of determination R^2 (0.9986, 0.9959 and 0.9933) and lowest mean of relative percentage p% (0.231, 0.0061 and 0.0017) at temperature range (30, 40 and 50°C) respectively. Safe % water and storage stability were all predicted from adsorption isotherm curve. 3.14, 3.19 and 3.10 were found to be the safe % water which corresponds to the 3.8aw for GAB adsorption graphs at 30, 40 and 50°C temperatures respectively. This safe % water will be useful in packaging and determination of shelf- life of Awara.

Keywords: Awara, sorption isotherm, BET and GAB models, water activity

INTRODUCTION

Awara food is commonly consumed in a daily basis in northern part of Nigeria. It is processed soybean (*Glycine max* species) available in fresh, sun dry and in fried form. It is a popular food called tofu in Asian countries like China and India, Awara (tofu) was invented in China (Fen and Jin, 2013). It is a curd made by coagulating soy protein by mineral salts or acid. In Awara making, a coagulant (calcium, magnesium salt, or glucono delta-lactone) is used to precipitate the soy protein from soymilk to form a jelly-like curd. Depending on the downstream process, this curd can be converted into different types of products. The jelly-like curd can be further pressed to make Awara with various hardness and moisture levels, flavours can be added to create different form and varieties (Fen and Jin, 2013).

Historically, tofu is a major source of protein in the Chinese diet in which meat supply was not in abundance until recent year. Calcium-coagulated tofu also serves as an important source of calcium, an essential macro mineral, which is deficient in typical Chinese diet (Fen and Jin, 2013).

Several mathematical models have been developed to describe sorption isotherm of foods (Alkali, J.S and satimehin, 2009; Palou et al, 1997). The models were tested and the constant evaluated for sorption data of many agricultural crops (Ajibola, et al, 2004). More than 23 commonly use models equation for fitting the sorption of different food materials have been review and discussed (Ajibola, et al, 2004). In more recent years, among the sorption models, the Guggenheim-Anderson-deBoer (GAB)

equation has been applied most successfully to various foods (Timmermann et al., 2001). Brunauer-Emmett-Teller (BET) sorption isotherm was the model with the greatest application to water sorption isotherms of foods and foodstuffs (Labuza, 1968; Iglesias and Chirife, 1976), although it was known to hold only for a limited range of water activity. Despite the theoretical limitations of the BET adsorption analysis, the BET monolayer concept was found to be a reasonable guide with respect to various aspects of interest in dried foods (Karel, 1973; Iglesias and Chirife, 1981). The monolayer moisture content (M_0) is the moisture content at which the rate of quality loss is negligible (Labuza, 1984).

The study was aimed to investigate the water material interaction phenomenon (desorption and adsorption moisture content) of Awara food and determine the best moisture sorption (MIS) model that would predict desorption and adsorption isotherm. This would help in determining the shelf- life of the Awara food, and also in designing and selecting materials for packaging.

MATERIALS AND METHOD

Moisture sorption isotherm determination was carried out in Lab one Food process Engineering Department Sam Higginbottom University of Agriculture Science and Technology Allahabad, India at three different temperatures in (30-50°C) over the range of water activity 0.1- 0.9 using the static gravimetric method.

For the moisture adsorption studies, the moisture contents of the 5.0g of samples were reduced to very low levels by oven drying step wisely from 40°C to 105°C. The samples were allowed to cool in separate desiccators, and thereafter, packaged in polyethylene bags and store in desiccators, from where they were removed and used for experiments.

For the desorption studies, the samples were rewetted to very high moisture levels (40 and 60% db) and then packaged in sealed polyethylene bag and stored in a refrigerator for 24 hours to attained uniform moisture distribution. Prior to investigation, the required sample were removed from the refrigerator and allowed to equilibrate with the ambient condition for 2 hours.

Material Procurement and Preparation

Bulk sample of fresh sample of Awarasealed in polyethylene bag and stored at ambient temperature, in order to take care of moisture migration within the material. Glass bottles oven and salt solution were used.

Experimental Procedure

Seven sealed glass bottle were used in this research (Gazor, 2010). Each bottle was provided with the sample holder. The holder kept the sample above the saturated salt solution in order to avoid contact with the solution. In each bottle three replication were used with 5.0g each sample. At each temperature (i.e. 30, 40 and 50°C) fourteen glass bottles were used for both adsorption and desorption. All the bottles were put in the oven and set at the required temperature. The weight loss of each sample in the bottle was measured daily and the salt solution was maintained at the saturated state. The experiment was completed when three consecutive readings were obtained.

The equilibrium moisture content of the samples were determined after each sorption experiment using a precision air-oven method at a temperature of 135°C for 2 hours until constant weight was achieved, according to the standard method of AOAC (2000). The mean values of three replications were obtained for each experiment. To establish the moisture sorption isotherms, the equilibrium moisture contents were plotted against water activity at constant temperature.

Fitting isotherm models to the experimental data

Base on the nonlinear analysis result obtained as given in table 6. GAP and BET parameters were calculated. The coefficient of determination (R²) and mean relative percentage deviation modulus (P %) were computed for desorption and adsorption at various temperatures (30, 40 and 50°C). The models were compared on the basis of R² and P (%). GAP was found to have R² closed to one for both temperatures and with least P (%) approximately zero. This is in accordance with rule of P (%) < 10%, indicated good fitness (Bernnan, 1991; Iomauro et al, 1985).

Adsorption and Desorption Equilibrium Moisture Contents (EMC) Determination

The adsorption and desorption equilibrium moisture contents (EMC) were determined for the samples using saturated salt solution method at 30, 40, 50°C over the range of relative humidity from 0.1 to 0.90. BET and GAB models were used in

predicting the best fit model. The monolayer moisture content (mm) values at different temperatures were calculated using BET and GAB equations.

Moisture Adsorption Isotherm Determination

To determine moisture adsorption isotherm, 5.0g of the samples at very low dry basis moisture content in triplicate were weighed into sample cans and placed inside glass bottle to maintain different constant relative humidity in the glass bottle (Gazor, 2010). Excess salt was maintained in each solution to ensure saturation. The bottle containing the salt solutions and the samples were marked and placed inside temperature controlled oven set at a specified temperature. The oven temperature was monitored.

The samples were weighed daily using an electronic balance with an accuracy of 0.001 g. Equilibrium was considered to have been attained when three consecutive readings were obtained. Three replications were used for each experiment. The dry matter content of sample was determined by oven drying step wisely from 40°C to 120°C until constant weight was obtained. The EMC was calculated in dry basis from weight change and dry matter weight. The average EMC obtained at each relative humidity (water activity when expressed in decimal) at a given temperature were recorded.

Moisture Desorption Isotherm Determination

The procedures followed in determining the adsorption EMC were repeated so as to obtain desorption EMC. The average EMC obtained at each relative humidity (water activity when expressed in decimal) at a given temperature were recorded.

Isotherm Plots

The data obtained on the adsorption and desorption EMC of the sample were used to carry out the following plots:

Moisture sorption isotherms of the sample in adsorption at different temperatures over the water activity range employed.

Moisture sorption isotherms of the samples in desorption at different temperatures over the water activity range employed.

The adsorption and desorption isotherms of the sample constructed at each temperature to investigate the occurrence of moisture sorption hysteresis.

Moisture Sorption Isotherm modelling equation

Several different isotherm models have been proposed and compared in the literature. These models were chosen because they are most widely used to fit experimental sorption data of various food materials and are used to evaluate thermodynamic functions of water in foods. The parameter of the sorption models were estimated from the experimental result using the nonlinear regression analysis (SPSS 9.0 for window 1998) which minimise the residual some of squares. Best fitting equations were evaluated with the mean relative percentage deviation (P) value. Defined as

$$P(\%) = \frac{100}{N} \sum_{i=1}^N \frac{|M_{ei} - M_{ci}|}{M_{ei}} \quad (1)$$

Where M_{ei} and M_{ci} are experimental and predicted moisture content values respectively, and N is the number of the experimental data. A models is considered acceptable if the P

value is below 10% (Boquet, Chirife and Iglesias, 1978; Lomauro et al., 1985).

The experimental equilibrium moisture content (EMC) data were processed using Microsoft Excel (2007) software and analysed using the linear trend method on transformed linearised equations for the BET parameters; whereas the polynomial regression method was used on the transformed binomial equation for the GAB parameters.

Brunauer Emmett-Teller (BET) Equation

The Brunauer Emmett-Teller (BET) Equation is

$$\frac{m}{m_o} = \frac{ca_w}{(1-a_w)(1-a_w+ca_w)} \quad (2)$$

Where m is the moisture content (kg/kg dry solid), m_o is the monolayer moisture content (kg/kg dry solid), a_w is water activity and c is a constant related to the net heat of sorption.

The two-parameter BET model can be expressed as follows (Brunauer, Emmett and Teller, 1938):

$$m = \frac{m_o ca_w}{(1-a_w)[1+(c-1)a_w]} \quad (3)$$

where: m = equilibrium moisture content, %, d.b., m_o = BET monolayer moisture content, %, d.b., a_w = water activity, decimal c = BET constant related to the net heat of sorption

To obtain the two characteristic constants, m_o and c , the BET equation (1) was linearised as follows (Timmermann, 2003):

$$\frac{a_w}{(1-a_w)m} = \frac{1}{cm_o} + \frac{c-1}{cm_o} a_w \quad (4)$$

Which can easily be transformed to a linear equation with the following form:

$$\frac{a_w}{(1-a_w)m} = Aa_w + b \quad (5)$$

where

$$A = \frac{c-1}{cm_o} \quad (6)$$

$$b = \frac{1}{cm_o} \quad (7)$$

The BET parameters were obtained by plotting $a_w / (1-a_w) M$ versus a_w which would usually give a linear part at low activities up to 0.55 a_w . From the slope (A) and the intercept (b), the BET parameters were determined using the following relationships:

$$m_o = \frac{1}{(A+b)} \quad (8)$$

$$c = \frac{A+b}{b} \quad (9)$$

Guggenheim-Andersiosn-de Boer (GAB) Equation

Guggenheim-Andersiosn-de Boer (GAB) Equation

$$m = \frac{m_o cka_w}{(1-ka_w)(1-ka_w+cka_w)} \quad (10)$$

where m = equilibrium moisture content, %, db., m_o = GAB monolayer moisture content, c = constant related to the

monolayer heat of sorption, k = factor related to the heat of sorption of the multilayer (Rizvi, 1986)

This equation has a similar form to BET, but has an extra constant k . BET is actually a special case of GAB, where $k=1$. The GAB parameters were determined following the method of the transformed form of the GAB isotherm (Timmermann et al., 2001).

The GAB model can be rearranged to a polynomial equation as follows:

$$\frac{a_w}{m} = \frac{k}{m_o(1/c)} a_w^2 + \frac{c-2}{m_o c} a_w + \frac{1}{m_o c k} \quad (11)$$

$$\alpha = \frac{k}{m_o(1/c)} \quad (12)$$

$$\beta = \frac{c-2}{m_o c} \quad (13)$$

$$\varepsilon = \frac{1}{m_o c k} \quad (14)$$

$$\frac{a_w}{m} = \alpha a_w^2 + \beta a_w + \varepsilon \quad (15)$$

A polynomial direct nonlinear regression method of a_w/M versus a_w was carried out using Microsoft Excel (2007) software in order to determine the values of the coefficient of the quadratic term α , the linear term coefficient β and the constant ε . Then the GAB parameters were calculated as follows:

$$k = \frac{f^{0.5}-\beta}{2\varepsilon} \quad (16)$$

$$m_o = \frac{1}{\beta+2k\varepsilon} \quad (17)$$

$$c = 2 + \frac{\beta}{k\varepsilon} \quad (18)$$

where

$$f = \beta^2 - 4\alpha\varepsilon \quad (19)$$

The temperature dependency of the GAB parameters was given by the following Arrhenius-type equations:

$$c(T) = c_o \exp\left(\frac{\Delta H_c}{RT_{abs}}\right) \quad (20)$$

$$k = k_o \exp\left(\frac{\Delta H_k}{RT_{abs}}\right) \quad (21)$$

$$m_o = m_o \exp\left(\frac{-E_a}{RT_{abs}}\right) \quad (22)$$

where c_o , k_o and m_o are adjustable constants for the temperature effect; ΔH_c = difference in enthalpy between monolayer and multilayer sorption (kJ/kmol), ΔH_k = difference between the heats of condensation of water and the heat of sorption of the multilayer (kJ/kmol), R = universal gas constant, 8.314J/mol K T_{abs} = absolute temperature, K

E_a = activation energy, KJ/mol

For the monolayer moisture content, an Arrhenius-type equation was calculated using a linearised form of Equation (22):

$$L_n(m_o) = L_n(m_o) - \frac{E_a}{RT_{abs}} \quad (23)$$

The coefficient of Equation (23) was easily obtained by plotting on an $L_n(m_o)$ versus $1/T_{abs}$ diagram. The E_a and m_o coefficient was subsequently related to the temperature by applying the regression technique.

E_a was calculated by plotting $L_n(m_o)$ versus the reciprocal of the absolute temperature. The slope of the curves found by applying the linear regression yields the coefficient E_a/R , while the intercept is equal to $\ln(m_o)$. The other GAB parameters' coefficients were determined using the same procedure.

RESULTS AND DISCUSSION

Moisture sorption isotherms

Various data were obtained for desorption and adsorption EMC for *Awaraat* temperature levels of 30, 40 and 50°C over the range of water activity 0.1-0.9 as given in fig 2, 3 and 4 respectively. The EMC for each desorption and adsorption represent the mean of three replication at a given a_w . The EMC increases with increase in a_w .

The isotherms plot showed sigmoid shape when EMC was plotted against a_w , this type of behaviour is commonly obtained for most food items more especially low sugar product (Labuza 1984). Although BET is more popular models that give the base fit model for food with high water content. (Labuza, 1968; Iglesias and Chirife, 1976), the reverse is the case in *Awara* food this may be due to high protein content. Hysteresis was observed between 0.3 to 0.8 water activities for all the temperatures.

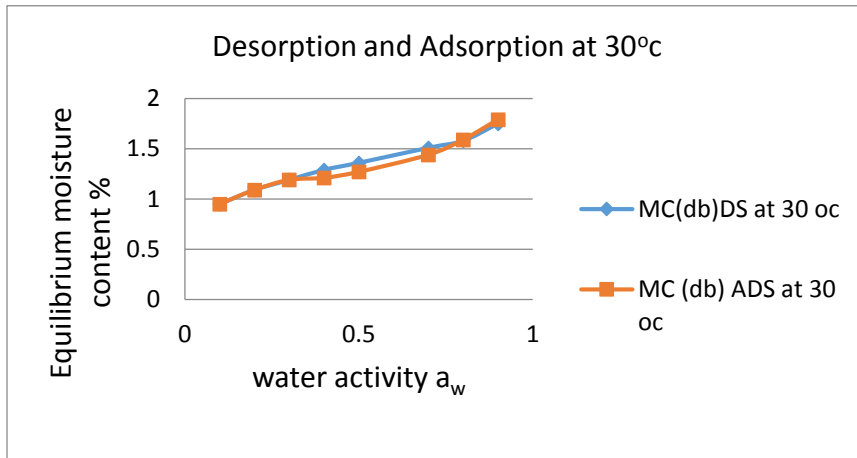


Fig 1: desorption and adsorption isotherm plot at 30°C

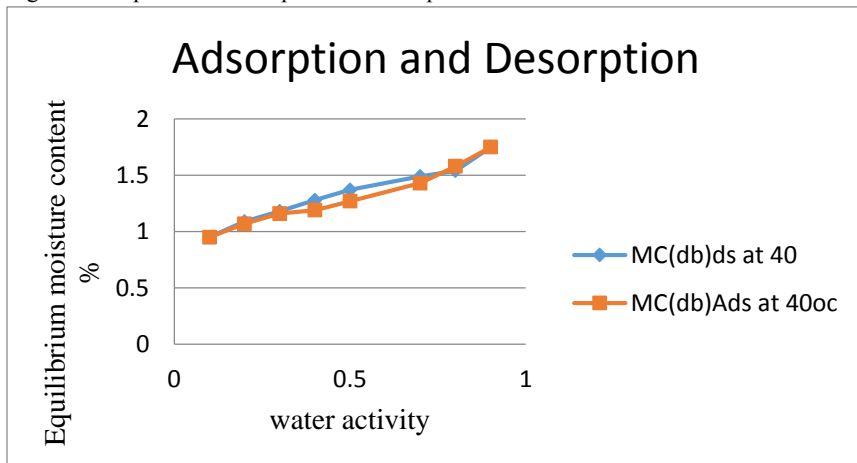


Fig 2: desorption and adsorption isotherm plot at 40°C

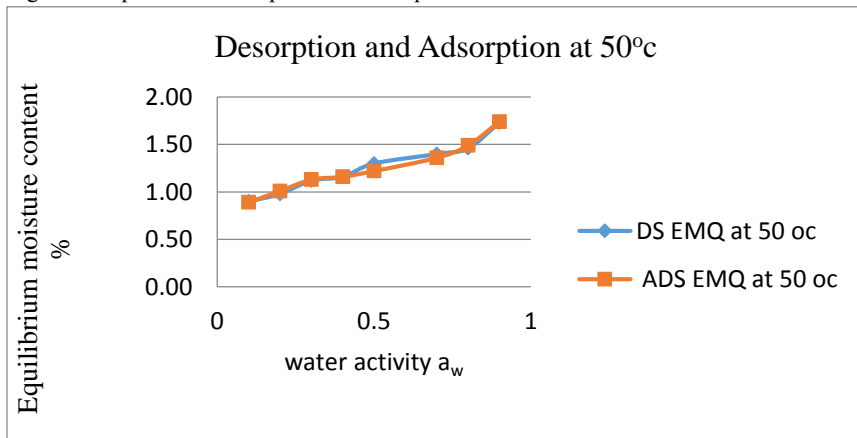


Fig 3: desorption and adsorption isotherm plot at 50°C

Desorption and adsorption

Desorption and adsorption temperature dependency was shown in table 6. The equilibrium moisture content increases with decrease in temperature as indicated in fig 4 and 5. The results were in agreement with this hypothesis as it was widely accepted by many researchers (Figen and Atil, 2003). Both desorption and

adsorption isotherms sigmoid curve increase with decrease in temperature. The curves for temperature 30 and 40°C were observed to almost over lap this could be due to the nature of the product of high protein content. An attempt to carry out the experiment above 50°C gave undesirable result this indicated that it might be practically impossible.

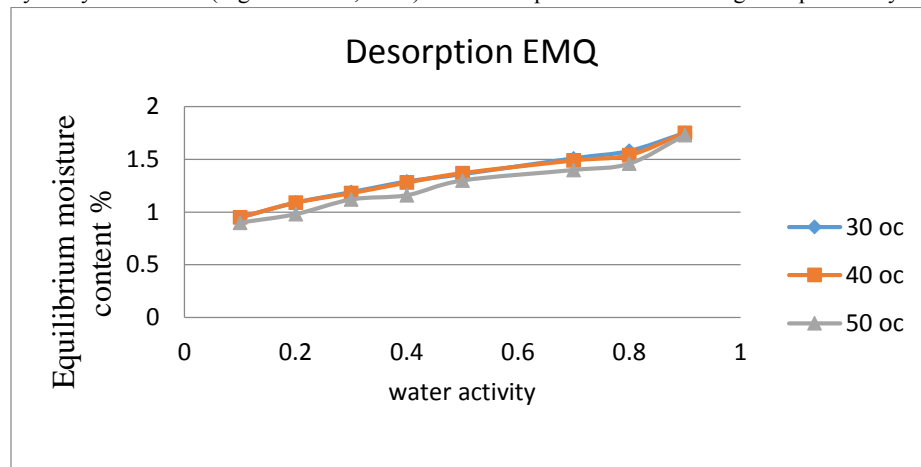


Fig 4: Desorption isotherm plot at 50, 40 and 30°C

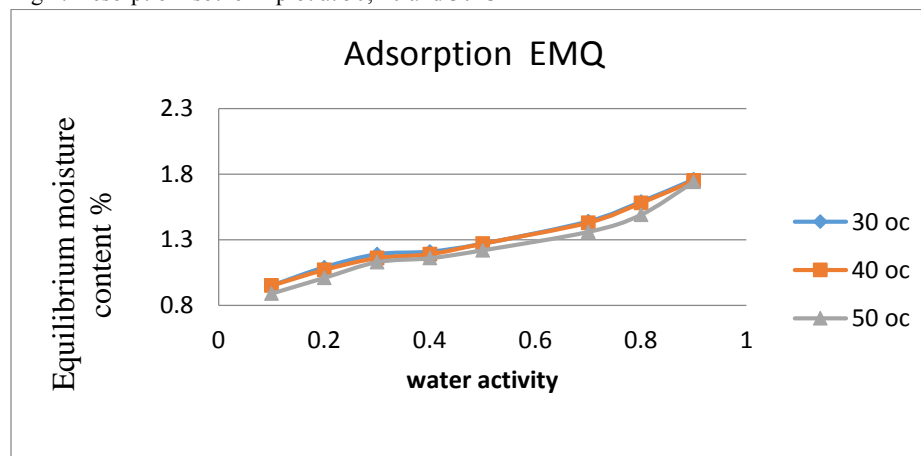


Fig 5: Adsorption isotherm plot at 50, 40 and 30°C

Table 6: Non-linear regression analysis

Model		Desorption			Adsorption		
		30 °C	40 °C	50 °C	30 °C	40 °C	50 °C
BET	C	-3.4322	-3.6371	-3.6949	-3.7311	-3.7168	-3.7561
	M	0.6922	0.2461	0.2357	0.2499	0.2459	0.2390
	R ²	0.7539	0.7603	0.7678	0.7714	0.7653	0.7799
GAB	P (%)	6.8210	7.6469	8.6859	9.3454	9.0671	9.9119
	C	0.8037	124.76	137.38	-431.09	-1753.67	-802.94
	M	1.1154	1.1038	0.9952	0.9452	0.9484	0.9000
	R ²	0.9986	0.9959	0.9933	0.9966	0.9983	0.9937
	P (%)	0.23	0.0061	0.0017	0.03877	0.0241	0.0320
	K	0.4025	0.4012	0.4433	0.5113	0.5010	0.5125

A non-linear regression analysis was used to fit both BET and GAB. GAB was found to generate higher coefficient of determination R² (0.9986, 0.9959 and 0.9933) and lowest mean

of relative percentage p% (0.231, 0.0061 and 0.0017) at temperature range (30, 40 and 50°C) respectively. Safe % water and storage stability were all predicted from adsorption isotherm

curve. 3.14, 3.19 and 3.10 were found to be the safe % water which corresponds to the $3.8a_w$ for GAB adsorption graphs at 30, 40 and 50°C temperatures respectively. This safe % water will be useful in packaging and determination of shelf- life of Awara.

CONCLUSION

Desorption and adsorption at all the temperatures were found to be sigmoid curve in shape as it is true with many food products and hysteresis behaviour was also clearly observed for all temperatures. GAP model was found to be the best fit model that could be used to predict the behaviour of the product (*Awara*).

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