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# **RESEARCH ARTICLE**

### **ADSORPTION ISOTHERMS AND THERMODYNAMIC PROPERTIES OF DRIED PUMPKIN FRUIT SLICES**

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### **INTRODUCTION**

According to Chen (2013) the equilibrium moisture content of the food material  $(M_e)$  corresponding to a given water activity and temperature is determined experimentally and is usually presented as the water or moisture sorption isotherm of the material. Studies by Janjai*et al.* (2011), Edoun*et al* (2010) revealed that sorption isotherm is useful to determine the shelf life and to assess the background of operations such as drying, conditioning, mixing, packaging, and storage.

*Brunauer-Emmett-Teller (BET) Equation:* The BET equation, which is the most widely used model in food systems, was first proposed by Brunauer, Emmett, and Teller (1938). It `holds at water activities from 0.05 to 0.45. It is an effective method for estimating the amount of bound water to specific polar sites in dehydrated food systems. BET equation is generally considered to be limited in its range of water activities and is expressed in the form:

$$
\frac{a_w}{(1-a_w)M_e} = \frac{1}{M_o C} + \left[\frac{C-1}{M_o C}\right] a_w
$$
\n(1)

The above equation is often abbreviated as:

$$
A=bx+c \tag{2}
$$

Where the constants are defined as  $b = (C - 1) / (M_0 C)$  and  $c =$  $1/ (M_0 C)$  are obtained from the slope and intercept of the straight line generated by plotting  $a_w / (1 - a_w)$  *M* against  $a_w$ . Studies by Staudt *et al.* (2013) revealed that the value of monolayer can be obtained from  $M_0 = 1/(b + c)$  and  $C = (b + c)$  $c$ / $c$ . Although not well defined, the monolayer  $(M<sub>o</sub>)$  is often stated to represent the moisture content at which water attached to each polar and ionic group starts to behave as a liquid-like phase and corresponds with the optimal moisture content for stability of low-moisture foods. According to Nurmata and Lim (2010), the monolayer moisture content of many foods has been reported by researchers to correspond with the physical and chemical stability of dehydrated foods. The BET equation has been found useful in defining optimum moisture content for drying and storage stability of foods and in the estimation of surface area of a food. Studies by Robitzer *et al.* (2011) revealed that surface area of sorption of biological materials is usually high especially those with intrinsic microporous structures. The surface area of sorption of the food is estimated using the apparent surface area which is expressed as:

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$$
S_O = \left[\frac{1}{M_s}\right] N_o \, AM_o \tag{3}
$$

Where,  $S_0$  = Apparent sorption area of the sorbate (m<sup>2</sup>/g solid),  $M_s$  = Relative molecular mass of water (18 g),  $N_o$  = Avogadros number (6.023 x  $10^{23}$  molecules/mole), A = Apparent surface area of one water molecule  $(1.05 \times 10^{-19} \text{ m}^2)$ 

*The Guggenheim–Anderson-de Boer (GAB) Equation:* The three-parameter GAB equation, derived independently by Guggenheim (1966), Anderson (1946), and de Boer (1953), has been suggested to be the most versatile sorption model available in the literature Somrmoli and Langrish (2015), Aktaset al.(2014). Fundamentally, it represents a refined extension of the Langmuir and BET theories, with three parameters having physical meanings. For sorption of water vapours, it is mathematically expressed as:

$$
M_w = \frac{M_o C K a_w}{(1 - K a_w)} (1 - K a_w + C K a_w)
$$
\n(4)

Where,  $M_0$  is monolayer moisture content, and  $C$  and  $K$  are the adsorption constants which are related to the energies of interaction between the first and further sorbed molecules at the individual sorption sites.

The GAB equation (4) can be rearranged as follows:

$$
\frac{a_{w}}{M} = \left(\frac{K}{M}\right)\left[\frac{1}{C-1}\right]a_{w}^{2} + \left(\frac{1}{M}\right)\left[\frac{2}{C}\right]a_{w}
$$
\n(5)

If  $\alpha = (k/M_o)$  [(1/*C*) – 1],  $\beta = (1/M_o)$  [1 – (2/*C*] and Y=  $(1/M<sub>o</sub>Ck)$  the equation transforms to a second order polynomial form:

$$
\frac{a_w}{M} = \alpha a_w^2 + \beta a_w + \gamma
$$
\n(6)

The numerical values of the equation parameters are then obtained as follows:

$$
K = \frac{\sqrt{\beta^2 - 4\alpha}}{2\gamma} \tag{7}
$$

$$
C = \frac{\beta}{\gamma} \tag{8}
$$

$$
M_o = \frac{1}{\gamma K C}
$$
 (9)

The constants are obtained by conducting a linear regression analysis on experimental values.

*Isosteric heat of sorption:* Important thermodynamic parameters derived from moisture sorption isotherms are the net isosteric heat of sorption, the enthalpy and entropy of sorption. The net isosteric heat of sorption provides useful information on the heat and free energy changes during moisture sorption processes in foods (Edoun and Kuitche 2014, Koua*et al*., 2014). Studies by Aktas *et al*. (2014), revealed that the heat of sorption is a good estimate of the minimum heat required to remove a given amount of water. The net isosteric heat of sorption gives an understanding about the food micro-structure and the physical changes occurring on the surfaces of food. Studies by Hassini*et al.* (2015) that worked on adsorption isotherms and thermodynamic properties of prickly pear seeds, Gonoli*et al*. (2010) who worked on water adsorption and thermodynamic properties of okra seeds revealed a widely used method for the calculation of heat of sorption of foods which is based on the integral solution to Clausius-Clapeyon equation given as:

$$
ln z_w = C_{st} - \left(\frac{\Delta H_{st}}{R}\right) \left(\frac{1}{T}\right)
$$
 (10)

Where,  $\Delta H_{st}$  = net isosteric heat of sorption (H<sub>st</sub> - H<sub>L</sub>), H<sub>st</sub> = isosteric heat of sorption,  $H_{\text{L}=}$  latent heat of vapourization of pure water and  $C_{st}$  = a constant related to entropy of sorption. The major water binding sites in food are polymers such as proteins, starch, sugars, cellulose and hemicelluloses. The polar groups of these polymers provide the binding sites for water. Tsami*et al,* (1990a), used an equation that describes the relationship between  $\Delta H_{\text{st}}$  and equilibrium moisture content in their work on sorption isotherms of raisins, currants, figs, prunes and apricots as:

$$
\Delta H_{st} = \Delta H_o \exp\left(\frac{M}{M_c}\right)
$$
\n(11)

Or

$$
ln\Delta H_{st} = ln\Delta H + \left(\frac{M}{M}\right)
$$
\n(12)

Where,  $M =$  equilibrium moisture content  $\Delta H_0 =$  isosteric heat of sorption of first molecule of water,  $M_c$  = characteristic moisture content of the food material. Plotting ΔHst versus M enables the calculation of  $M_c$  from the slope and  $\Delta H_0$  from intercept. Studies by Rizvi, (2005) revealed that, when a polymer sorbs water, various changes occur such as constitution and dimension and the enthalpy that is developed as a result of these changes is a measure of the energy variations occurring when water molecules combine with sorbent during sorption processes.

*Entropy of sorption:* The relation between net isosteric heats of sorption otherwise known as differential enthalpy  $( \Delta H_{st})$ and differential entropy  $(\Delta S^{\circ})$  of sorption is described by Ariahu *et al*., (2006) as:

$$
lnq_{v} = -\Delta H_{s}/(R\hat{T} + (\Delta S/R) \tag{13}
$$

Where;  $a_w$  = water activity, R = universal gas constant (0.008314 KJ/mol°K) and T is absolute temperature (K). A plot of  $ln_1 a_w$  versus 1/T gives a slope and intercept from which values of  $\Delta H_{st}$  and  $\Delta S^{\circ}$  are computed respectively. When this is applied at different moisture contents, it gives room for determination of the dependence of  $\Delta H_{st}$  and  $\Delta S^{\circ}$  on moisture content. Studies by Bajpai and Pradeep (2013), described the enthalpy-entropy compensation theory using a linear relationship between  $\Delta H_{st}$  and  $\Delta S^{\circ}$  as:

$$
\Delta H_{st} = T_c \Delta S + \Delta G \tag{14}
$$

Where,  $T_c$  = isokinetic temperature (K) and  $\Delta G^{\circ}$  = free energy (kJ/mol)

The harmonic mean temperature is given as:

$$
T_{hm} = \frac{n}{\Sigma(1/T)}
$$
\n(15)

W here,  $n =$  Number of treatments and  $T =$  Absolute temperature. Studies by Bajpai and Pradeep (2013) revealed that, the compensation theory only applies when  $Tc \neq T_{hm}$ . If  $T_c$ > $T_{hm}$ , the moisture sorption process is enthalpy driven, while if  $Tc < T<sub>hm</sub>$ , the process is considered to be entropy controlled. This is a statistical analysis test used to corroborate the compensation theory and was proposed by Krug *et al* (1976). This study was carried out to determine the safe moisture range of dried pumpkin fruit slices and to investigate the spontaneity of the sorption process.

### **MATERIALS AND METHODS**

*Sources of Raw Material:* Matured pumpkin fruits of average moisture content (94.4 %  $\pm$  0.21) and 3.8<sup>o</sup>  $\pm$  0.1 Brix were harvested the same time from a private garden. The fruits were washed using tap water to remove soil and other debris. They were then kept in an incubated refrigerator at  $4 \degree C$  for temperature to equilibrate before use for processing. Sodium metabisulphite (250 g) of laboratory grade was obtained from a chemical shop in Makurdi, Nigeria.

*Sample Preparation:* Pumpkin fruits were peeled using a stainless steel knife. The pulps of the fruits with seeds were removed and the thick mesocarp sliced into pieces of 4 mm and 5 mm thickness with the aid of a digital vernier calliper. The choice of thinness was to simulate market samples based on the information obtained from the local producers. The sliced fruits were weighed in five portions of 50 g for each of the two sizes. Sodium metabisulphite solutions of 1.0, 1.5, 2.0 and 2.5 % were prepared and placed in 500 ml beakers. To each concentration of salt, 50 g of 4 mm and 5 mm pumpkin fruit slices each were soaked in the prepared concentrations of sodium metabisulphate for 5 minutes to obtain four samples for the 4 mm size namely; P1, 4; P1.5, 4; P2, 4; P2.5, 4 and a non sulphited sample P0, 4 which was the control. Same thing was done for the 5 mm size of slices to obtain samples P1, 5; P1.5, 5; P2, 5; P2.5, 5 and a non sulphited sample P0, 5 was used as the control. The sample slices were removed, allowed to drain and arranged on non- perforated trays in a single uniformly thin layer within a hot air tray dryer so that hot air passed around one side of the slices.

This was done for the controls as well which were soaked in water containing no salt as described by Doymaz et al. (2015). Drying was carried out at 60, 65, 70, 75, and 80 °C at a drying air velocity of 1.53 m/s, during which weights of the samples were taken at the intervals of 30 minutes until constant weight of sample was achieved. Samples were subjected to descriptive sensory evaluation to obtain sample P1, 4 dried at 75 °C as the best among 4 mm thick samples and P2, 5 dried at 75 °C as the best among 5 mm thick samples. Subsequent analysis were determined on these samples and their non sulphited counterparts (controls)

#### **Moisture Sorption Studies**

Sorption studies were as described by Ariahu*et al*. (2006) with modifications. Sulphuric acid solutions were used to provide water activities ranging from 0.01 to 0.92 where, 200 ml of acid solution were introduced into each of the 500 ml airtight plastic containers arranged for the sorption experiment. In each plastic container, wire gauze was forced into place over the sulphuric acid solution to form support for the samples. Triplicate samples each of 0.5 g of the ground dried products were rewetted with known quantity of distilled water followed by mixing with a spatula for the adsorption studies. Mould growths were suppressed by use of 0.1 % (m/m) toluene. The containers were covered tightly with lids and allowed to equilibrate in a refrigerated incubator at 10, 20, 30 and 40°C respectively. The temperatures were selected to simulate temperate and tropic atmospheric room temperatures. The samples were removed and weighed every 2 days until difference between consecutive readings were < 0.5 % of each sample weight. The total time for removal and putting back in the air tight containers was about 2-5 min as recommended by the Cooperative Project, COST 90 (Ayala-Aponte 2016). This minimized atmospheric moisture sorption or adsorption during weighing. The equilibrium moisture contents were determined by material balance from the initial moisture contents (Akin *et al*., 2009).

*Determination of sorption derivatives:* BET constant and monolayer were calculated from equation (1) and the apparent sorption area of the sorbate (food material which is pumpkin) which is a derivative of BET was determined using equation (3) as shown in Table 1 for the adsorption mode of sorption. The GAB constants and monolayer were calculated from equations (7), (8) and (9). Heat of sorption was estimated using the net isosteric heat of sorption given by the Clausius-Clapeyon equation in (10) by regressing lna<sub>w</sub> at constant moisture content with the inverse of absolute temperature. The net isosteric heat of sorption was obtained from the slope while the net entropy was calculated from the intercept. Variation of isosteric heat of sorption and moisture content were evaluated using the method described by Bajpai*et al.* (2011). The enthalpy- entropy compensation theory statistical analysis test was conducted using equations (14) and (!5). .

#### **RESULTS AND DISCUSSION**

*Determination of Sorption Isotherms:* Sulphuric acid was used for generating environments of defined conditions for adjustment of  $a_w$  rather than solutions of saturated salts and glycerol for the fact that salt solutions are most popular, however, they are limited in that, they provide only discrete  $a_w$ values at any given temperature.



**Figure 1. Moisture Desorption Isotherms of Sulphited and Non-sulphited Pumpkin Fruit Slices**

**Table 1. BET Moisture Adsorption Parameters and Derivatives of Sulphited and Non-sulphited dry Pumpkin Fruit Slices**

Sample	Temp. $(^{0}C)$	$C_B$	$Mo$ (gH <sub>2</sub> O/g solid)	$S_0$ (m <sup>2</sup> /g solid)	$r^2$
$P0$ , 4	10	13.5000	12.3457	412.2402	0.848
	20	31.3333	10.6383	374.7639	0.937
	30	36.6667	9.0909	334.2489	0.973
	40	40.0000	8.3333	319.8416	0.971
P1, 4	10	8.1111	13.6986	508.2415	0.756
	20	9.6250	12.9870	412.2403	0.931
	30	9.7778	11.3636	367.3449	0.981
	40	13.6250	9.1743	301.6393	0.974
$P0$ , 5	10	44.0000	11.3636	407.7102	0.848
	20	97.0000	10.3093	382.4910	0.968
	30	20.6000	9.7087	325.4529	0.948
	40	37.5000	6.6667	259.4520	0.991
P2, 5	10	26.6667	12.5000	447.0276	0.834
	20	9.3333	11.3636	403.2786	0.903
	30	20.0000	10.00007	350.3574	0.958
	40	29.7500	8.4034	301.6393	0.921

**Table 2. GAB Regression Parameters and Derivatives for Moisture Adsorption of Sulphited and Non-sulphited Dry Pumpkin Fruit Slices**



C & K are GAB's constants,  $r^2$  coefficient of determination, M<sub>o</sub>=Monolayer moisture contentP0,4=Non sulphited pumpkin fruit slices of 4 mm,P1, 4-=Pumpkin fruit slices of 4 mm thickness, 1 % sulphited, P0, 5=Non sulphited pumpkin fruit slices of 5 mm thickness,P2, 5=Pumpkin fruit slices of 5 mm thickness, 2 % sulphite



**Figure 2. Variation of Net isosteric Heat (∆Hst) Plot A and Net Entropy (∆S) Plot B with Moisture Content for Desorption Isotherms of Sulphited and Non-sulphited Pumpkin Fruit Slices**





P0, 4=Non sulphited pumpkin fruit slices of 4 mm,P1, 4-=Pumpkin fruit slices of 4 mm thickness, 1 % sulphited, P0, 5=Non sulphited pumpkin fruit slices of 5 mm thickness, P2, 5=Pumpkin fruit slices of 5 mm thickness, 2 % sulphited.  $R^2$ =Coefficient of determination, Tc= isokinetic temperature [T<sub>hm</sub> = harmonic mean temperature (297.5802)]

Although the equilibrium relative humidity values of various saturated salt solutions at different temperatures have been tabulated and reviewed in the literature, the reported values do not always agree. The  $a_w$  of most salt solutions decrease with increase in temperature because of the increased solubility of salts and their negative heat of solution. Equilibrium moisture contents of each sample were plotted against their corresponding water activities to obtain moisture sorption isotherm curves for the adsorption mode of sorption at fixed temperatures of 10, 20, 30 and 40°C as shown in Figure (1)**.**  The curves of the moisture adsorption isotherm indicate the type ІІ isotherms which are unique for dry pumpkin fruit slices. This is in agreement with the report of Bajpai and Pradeep (2013), that the shape of isotherms is unique for each product due to the difference in capillary effects, colligative effects of dissolved compounds (such as salts and sugar) and surface interactions. The adsorption isotherms as shown in Figure 1 indicated that, equilibrium moisture content increased with increasing water activity at constant temperature. Togrul and Arslan, (2006) attributed these changes in equilibrium moisture content as being due to the inability of the food material to maintain vapour pressure at unity with decreasing moisture content. As moisture content in a food material decreases, the foods moisture tend to exhibit a lower vapour pressure, behaving as if it is in solution, and changes with atmospheric humidity. These changes in vapour pressure with atmospheric pressure are responsible for the characteristic sigmoid shape of moisture sorption isotherms of foods. Dry pumpkin fruit slices showed a sigmoid shape of type II according to BET classification in agreement with Ayala-Aponte (2016), who worked on the moisture sorption of cassava flour, Farahnaky*et al*. (2009), worked on the moisture sorption of figs.

*Determination of Sorption Derivatives:* BET constant and monolayer were calculated from equation (1) and the apparent sorption area of the sorbate (food material or pumpkin) which is a derivative of BET was determined using equation (3) as shown in Table 1. The GAB constants and monolayer were calculated from equations 7, 8 and 9. The result of GAB constants and monolayer is as shown in Table 2. Heat of sorption was estimated using the net isosteric heat of sorption given by the Clausius-Clapeyon equation in equation.(12) by regressing lnaw at constant moisture content with the inverse of absolute temperature. The net isosteric heat of sorption was obtained from the slope while the net entropy was calculated from the intercept. Variation of isosteric heat of sorption and moisture content were evaluated using the method described by Bajpai*et al.* (2011) and the result is presented in Figure 2.

The curves of the moisture adsorption isotherms indicate the type ІІ isotherms which are unique for dry pumpkin fruit slices. This is in agreement with the report of Bajpai and Pradeep (2013), that the shape of isotherms is unique for each product due to the difference in capillary effects, colligative effects of dissolved compounds (such as salts and sugars) and surface interactions. The adsorption isotherms as shown in Figure 1 indicates that, equilibrium moisture content increased with increasing water activity at constant temperature. Togrul and Arslan, (2006) attributed to these changes in equilibrium moisture content as being due to the inability of the food material to maintain vapour pressure at unity with decreasing moisture content. As moisture content in a food material decreases, the foods moisture tend to exhibit a lower vapour pressure, behaving as if it is in solution, changes with atmospheric humidity. These changes in vapour pressure with atmospheric pressure are responsible for the characteristic sigmoid shape of moisture sorption isotherms of foods. Dry pumpkin fruit slices showed a sigmoid shape of type II

according to BET classification in agreement with Ayala-Aponte (2016), who worked on the moisture sorption of cassava flour, Farahnaky*et al*. (2009), worked on the moisture sorption of figs.

*BET of sulphited and non sulphited dry pumpkin fruit slices:*  The BET sorption model as presented in Table (1) for adsorption, showed that monolayer moisture content decreased with increasing sorption temperature as expected for the fact that at high temperatures, molecules are in their excited state therefore, they tend to drift away from the active sites such that less water is bound. This is in agreement with literature findings of Aktas*et al*. (2014), Al- Mahasneh*et al*. (2014) and Bup*et al*. (2012). Surface area of sorption decreased in a similar manner like monolayer. According to Sokolowska*et al*. (2008), the values of apparent surface area of sorption obtained from the adsorption mode data may be treated as a measure of the real amount of the strongly bond water. The high values of apparent surface area of sorption obtained from dry pumpkin fruit slices are in agreement with the results of other agricultural materials like plants roots, peat and Moorish as reported by Szatanik – Kloc (2014) Sokolowska (2004), Jozefaluk and Kloc (2003) respectively.

*GAB regression parameters and derivatives:* The result of GAB regression parameters is presented in Table 2. The result showed very high values of coefficient of determination (0.896 – 0.999). This is in agreement with the result of Tsami*et al.* (1990a) who researched on water sorption isotherms of raisins, currants, figs, prunes and apricots, Aktas*et al*. (2014) that worked on sorption isotherms and net isosteric heat of sorption of plum osmotically pre-treated with trehalose and sucrose solutions and Nurtama and Lin (2010) who worked on sorption isotherm characteristics of taro flour. Monolayer of GAB decreased with increase in sorption temperature. This was due to the fact that molecules of water were in their excited state at higher temperatures and some could not bind to the active sites.

*Variation of net isosteric heat*  $(\Delta H_{st})$  with moisture content: The result of the variation of net isosteric heat of sorption with moisture content Figure 2 for pumpkin fruit slices was observed to decrease with increasing moisture content for all the samples. The decreasing isosteric heat of sorption corresponded to an endothermic process that may be associated with swelling of the pumpkin fruit that exposes new active sites where new water molecules can be absorbed by an exothermic process that compensates for the endothermic heat. The maximum enthalpy values indicate the covering of the strongest binding sites and greater water – solid interactions. The decrease in enthalpy as water content of dry pumpkin fruit denotes the covering of a less active adsorption sites and the formation of multilayers. This result is similar to the result obtained by Oleivera*et al* (2009) that determined the moisture sorption characteristic of microalgae (*Spirulinaplatensis*), Edoun and Kuitche (2014) worked on *Manihotesculenta* leaves and revealed increase in isosteric heat of sorption with increasing moisture content.

**Variation of net entropy ((∆S) with moisture content:** The net entropy of sorption variation with moisture content Figures 3 showed that, as moisture content increased, net entropy increased for all the samples. Earlier report by Perez–Alonso *et al* (2006) suggest that the minimum value of net entropy indicates the level of strongest stability of the food sample

material or the fact that the level of disorderliness is minimum, encouraging the liquid-solid interactions that makes water unavailable for microbiological, chemical and biochemical reactions. Studies by Ayala-Aponte (2016) revealed that, the increase in net entropy with increased EMC may be associated with increased mobility of water which promotes the formation of multilayer and the net entropy tends to approach the entropy of free liquid water.

**Enthalpy – Entropy relationship:** The result of the relationship between enthalpy and net entropy is presented in Table 3. The result showed that the compensation theory holds for the sorption process of dry pumpkin fruit slices for the fact that, the isokinetic temperatures ( $T_c$  = temperature at which all reactions in the series occur at the same rate) of both the sulphited and non sulphited samples was less than the harmonic mean temperature  $(T_{hm})$  with a value 297.58 °C. This indicates that the sorption process by dry pumpkin fruit is entropy controlled. This result agrees with the report of Simal*et al* (2007) that the compensation theory holds for the sorption process of a food material if  $T_c$  is not the same as  $T_{hm}$ and when  $T_c$  is greater than  $T_{hm}$ , the sorption process is enthalpy controlled however, if  $T_c$  is less than  $T_{hm}$ , the sorption process is entropy driven. This result is in agreement with the ealier report of Igleesias and Chirife (1976) in their review of isosteric heat in literature. The free energy  $( \Delta G )$  as exhibited in Table 3 shows that  $\Delta G$  is positive implying that, the sorption in dry pumpkin fruit was not a spontaneous process. This result is similar to the result obtained by Ayala- Aponte (2016) in his work on thermodynamic properties of moisture sorption in cassava flour.

#### **Conclusion**

It can be inferred from the study on adsorption isotherms and thermodynamic properties of dried pumpkin fruit slices that,the monolayer of BET is less than the monolayer of GAB. This implies that the monolayer of BET-GAB at a given sorption temperature, gives a safe moisture range that will offer the best storage stability of this product. It can also be deduced from the study that ∆G (free energy) is positive indicating that the sorption process in dried pumpkin fruit slices does not occur spontaneously.

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