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Moisture Sorption Characteristics of Banana *Shrikhand*

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ABSTRACT

Moisture desorption and adsorption phenomenon in banana *Shirkhand* were investigated at 10^0C, 25^0C and 40^0C. The isotherm plots obtained exhibited sigmoid shape at all the three temperatures and are classified as type-II. The equilibrium moisture content rose gradually at the lower water activities followed by a steep rise at the higher water activities. The effect of temperature was more pronounced on the desorption isotherm than an adsorption isotherm. Between the three models (GAB, BET and Caurie) tested for predicting the sorption data, GAB gave the best fit at all the three temperatures as indicated by % RMS and R^2 values. Properties of sorbed water viz; number of adsorbed monolayers, density of sorbed water, bound or non-freezable water and surface area of sorption were calculated for adsorption and desorption process from Caurie equation. The isosteric heat of sorption was calculated using the Clausius-Clapyron equation. Sorption isosters for each moisture contents are also been reported.

Highlights

- The monolayer values obtained using GAB, BET and Caurie decreased with increasing temperature.
- EMC shows that the banana *Shrikhand* is highly hygroscopic

Keywords: Adsorption, desorption isotherm, EMC, BET, GAB, water activity

The fermented milk and milk products occupy an important place in satisfying nutritional requirements of human being since the time antiquity. Fermented milk products have been well recognized to have therapeutic, anticholeterolemic, anticarcigenic properties (Gardiner *et al.* 2002). Amongst the various fermented milk products, dahi a well-known indigenous fermented milk products prepared by lactic acid fermentation is being converted in to *Shirkhand* because of its better shelf-life (Devshete *et al.* 2012). *Shirkhand* is a popular Indian dessert prepared by the fermentation of milk. It has a semi-soft consistency and a sweet and sour taste. *Shirkhand* originated in Persia using Frasi-shir (milk) and khand (sugar), and was later brought to the shores of Gujarat state of India by the Parsi Zohrastrian settlers (Narayana and Lingam 2013).

Milk can also serve as an excellent carrier product for an extra nutrient, and if enriched or fortified it can satisfy the nutritional needs of the population (Jana *et al.* 2011). The nutritional value of *Shirkhand* could be enhanced when it is supplemented with some fruits. Bananas (*Musa acuminata, Musa balbisiana*, and *Musa paradisiaca*) are an excellent source of vitamin B_6 and contain moderate amounts of vitamin C, manganese and dietary fiber. The study was reported on the preparation of banana *Shirkhand* using banana pulp (Narayana and Lingam 2013). For process up gradation and shelf life prediction studies, the data of the moisture sorption properties are needed. Moisture sorption isotherm describes the relationship between moisture content and water activity in food (Ricardo *et al.* 2011). The information is useful to understand re-hydration properties, drying conditions, packaging and to develop ready-to-use products.

Moisture Sorption Isotherms can be mathematically described with the help of several empirical, semiempirical and theoretical models. Brunauer, Emmett and Teller (1938) and Caurie (1981) developed two-parameter equations for describing the sorption isotherms which could help in evaluating the properties of sorbed water. In the COST 90 project on water activity, Bizot (1983) demonstrated that the 'Guggenheim-Anderson-de Boer' (GAB) equation was the three parameter theoretical model in giving the best fit for most food isotherms over a wide range of water activity, which also provides a better evaluation of the amount of water tightly bound by the primary adsorption sites. Furthermore, the knowledge of temperature dependence of sorption phenomenon provides useful information on the energetic of water sorption process in foods (Rizvi 1995). Both adsorption and desorption isotherms are necessary depending upon the particular task in hand (Varghese *et al.* 2014).

The data on moisture sorption properties on banana *Shrikhand* were not available. Hence, attempts have been made to establish moisture sorption properties of banana *Shrikhand* at 10°C, 25°C and 40°C.

The objectives of the present studies were; to review the process standardization of the selected product, to prepare the product as per the standardized method, to establish proximate compositional analysis of the product, to establish moisture sorption characteristics of the selected product at different temperatures, to fit the mathematical models to sorption data, to establish the critical parameters related to shelf-life of the product.

MATERIALS AND METHODS

Preparation of Banana *Shrikhand*: Banana *Shrikhand* was prepared as per the standardized method reported by Narayana and Lingam 2013. Fresh mixed milk was procured from the local market with 3.5% fat and 8.5% solid not fat (SNF) was used for the preparation of *dahi* using local starter culture. Milk was boiled and cooled to 30±1°C for inoculation (1.5%) and incubated at 32±1°C for 10 to 12 h until a firm coagulum was formed. Coagulum was transferred to a double muslin cloth and kept hanged for an expulsion of whey for 8 to 10 h in the refrigerated conditions (4 ± 1°C). The semi-solid *chakka* obtained after the drainage of whey was used as the base material for *Shirkhand*. 40% good quality ground cane sugar was added and mixed uniformly with the *chakka* to obtain plain *Shirkhand*. Banana *Shirkhand* was prepared by supplementing *chakka* with 20% banana pulp prepared previously from fresh bananas.

For an adsorption study the dried banana *Shrikhand* was prepared by thin layer drying in well ventilated oven at 40°C for 48 h. The final drying was achieved in a desiccator using concentrated sulphuric acid as a desiccant.

Determination of sorption isotherm

To determine the moisture sorption isotherm of Banana *Shrikhand*, sorption devises as suggested by Labuza (1968) was used for the equilibration studies. The sorption apparatus consisted of airtight wide mouth glass jar in which the sample was kept in a sample container, placed on the glass bids exposed to the humid atmosphere in a temperature controlled chambers maintained at 10°C, 25°C and 40°C.

To equilibrate the sample with each relative humidity reagent grade salt solutions as recommended by Greenspan (1977) viz., Lithium chloride (LiCl), potassium acetate (CH_3COOK), magnesium chloride ($MgCl_2$), potassium carbonate (K_2CO_3), magnesium nitrate ($MgNO_3$), sodium chloride (NaCl), ammonium sulfate ($(NH_4)_2 SO_4$), potassium chloride (KCl), potassium nitrate (KNO_3) and potassium sulfate (K_2SO_4) were used to obtain water activities from 0.11-0.97. An extra pure grade salts were dissolved in the distilled water, initially heated to 100°C and cooled to each test temperature to form the saturated slush. The level of saturated salt solution (slush) was kept at the bottom of each jar to a depth of about 0.4 cm. After taking the tare weight of each sample container, approximately 2 g of freshly made homogenous sample of banana *Shrikhand* was weighed. To prevent mold growth approximately 5 mg potassium sorbet was added to each sample. At each water activity and temperature maintained, the equilibrium was carried out for two to three weeks. The weights were recorded at regular intervals (after every 72 hrs) and the equilibrium was judged to have been attained when the difference between the three consecutive weighing did not exceed 1 mg.

Compositional Analysis

Compositional analysis of the banana *Shrikhand* and dried banana *Shrikhand* were done as per the

AOAC methods by William and George (2005) and BIS: SP: 18 (Part XI).

Mathematical models and calculations

The BET, GAB and Caurie models were chosen to fit to an experimental sorption data for banana Shrikhand. BET equation is useful in determining the multi-layer sorption isotherms. It is useful for determining the optimum moisture conditions for the storage stability of dehydrated food products. However, as reported BET relationship is valid up to a_w=0. 55 (Labuza 1985).

$$a_w / (1-a_w)m = 1/m_o C + (C-1/ m_o C)a_w \qquad \ldots(i)$$

Where, m is the moisture content (db), a_w is the water activity, m_o is the BET monolayer moisture content, and C is a surface heat constant. GAB model is widely accepted as the most useful model for characterizing an isotherm over the entire a_w range (0-0.9). The co-efficient of GAB have theoretical and physical meaning, such as providing monolayer moisture content. The GAB parameters were calculated by fitting, moisture sorption data to a 2nd degree polynomial.

$$m = m_o k_b\, aw/ [(1 - k_b\, a_w)\,(1 - k_b a_w + k_b C a_w)] \qquad \ldots(ii)$$

Where, k_b is a multilayer factor.

Caurie equation is an improvement in BET equation and was used to calculate the properties of sorbed water.

$$\ln 1/W = - \ln 1/ CW_0 + 2C/ W_0 \ln[1 - a_w/ a_w] \qquad \ldots(iii)$$

Where, W is moisture content in kg water/ kg dry solid, W_o is moisture content corresponding to saturation of all the primary adsorption sites by one water molecule (equivalent to monolayer in BET theory) and C is the density of sorbed water. Caurie's plot of $(1 - a_w/ a_w)$ vs. ln $1/W$ over the a_w range was used to obtain Caurie's slope.

Properties of Sorbed water

The number of adsorbed monolayers was obtained by the formulae (Caurie 1981):

$$S = 2/N \qquad \ldots(iv)$$

Percent bound water or non freezable water is the product of monolayer value W_o in the equation and the number of the adsorbed monolayer (N).

The surface area of adsorption 'A' was determined by the formula:

$$A = 5454/S \qquad \ldots(v)$$

Where S is Caurie's slope.

Net isosteric heat of sorption

The net isosteric heat of sorption (q_{st}) provides information on the amount of heat required to be removed in the excess of latent heat of vaporization of pure water. It can be evaluated at different moisture content using the best fitted isotherm mode as sorption data are obtained at different temperatures. The Net isosteric heat of sorption was determined using Clausius-Clapeyron equation (Labuza 1985):

$$[\partial \ln a_w / \partial 1 / T]_M = Q^{st} / R \qquad \ldots(vi)$$

Where,

Q^{st} is the isosteric sorption heat (kJ/mol), R is the gas constant (0.00831434 kJ/mol K) and T is an absolute temperature (K). Slope of the plot of ln(a_w) versus $1/T$ at constant moisture content (M) gave the net isosteric heat of sorption.

In order to ascertain the precision of fit of the sorption data, statistical tools were used such as coefficient of regression (R^2) and root mean square (%RMS) of the error.

$$R^2 = \sum_{i=1}^{n} \frac{\left(x_i - \widehat{x}_i\right)^2}{\left(x_i - \overline{x}_i\right)^2} \qquad \ldots(vii)$$

$$\%\ RMS = SQRT\ 1/n \sum\nolimits^{n}_{i=1} (x_i - x_i^{\wedge}/x_i)^2 * 100 \qquad \ldots(viii)$$

In all the equations, n is the number of observations,

x_i is the experimental value, $\widehat{x}_i$ is the value obtained by the fitting model for the i^{th} observation,

$\overline{x}_i$ is the absolute mean.

RESULTS AND DISCUSSION

Proximate composition of Banana *Shrikhand*

The proximate composition of Banana *Shrikhand* obtained for moisture, fat, protein, lactose, sucrose, starch, ash content and acidity was 56.4, 5.23, 3.56, 5.02, 25.06, 2.88, 0.65 and 0.61 respectively. Whereas that for dried banana *Shrikhand* the same was

2.83, 12.35, 6.83, 39.74, 39.46, 4.68, 1.15 and 1.43% respectively.

Moisture sorption characteristics

The moisture desorption and adsorption process for banana *Shrikhand* was determined at the temperatures of 10°C, 25°C and 40°C and within the water activity range of 0.11-0.97. The resultant isotherm plots are presented in Fig. 1 to 3.

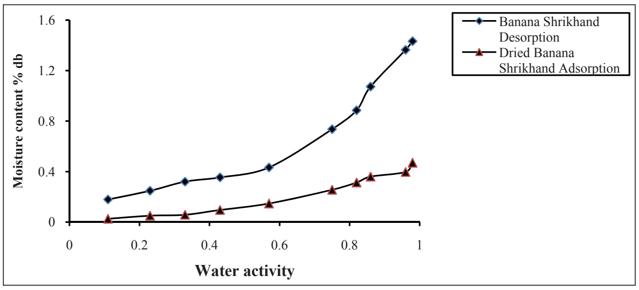

Fig. 1: Sorption isotherm for banana *Shrikhand* at 10°C

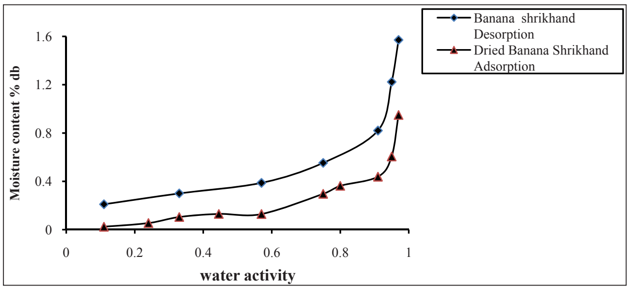

Fig. 2: Sorption Isotherms for banana *Shrikhand* at 25°C

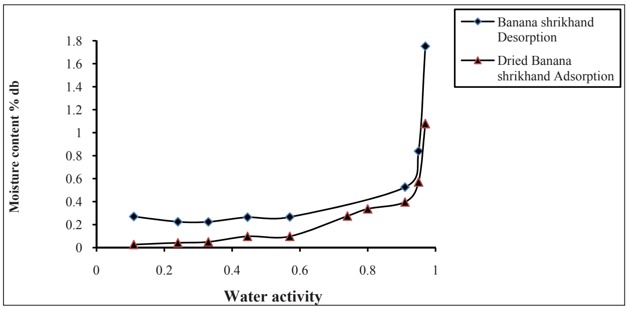

Fig. 3: Sorption Isotherms for banana *Shrikhand* at 40°C

The plots exhibited the sigmoid shape and correspond to type II as per the classification given by Brunauer *et al.* (1940). According to Bolin (1980) and Bandyopadhyay *et al.* (1987) type II isotherms are typically foods high in carbohydrates. Moisture sorption reported for many other food products typically shows the same behaviour (Khojare 2014, Shawney *et al.* 1991; Bandyopadhyay *et al.* 1987). Two bends were noted in each curve, which divides the isothermin three regions. Each region indicates the specific state of moisture in foods. As reported by Mohammad Shafiur Rahman (2010) region-I is considered as a monomolecular region (a_w< 0.05 > 0.234) in which single layer of water forms around the food which is strongly adsorbed on the individual polar groups of the substance by an ionic and Vander Waals force. Region-II indicates multimolecular region (a_w< 0.234 > 0.757). In this region more than one layer of water is bonded to food by hydrogen bonds. In this region, chemical and biochemical reactions requiring solvent water starts to take place because of the increased mobility of the solution. Region III is considered as the capillary condensation region (a_w< 0.76 >0.9) and water molecules condense on the porous structure of the food. Water in this region is in the Free State. The EMC increases gradually with an increase in water activities and temperatures. Higher levels of a_w than 0.9 could result in a marked increase of EMC and susceptibility to spoilage by micro-organisms.

Effect of Temperature on sorption isotherm

The effect of temperature on sorption isotherm of banana *Shrikhand* was observed at 10°C, 25°C and 40°C. The EMC increased as temperature increased at all the water activities in banana *Shrikhand*. The equilibrium moisture content increased from 0.17 g of water/ g solids to 0.27 g of water/ g solids as temperature increased from 10°C to 40°C at the constant water activity of 0.11. This shows that the banana *Shrikhand* is highly hygroscopic. This could be attributed to the presence of banana pulp in *Shrikhand* which adds hygroscopicity to the product. Jonnahta *et al.* (2014) reported the hygroscopic behaviour of banana flour and concluded that the value of monolayer moisture content indicated the drying process of *Nanicao* banana should not proceed to moisture content lower than 7.10g of water/100 g dry solids. Banana contains 2.88% starch, which gives the hygroscopic property to banana *Shrikhand*. The increase in water activity with the temperature indicates that the desorption in banana *Shirkhand* is an exothermic process.

However, in dried banana *Shrikhand* the effect of temperature on EMC was observed to be negligible at all water activities. The equilibrium moisture content increased from 0.026 g of water/ g solids to 0.027 g of water/ g solids when the temperature increased from 10°C to 40°C at the constant water activity of 0.11.

Increase in temperature leads to increase in water activity at the same moisture content which in turn causes an increase in reaction rates, leading to quality deterioration (Van den Berg and Bruin 1981). Several researchers also reported an increase in a_w with an increase in temperature. (Lavoyer *et al.* 2013; Khojare 2009). As the temperature increases, the structure and constituents of the materials were affected, resulting in reduction in the sorption sites and decrease in EMC. At high storage temperatures there would be a shift in water activity to the values above the critical level, for the storage of product even though the EMC is constant. Hence the product could deteriorate at higher temperatures than it would at the lower temperatures even when the EMC remains constant (Alakali and Satimehin 2009).

Modeling of sorption isotherms

A large number of sorption models have been proposed to determine the sorption isotherms (Rockland 1981; Van den Berg 1981; Bizot 1983). For banana *Shrikhand* BET, GAB and Caurie models were tested to predict the moisture sorption data and to establish the sorption behaviour of the product. The results of fitting, moisture sorption data in the models are presented in Table 1 and 2.

The BET, GAB and Caurie models appeared to be suitable to describe the sorption relationship in banana *Shrikhand*. The GAB model emerged as the best model and is widely used for foods with high sugar content (Elanin and Dieter 2013). Banana Shrikhand and dried product prepared for the study contains 32.98% and 79.21% of the total sugar respectively. From Table 1 and 2 it can be noted that the BET and Caurie equations gave relatively good fit at 10°C, 25°C and 40°C. The GAB equation was the best fit for the sorption data of banana Shrikhand. In GAB model, the value of K provides a measure of interactions between the molecules in the multilayer with the adsorbent, and it tends to fall between the energy value of the molecules in the monolayer and that of free water (Lavoyer *et al.* 2013). In the present study, the K value for banana *Shrikhand* decreased from 0.93 to 0.78 as the temperature increased from 10°C to 40°C. However, for dried product Kvalue increased from 0.83 to 1.67 as the temperature increased from 10°Cto 40°C. The results are in agreement with Kumar and Mishra (2006) for mango-soy-fortified yoghurt powder. Prediction of water sorption is required to establish water activity and water content relationship for materials (Roos 1995).

Table 1: Estimated parameters of BET and GAB isotherm equation fitted to sorption data of Banana *Shrikhand* and Dried Banana *Shrikhand* at different temperatures

Model	Temp (◦C)	m_0 g water/ 100g solids	K	C	R^2	%RMS
Banana *Shrikhand*						
BET	10	35.26	--	3.23	0.98	0.69
	25	17.14	--	-20.72	0.84	26.83
	40	17.00	--	-3.99	0.91	31.24
GAB	10	21.19	0.93	78.97	0.93	3.75
	25	17.84	0.89	-11.84	0.93	8.90
	40	16.16	0.78	-20.97	0.99	3.56
Dried Banana *Shrikhand*						
BET	10	15.10	—	-10.54	0.82	1.80
	25	14.29	—	-2.15	0.94	5.34
	40	7.58	—	2.79	0.87	7.20
GAB	10	10.15	0.83	2.61	0.85	8.86
	25	4.94	1.08	-5.80	0.97	6.94
	40	2.51	1.67	40.34	0.97	1.88

Critical Parameters

Monolayer moisture Content

It is important to know the monolayer value, because the corresponding water content of the material is considered optimal from the point of view of food stability and allows proper storage condition (a_w) to be selected (Falade *et al.* 2004). The adsorption and desorption monolayer values of banana *Shrikhand* obtained from BET,GAB & Caurie equation are presented in Table 1 to 2. The higher BET m_0 value of banana *Shrikhand* as 35.26 g water/ 100g of solids and of dried banana *Shrikhand* as 15.10 g of water/100 g of solids at 10°C may be due to the presence of starch in banana. The high monolayer value has also been attributed to combine the effect of starch, sugar and milk constituents in banana *Shrikhand*. The GAB monolayer decreased from 21.19 g of water /100g of solid at 10°C to

17.84g of water /100g of solids at 25°C and further reduced to 16.16 g of water/100g of solids at 40°C. The Caurie monolayer values for banana Shirkhand were significantly lower when compared to BET and GAB monolayer.

Properties of sorbed water

The properties of sorbed water are useful to understand the behaviour of the moisture at different temperatures and also for shelf-life determination. Caurie equation was used to estimate the properties of sorbed water for banana *Shirkhand* and dried banana *Shirkhand* at 10°C, 25°C and 40°C and values obtained are presented in Table 2.

The bound or non-freezable water in banana *Shrikhand* increased from 13.81 percent at 10°C to 29.86 percent at 40°C. The density of sorbed water in banana *Shirkhand* was observed to decrease from 0.73 g/cc at 10°C to 0.64 g/cc at 25°C. However, the density value increased to 0.75 at 40°C. This variation may be due to the varying impact of temperature on different milk constituents, particularly sugar. A similar conclusion was given by Prateek Sharma (2008) for the ready-to-use Basundi mix in which the enthalpy-entropy compensation indicates that sorption mechanism involved is enthalpy driven over the entire range of moisture content studied. Also, Jayendra *et al.* (2005) showed that there is a negative temperature effect on EMC at low water activities.

The surface area of sorption increased from 118.82 m²/g at 10°C to 174.25 m²/g at 25°C and decreased to 172.59 m²/g at 40°C. The number of adsorbed monolayer increased from 4.35 to 6.32 with an increase in temperature from 10°C at 40°C.

In dried product the bound or non-freezable water decreased from 14.77 percent at 10°C to 14.66 percent at 25°C and further decreased to 11.39 percent at 40°C. The density of sorbed water for dried banana Shirkhand decreased from 1.76 g/cc at 10°C to 1.64 g/cc at 25°C. However, it increased to 2.04g/cc at 40°C. The surface area of sorption increased from 78.92 m²/g at 10°C to 81.52 m²/g at 25°C. However, it was decreased to 64.46 m²/g at 40°C. The number of adsorbed monolayer decreased from 2.89 to 2.36 with an increase in temperature from 10°C at 40°C.

Net Isosteric Heat of Sorption

The net isosteric heat of sorption, which is the difference of the Total heat of sorption (Q_{st}) and the heat of vaporization of water, shows the energy requirement for removing moisture from the food materials (water-solid binding strength) and has a practical use in drying calculations and modeling of energy requirements (Bajpai 2011). The net isosteric heat of sorption of banana *Shrikhand* as a function of EMC was calculated using Claisisu-Clapeyron equation at 10°C and 40°C. For banana *Shrikhand* and dried banana *Shrikhand*, the net isosteric heat of sorption was plotted against moisture content and presented in Fig. 4. In banana *Shrikhand*, the net isosteric heat of sorption decreased from 38.41 KJ/Mol, K at 23.9 g of water /100 g solid to 0.51 KJ/Mol, K at 145.15 g of water /100 g solid which shows that the removal of low moisture requires high amount of heat energy when compared to high moisture content which can be evaporated using less quantity of heat. Similar behaviour has also been reported by Al-Muhtaseb *et al.* (2004) who studied the water sorption and the thermodynamic properties of

Table 2: Properties of sorbedwater in banana *Shrikhand* and Dried Banana *Shrikhand at* different temperatures

Temp (◦C)	m_0 g water/ 100g solids	Caurie's Slope	Density of Sorbed Water (g/cc)	No. of Adsorbed Monolayers	Bound or non freezable water (%)	Surface area of Sorption (m²/g)	R^2	%RMS
				Banana *Shrikhand*				
10	3.17	0.45	0.73	4.35	13.81	118.82	0.98	7.06
25	4.08	0.31	0.64	6.38	26.07	174.25	0.99	17.97
40	4.71	0.31	0.75	6.32	29.86	172.59	0.94	16.21
				Dried Banana *Shrikhand*				
10	5.10	0.69	1.76	2.89	14.77	78.92	0.99	12.94
25	4.90	0.66	1.64	2.98	14.66	81.52	0.95	15.26
40	4.82	0.84	2.04	2.36	11.39	64.46	0.96	8.85

starch powder. The isosteric heat of sorption for fresh plantain (Musa, AAB) ranged from 47.4 KJ/Mol at 18% moisture, dry solids basis, to 73.2 KJ/g Mol at 2% moisture, dry solid basis. (Johnson and Brennan 2014). It can also be attributed from Fig. 4 that the net isosteric heat of sorption is high for low moisture content which indicates a strong link between the adsorbate (water) and the adsorbent.

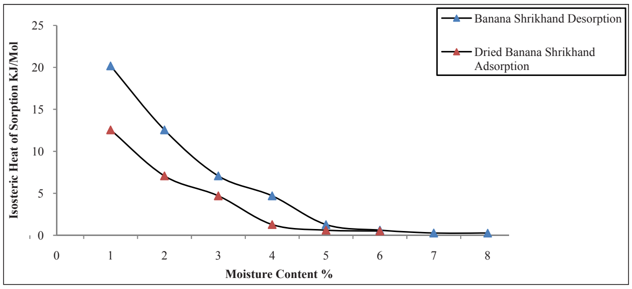

Fig. 4: Net Isosteric Heat of Sorption of Banana Shrikhand Desorption and Dried Banana Shrikhand Adsorption

The decrease in the net isosteric heat of sorption with increase in moisture content could be explained quantitatively as adsorption occurs initially at most active sites involving high energies of interaction, (Alakali and Satimehin 2009) and as these active sites become occupied, the adsorption subsequently occurs on less active sites involving lower interaction energies. Similar behaviour was reported in Indian milk products by Sawhney *et al.* (1991) Forkhoa and Jayaraj *et al.* (2006) for chhana podo. For dried banana *Shrikhand*, the net isosteric heat of sorption decreased from 20.17 KJ/Mol, K at 3.79 g of water /100 g solid to 0.26 KJ/Mol K at 41.58 g of water /100 g solid.

CONCLUSION

Moisture desorption and adsorption Isotherm curves for banana *Shrikhand* exhibited sigmoid shape and was described as type II. The EMC values of banana *Shrikhand* increased with increase in water activity and decreased with increasing temperature. Three models viz., BET, GAB and Caurie were tested over a water activity range of 0.11-0.97. The GAB model was found as the best fit for experimental sorption data in banana *Shrikhand* and dried banana *Shrikhand*. The monolayer values obtained from GAB, BET and Caurie equation decreased with increasing temperature. Caurie equation was used to determine the properties of sorbed water in banana *Shirkhand*. The net isosteric heat of sorption in banana *Shrikhand* as observed decreased with increasing moisture content which shows that the removal of lower moisture requires high amount of heat energy. Moisture sorption data generated will be useful for the selection of packaging material, drying conditions and process up gradation for banana *Shrikhand*. Future work on the shelf life modelling of this product may be carried out.

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