Moisture sorption characteristics of *chhana murki*

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Received: 02 August 2020 / Accepted: 30 October 2020 / Published online: 27 December 2020
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Abstract: Moisture sorption characteristics of *Chhana murki* were investigated at a temperature of 20°C, 30°C, and 40°C using the static gravimetric method. Sorption curves obtained were sigmoid in shape and classified as type II. Equilibrium moisture content increased with an increase in water activity and decreases with an increase in temperature. Among the BET, GAB, and Caurie models tested, the BET and GAB models were found better to predict EMC data. BET monolayer decreased from 5.77 g H₂O /100 g of solids at 20°C to 4.83 g H₂O /100 g of solids at 40°C. The GAB monolayer value decreased from 5.90 g H₂O /100 g of solids at 20°C to 5.14 g H₂O /100 g of solids at 40°C. The properties of sorbed water viz., number of adsorbed monolayer, percent bound and non-freezable water and surface area of sorption were determined using Caurie equation. Net isosteric heat of sorption decreased with a corresponding increase in moisture content, initially rapidly up to 15.89 g of water/100 g of solids and then approaching a constant value.

Keywords: *Chhana Murki*, EMC, Sorption isotherm, Water activity

Introduction

Chhana Murki is a popular Indian milk product prominent in northern and eastern regions of the country. It is small size cubes coated with sugar possessed firm body and knit-like texture. It is

prepared by heat desiccation of paneer cubes with sugar syrup in an open pan (Aneja et al. 2002). Despite its great potential as ready to eat Indian convenience food, tit-bit food for children and having good shelf life due to sugar coating, the scope for organized marketing is limited. This is due to the lack of systematic studies on production, packaging and shelf life. For process up-gradation, ready to eat food formulations, drying, packaging, and shelf-life determination, moisture sorption studies are very useful. Therefore, moisture sorption studies were carried out at the temperatures of 20°C, 30°C and 40°C to determine moisture sorption characteristics in *Chhana murki*.

Several mathematical models have been reported for assessing moisture sorption isotherms of foods by Boquet et al. 1978; Chirife and Iglesias, 1978a; Van den Berg and Bruin, 1981. Different properties of sorbed water could be obtained from the model proposed by Caurie (1981). The knowledge of temperature dependence of sorption phenomenon provides information for modeling and thermodynamics of the food system. The isosteric heat of sorption (q_{st}) yields a measure of water-solid binding strength via intermolecular attraction forces between the sorption sites and sorbet. The change in q_{st} with the change in the moisture content of the sample indicates the availability of polar sites to water vapor as the desorption/adsorption proceeds (Chung and Pfost, 1967).

Sorption isotherms of several western dairy products including milk powder, whey protein, lactose, casein, whey powder, yoghurt, and cheese have been reported, but the published report on Indian dairy products is limited. Information on moisture sorption isotherm of *Kalakand* (Deshmukh et al. 2018), *Shrikhand* (Khojare, 2018), *Bottle Guard Burfi* (Quadri et al. 2016), *Cham-Cham* (Puri and Khamuri, 2015), *Sandesh* (Khojare, 2014; Khojare and Hembade, 2015; Sahu and Das, 2010), *Dietetic Rabri* (Ghayal et al. 2013), *Dietetic Chhana Kheer* (Gautam et al. 2018), *Peda* (Biradar et al. 1985; Kumar et al. 2012), ready to use *Basundi* mix (Sharma et al. 2009), *Curd powder* (Varghese et al. 2008), *Chhana podo* (Rao et al. 2006.), *Milk Burfi* (Ramakrishna et al. 2005), *Kheer* (Jayendra Kumar et al. 2005), *Dudh churpi* (Hossain et al. 2002), *Khoa* (Sawhney and Cheryan, 1988) and *Chhana powder* and *Casein* (Bandyopadhyay et al. 1987) have been reported. However, the data on moisture sorption characteristics of *Chhana*

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murki was not available. Therefore, the research work was undertaken to determine the moisture sorption characteristics of *Chhana murki* at temperatures of 20°C, 30°C and 40°C; to fit EMC models to data; to estimate properties of sorbed water and to obtain sorption thermodynamics parameters.

Materials and Methods

Preparation of *Chhana Murki*

Good quality cow milk obtained from the local market and standardized to 3.5% fat and 8.5 % SNF was used for preparation of *Chhana murki*. Initially, milk was heated to 90°C and cooled to 70°C. The hot milk was coagulated by using 1% solution of food-grade citric acid as an acidulant that was previously heated to the milk temperature, adding and stirring gently without breaking the coagulum, till the clear whey appears. The precipitation of milk involves the formation of large structural aggregates of protein. The whey formed during the process was removed by draining through the cleaned and sanitized muslin cloth by hanging the mass till the dripping of the whey ceased. The *Chhana* obtained is kneaded and formed into a 10 mm thick flat slab; it is then cut into small cubes of about 10 mm. The cubes are cooked in boiling sugar syrup for five minutes with gentle stirring. The stirring is continued off heating till the sugar gets crystallized and coated uniformly around the cubes. After cooling *Chhana murki* is ready and may sprinkle with color and flavor and decorated with dry nut flakes (Aneja et al.2002). The proximate analysis of *Chhana murki* was carried out according to ISI,1981, and AOAC,2005 standards and obtained as moisture 28.58%, protein 8.33%, fat 9.45%, ash 0.60%, total sugars 53.04%.

Equilibrium moisture content (EMC) studies

EMC data was obtained by using the static gravimetric method according to set up by Sablani et. al., (2001). The set up was made up of sample containers in a support beaker placed on glass beads support inside the wide mouth glass bottle with vapor tight lid. Each glass bottle contained different saturated salt solutions at the bottom up to the level of 0.4 cm, represented specific relative humidity. The temperature was controlled by placing duplicate sorption containers in the thermo-regulated chamber maintained at 20°C, 30°C, and 40°C.

Ten reagent grade salt solutions in the water activity range of 0.11-0.97 adopted from Greenspan (1977) were used to equilibrate the product sample with the respective water activity. The salts were dissolved in distilled water to form saturated slush according to Jayendra Kumar et. al. (2005). The sorption containers were allowed to equilibrate to test temperatures for four days before 2 g of *Chhana murki* sample was placed into them. To prevent microbial growth 5mg of potassium sorbet was added in each sample. Equilibrium was carried out at each water activity and temperature maintained. The weights of the samples were

recorded after every 72 h interval till the equilibrium reached. The equilibrium was judged when the difference between the three consecutive weights did not exceed 1 mg. *Chhana Murki* samples took 3 to 4 weeks to equilibrate.

Fitting sorption models to EMC data

The different sorption models reported in the literature (Chirife and Iglesias, 1978) for various foods. Considering general applicability to predict sorption parameters, three models viz., BET, GAB and Caurie were chosen to fit the experimental sorption data of *Chhana murki*.

BET:

$$\frac{a_w}{(1-a_w)W} = \frac{1}{W_n C} + \frac{C-1}{W_n C} \times a_w \quad \text{.....Eq (1)}$$

GAB :

$$\frac{W}{W_n} = \frac{Gka_w}{(1-ka_w)(1-ka_w + Gka_w)} \quad \text{....Eq (2)}$$

Caurie equation (Caurie, 1981) given below was used to determine the properties of sorbed water.

$$\ln \frac{1}{W} = -\ln \frac{1}{CW_n} + \frac{2C}{W_n} \ln \frac{(1-a_w)}{a_w} \quad \text{.....Eq (3)}$$

Where a_w is water activity, W is equilibrium moisture content, W_0 is monolayer moisture content at percent dry basis, 'C' is a constant in respective equation, 'G' is the Guggenheim constant and 'k' is a correction factor for the bulk liquid.

Caurie's plot of $(1 - a_w / a_w)$ vs. $\ln 1/W$ was used to obtain slope 'S'. The numbers of the adsorbed monolayer (N) were obtained using the formula:

$$S = \frac{2}{N} \quad \text{.....Eq (4)}$$

Percent bound or non-freezable water is the product of monolayer value in the Caurie equation and the number of adsorbed monolayer. The surface area of adsorption was determined using following formula:

$$A = \frac{54.54}{S} \quad \text{.....Eq (5)}$$

To analyze the precision of fit of the sorption data to models tested the coefficient of regression (R^2), percent root mean square

Fig. 1 Moisture sorption Isotherms of *Chhana Murki* at different temperatures

(%RMS) error and percent deviation modulus (%P) were estimated from the statistical formulae below:

$$R^2 = \sum_{i=1}^n \frac{(x_i - \hat{x}_i)^2}{(x_i - \bar{x})^2} \quad \text{Eq (6)}$$

$$\% \text{ RMS} = \sqrt{\frac{1}{n} \left[\sum_{i=1}^n \left(\frac{x_i - \hat{x}_i}{x_i} \right)^2 \right]} \times 100 \quad \text{Eq (7)}$$

$$\% \text{ P} = \frac{1}{n} \sum_{i=1}^n \frac{(x_i - \hat{x}_i)}{x_i} \times 100 \quad \text{Eq (8)}$$

Where n is the number of observations; x_i is the experimental value; $\hat{x}_i$ is the value obtained by the fitting model for the i^{th} observation and $\bar{x}$ is an absolute mean.

Net Isosteric heat of sorption

The Clausius-Clapeyron equation (Rizvi, 1995) relates to the water activities and temperatures at fixed moisture content.

$$q_n^{st} = R \left[\frac{T_1 T_2}{T_2 - T_1} \right] \left[\ln \frac{a_{w2}}{a_{w1}} \right] \quad \text{Eq (9)}$$

Where, ' q_n^{st} ' is the net isosteric heat of sorption (kJ/mol), T_1, T_2 are the absolute temperatures and 'R' is the universal gas constant (8.314 J deg⁻¹ mol⁻¹ K⁻¹). The curve of net Isosteric heat of sorption plotted against moisture content at the mean temperature represents energies for water molecules binding at a particular hydration level.

Result and Discussion

Moisture sorption isotherm

The sorption isotherms of *Chhana murki* were measured at the temperatures of 20°C, 30°C and 40°C. The sorption isotherms

from the plot of equilibrium moisture content (EMC) versus the various water activities (a_w) at the different temperatures are presented in Figure 1. The *Chhana murki* isotherms obtained exhibited a sigmoid shape and corresponded to type – II BET isotherm classification according to Iglesias and Chirife (1982). This type of isotherm is typical for high carbohydrate foods as reported by Bolin (1980) and Bandyopadhyay et al. (1987). *Chhana murki* is high in carbohydrates as it contains 53.04% total sugars by weight. Two bends were observed in the isotherms and these could be said to be as a result of changes in the magnitude of the separate physical-chemical effects.

This could be observed from Figure 1 that the slope of the isotherms was moderate at $a_w < 0.55$ where relatively low moisture was absorbed for an increase in a_w . Above this level, there was a steep rise in the slope of isotherms for a small rise in a_w . In the low water activity region (0.11 - 0.55), there was a small increase in EMC for a considerable increase in a_w . However, in the high a_w region, particularly in the range of 0.68-0.97 a_w , the EMC increased significantly for a small increase in a_w . The initial moisture content of *Chhana murki* was 39% (dry basis) and this corresponds to water activities of 0.90, 0.90, and 0.91 at 20°C, 30°C and 40°C, respectively. This implies that the product would lose moisture when stored at relative humidity lower than 90%.

Two bends and three regions could be identified for *Chhana murki* from figure 1, viz., region I (a_w 0.00 to 0.33), region II (a_w 0.33 to 0.68), and region III (a_w 0.68 to 0.97). A similar observation has also been reported by Rao et. al. (2006) for *Chhana podo*. Moisture uptake was slow in region I, followed by a linear and steady rise in region II and subsequent rapid rise in region III. This may be due to the solubilization of sugars at high water activity range (above 0.65 a_w). Region I considered as monolayer moisture region, region II indicates multilayer moisture-holding which is under transition to natural properties of free water. Some of this water is available for chemical reactions. Water in region III is in the free state held into voids, crevices, and capillaries (Kinsella and Fox, 1987).

Effect of temperature on sorption isotherm

The effect of temperature on EMC was clearly shown in Figure 1. As the temperature increased, the EMC decreased, this implies that at any a_w , *Chhana murki* becomes less hygroscopic with an increase in temperature. Since water sorption generally decreases with increasing temperature, in *Chhana murki*, equilibrium moisture content was lower at higher temperatures. An increase in temperature at constant moisture content causes consistent shifting of isotherm curves on the lower side which would lead to an increase in a_w , making the product more susceptible to the microbial spoilage (Labuza et al., 1985; Bolin, 1980; Alakali and Santimehin, 2009). The negative temperature effect on the EMC has often been reported in foods with high protein content (Okos et al. 1992; Delgado and Sun, 2002a, b; Jayendra Kumar et al. 2005). The change in water activity due to temperature has been ascribed to changes in water binding, dissociation of water, and or increase of solute solubility in water as reported by Rahman (1995). It is also reported that a higher temperature results in a greater activation of the water molecules, which then breaks away from the water binding sites thereby lowering the EMC (McMinn and Magee, 2003; Sharma et al. 2009). The clear cut inversion was noticed above 0.61 a_w as all the three curves overlapped on each other for a shorter length of a_w and latter followed the separate trends as earlier. The curves at 20°C, 30°C overlapped intermittently on each other. This behavior is typical for sugar-rich food systems as a soluble component such as sugar sorb more water at higher water activity thereby overcoming the negative temperature effect due to an increase in solubility of sugar in water (Sharma et al. 2009). The effect of temperature on EMC of *Chhana murki* implies that at the same moisture content, the higher storage temperature will cause an increase in water activity values of the stored product above the critical level which may lead to deterioration.

Evaluation of Sorption Models

Three models viz, BET, GAB and Caurie (Eqs.1–3) were used to fit experimental data for sorption in *Chhana murki* and the values of the model parameters calculated are presented in Table 1 to 3.

Table 1 Estimated parameters of BET isotherm equation fitted to sorption data of *Chhana Murki* at different temperatures

Temp°C	$W_{\text{og water/}}^{\text{100g solids}}$	C	R ²	%P	%RMS
20	5.77	-16.70	0.9853	6.56	8.59
30	5.62	-9.16	0.988	6.79	11.24
40	4.83	-46.38	0.9774	5.16	6.26

Table 2 Estimated parameters of GAB equation fitted to sorption data of *Chhana Murki* at different temperatures

Temp°C	$W_{\text{og water/}}^{\text{100g solids}}$	k	C	R ²	P	% RMS
20	5.90	0.945	-18.588	0.9572	5.94	6.30
30	5.77	0.948	-12.844	0.952	6.17	6.04
40	5.14	0.959	-	0.9725	6.51	6.46

The statistical values of precision of fitness of sorption data are also given. It could be observed from the tables that R², %RMS, and %P values for BET, GAB & Caurie models were validated their reliability in predicting moisture sorption behavior of *Chhana murki*.

Since BET model being limited to the prediction up to 0.55 a_w , GAB model could be considered as better model to predict the experimental moisture sorption phenomenon and to characterize the moisture sorption behavior in *Chhana murk*. GAB model is suitably fitted over an entire range of water activity and validated by R², %P and %RMS error values presented in Table 2.

Figure 2 shows the predicted GAB plot of *Chhana murki* obtained at different temperatures. This agrees with the observations of other authors who suggested that the GAB model is a useful model to predict sorption data in milk proteins and powders (Kinsella and Fox, 1987; Joupilla and Roos, 1994). Rao et al. (2006) also reported the reliability of GAB model for predicting sorption data of *Chhana podo*. Figure 2 shows the GAB plot of *Chhana murki* obtained at different temperatures. The Caurie model showed relatively good predictability to define moisture sorption phenomenon in *Chhana murki*.

Properties of sorbed water

The values of monolayer moisture contents were obtained using GAB equation presented in Table 1. The monolayer moisture content indicates the amount of water that is strongly adsorbed to specified sites and considered the values at which food is most stable as chemical reactions are expected to be slow in this region.

Generally monolayer moisture content (W_0) decreases with an increase in temperature (Iglesias and Chirife, 1976a,b). In *Chhana murki* the BET monolayer value (Table 1) was 5.77 g H₂O/100 g of solids at a temperature 20°C and decrease to 4.83 g H₂O/100 g of solids at 40°C. The GAB monolayer value (Table 2) was 5.90 g H₂O/100 g of solids at a temperature 20°C and decrease to 5.14 g H₂O/100 g of solids at 40°C.

Fig 2. GAB plot of sorption isotherms of *Chhana Murki* at different temperatures

Fig 3. Net isosteric heat of sorption of *Chhana Murki* at different moisture content

Table 3 Properties of sorbed water in *Chhana Murki* at different temperatures

Temp. °C	W_{og} water/ 100g solids	No. of Adsorbed monolayers(<i>N</i>)	Bound or nonfreezable water(%)	Surface areaof sorption (m ² /g)	R ²	%P	%RMS
20	5.62	3.99	22.42	108.75	0.97	10.28	11.99
30	5.59	3.72	20.78	101.43	0.99	10.02	11.04
40	5.80	3.53	20.48	96.26	0.99	15.62	18.69

Hossain et al. (2002) and Rao et al. (2006) used Caurie model to characterize the sorption behavior of *dudh churpi* and *chhana podo* and suggested that Caurie model could be rated as better fit since parameters like number of adsorbed mono-layers, surface area of adsorption and percent bound water could be elucidated

from the equation. Properties of sorbed water as calculated from Caurie equation for *Chhana murki* are presented in Table 3. It is observed from Table 3 that the number of adsorbed monolayers decreased from 3.99 to 3.53 when temperature increases from 20°C to 40°C. The bound or non-freezable water in *Chhana murki* decreased from 22.42% to 20.48%. The surface

area of sorption also decreased from 108.75 m²/g to 96.26 m²/g when temperature increases from 20°C to 40°C as shown in Table 3. Similar trends were also observed by Sharma et al. (2009) in ready to use *basundi* mix and Jayendra Kumar et al. (2005) in *kheer*. It was reported by Saravacos and Stinchfield (1965) that adsorption of water can be attributed to the basic components of food such as polymeric materials viz., proteins, and soluble components e.g. sugars at high moisture content. Since all these components are present in *Chhana murki*, the temperature does not necessarily have a similar effect on their interaction with water and it could be said to have a varying impact of temperature on different constituents.

Net isosteric heat of sorption

The net isosteric heat of sorption represents energies for water molecules binding at a particular hydration level. This was obtained by applying the Clausius-Clapeyron equation (9) by considering 10°C and 40°C as T_1 and T_2 . The curve shows the plot of Isosteric heat of sorption against moisture content of *Chhana murki*, plotted at the mean temperature as 25°C. It is observed from Figure 3 that the net isosteric heat of sorption decreased with a corresponding increase in moisture content, initially rapidly (up to 15.89 g of water/100 g of solids) and then approaching a constant value. Similar trends were observed in studies involving many other Indian dairy products (Sharma et al. 2009; Jayendra Kumar et al. 2005; Sawhney et al. 1991).

The decrease in the net isosteric heat of sorption in *Chhana murki* with an increase in the amount of water sorbed is due to the availability of active sites which leads to higher energy of interaction between the sorbate and the sorption sites. As these active sites reduced, sorption occurred on less active sites giving a lower heat of sorption (Iglesias and Chirife, 1976b,c; Delgado and Sun, 2002b; Jayendra Kumar et al. and 2005; Rao et al. 2006, Zungur et al. 2017). The high heat of desorption at lower moisture content could be attributed to the chemisorptions on the polar sites and also due to strained hydrogen bonds in the food solids on dehydration (Wang and Brennam, 1991).

Conclusions

Moisture sorption isotherms of *Chhana murki* determined were sigmoid in shape and classified as type II of BET classification. The slope of the isotherms was moderate at $a_w < 0.55$ and **latter** there was a steep rise in the slope of isotherms for a small rise in a_w . The clear cut inversion was noticed in sorption curves above 0.61 a_w as all the three curves overlapped on each other for a shorter length of a_w , which is typical for sugar rich foods. Among the three models tested GAB model was found adequately good to predict the experimental moisture sorption data and to characterize the sorption behavior in *Chhana murki*. There was no significant difference observed in the monolayer values obtained from BET, GAB and Caurie models. The properties of sorbed water calculated using the data obtained

from Caurie equation. The net isosteric heat of sorption estimated using GAB parameters decreased with a corresponding increase in moisture content, the energy requirement for drying process would decrease considerably below 15.89% (d.b.) moisture content in *Chhana murki*.

Acknowledgement

The author acknowledges the financial support from University Grants Commission (UGC), New Delhi, INDIA for the major research project and he is grateful to the Principal, Vivekanand College, Aurangabad (Maharashtra) for providing necessary facilities to carry out this research work.

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