Moisture sorption characteristics of *Rasomalai* at different temperatures

Khojare Ajit

Received: 21 November 2020 / Accepted: 15 April 2021 / Published online: 07 September 2021
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Abstract: The sorption characteristics of *rasomalai* were measured at the temperatures of 20°C, 30°C, and 40°C and found to be in the water activity range of 0.11 - 0.97. The isotherm exhibit sigmoid shape and correspond to type II. In *rasomalai*, at all water activities values of equilibrium moisture content decreased with an increase in storage temperature. However, a clear cut inversion was observed intermittently at 30°C and 40°C. The BET and GAB equation fitted hold good as indicated by the statistical values R^2 , P , %RMS & chi square. The BET monolayer value in *rasomalai* decreased from 5.11 g of water/100g of solids at 20°C to 3.80 g of water/100 g of solids at 40°C. The GAB monolayer value for *rasomalai* decreased from 5.04 g of water/100 g of solids at 20°C to 3.86 g of water/100 g of solids at 40°C. The Caurie equation was used to determine properties of sorbed water viz., density of sorbed water, number of adsorbed monolayers, bound or non freezable water and surface area of sorption in *rasomalai*. The net isosteric heat of sorption in *rasomalai* ranged from 58.24 kJ/mol at 6.63 % moisture content to 0.44 kJ/mol at 32.75 % moisture content.

Keywords: Models, *Rasomalai*, Sorption Isotherm, Sorbed water, Water Activity,

Introduction

The history of Indian milk products is perhaps as old as Indian civilization itself. Traditional milk products represent the most prolific segment of our Indian Dairy Industry. *Rasomalai* is very popular traditional Indian dairy products originated in eastern parts of India. It is flattened *chhana* patties floating in thickened sweet milk. It is a very delicate, chewy/spongy sweet (Aneja et al. 2002).

India is world's largest milk producer with annual milk production of around 187.70 million tonnes in the year 2018-2019. Surplus milk (about 50%) converted into a variety of traditional products as a means of preservation (Rajorhia, 2020). Despite the immensity of volume of milk handled, preparation and marketing of these products are mostly confined to the unorganized sector due to lack of process up-gradation and standardization. *Rasomalai* is still produced manually in small sector with variable qualities depends upon the skills of *halwais* (Sharma et al. 2014). For the process upgradation, packaging and storage, data of equilibrium moisture content are needed which may be expressed in moisture sorption isotherms.

The moisture sorption isotherms describe the equilibrium relationship between water activity and moisture content of food, at constant temperature and pressure, wherein water molecules combine reversibly with food solids. The knowledge of sorption behaviour and water activity is useful to predict chemical, physical and microbiological stability of foods as a function of moisture content of the product (Rao et al. 2006). Moisture sorption behaviour of milk products and their constituents are important for understanding the problems of caking, swelling, browning reactions, mold growth and development of rancidity (Bandopadhyay et al. 1987).

The relation between the water activity and the moisture content in food can be expressed mathematically. Diverse linear, non linear and regression models have been developed which explains each one of the three zones of sorption isotherm (Ricardo et al, 2011).

Ajit Khojare (✉)
Department of Dairy Science & Technology, Vivekanand College, Samarth Nagar, Aurangabad, 431 001, M.S., INDIA
Email: khojare@yahoo.com

The BET and GAB equations are widely used for modelling of sorption isotherms of foods (Rahman & Rashid, 2006). Different properties of sorbed water could be obtained from the model proposed by Caurie (1981).

The isosteric heat of sorption (Q_{st}) is the measure of binding energy of water molecules sorbed by the solutes of the product and it helps in estimating the energy needed to remove the portion of water from the product (Sormolai and Langrish, 2015). This thermodynamic property is quite a valuable tool in understanding a mechanism of sorption behaviour in foods (Rizvi, 2005).

The moisture sorption characteristics data is extremely valuable in describing the intrinsic processing and storage induced changes in food quality. Therefore the study was carried out to establish the moisture sorption characteristics and properties of sorbed water of *Rasomalai* in the temperature range of 20 to 40°C.

Material and Methods

Preparation of *Rasomalai*

The *Rasomalai* was prepared as per the method reported (Aneja et al. 2002). Fresh cow milk obtained from the local market and standardized to 3.5% fat and 8.5 % SNF was used for preparation of *Rasomalai*. 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 whey was removed by draining through the cleaned and sanitized muslin cloth by hanging the coagulated mass till the dripping of the whey ceased. The obtained *channa* is kneaded in to smooth dough. The dough is portioned and rolled into balls of size 15 mm having smooth texture without cracks. The balls were then flattened like patties. These flattened patties were cooked in clear sugar syrup (40°Brix) for 15 minutes. During cooking small amount of water was added to makeup water loss due to evaporation and to maintain sugar syrup concentration. After cooking the *channa* patties were transferred to a container with water at 30-35°C for texture stabilization and colour improvement for 10 minutes. The texturized *channa* patties were then transferred to 50-60% sugar syrup for first stage dipping for two hours followed by second dipping in 40-50% sugar syrup for two hours. The sweet swollen *channa* patties were subsequently stored in sweetened milk (5 to 6 percent sugar (w/v)) condensed to one fourth of its volume. The flattened patties of *rasomalai* soaked in sweetened milk were removed and used for moisture sorption experimentation.

Compositional analysis

The proximate compositional analysis of *rasomalai* was done as per BIS: SP: 18 (Part XI)-1981 and AOAC (2005). The approximate

composition of the products obtained as moisture 54.34%, Protein 8.24%, Fat 7.27%, Ash 2.2% and total sugars 27.95%.

Determination of sorption isotherm:

To study the moisture sorption phenomenon in *rasomalai* the static gravimetric method was used as recommended by Sablani et al (2001). The sorption devices consisted of airtight sealed wide mouth glass bottles in which sample was placed in small container and exposed to the humid atmosphere created in the bottle. The array of sample bottles in triplicate were placed in the temperature controlled chambers at 20°C, 30°C and 40°C. To create humid atmosphere, reagent grade salt solutions (saturated slush) were prepared for the water activity values of 0.11 – 0.97 (Greenspan, 1977). Approximately 2 g of *rasomalai* was weighed in each sample container. To prevent microbial growth approximately 0.005g of potassium sorbate was added to each sample. The weight of the samples were taken at the regular intervals after every third day until there was no change in weight (± 0.0001 g) for three consecutive readings. The *rasomalai* samples took three weeks to reach equilibrium. The total time of weighing and replacing the sample did not exceed 30s for all trials. An average of triplicate measurement was used to develop the moisture sorption isotherms.

Isotherm models and calculations

BET (Brunauer-Emmet-Teller) and GAB (Guggenheim-Anderson-DeBoer) equations are widely reported for modelling of sorption isotherms of foods (Rahman & Rashid, 2006) and therefore both the models were selected to ascertain their predictability for moisture sorption data of *rasomalai* and to estimate critical moisture sorption parameters. The BET monolayer moisture content is sometimes considered the most stable moisture content in food. However, BET relationship is only valid up to $a_w = 0.55$. BET model is given as:

$$\frac{a_w}{(1 - a_w)W} = \frac{1}{W_0 C} + \frac{C - 1}{W_0 C} \times a_w \quad \text{Eq(1)}$$

Where, ' W ' is the moisture content (db), a_w is the water activity, W_0 is the monolayer moisture content, C is a surface heat constant. The slope obtained from the plot of $1/(1 - a_w)$ versus a_w allows the determination of BET parameters. The GAB model is most popular model reported to fit for entire range of water activity by second degree polynomial. It is given as:

$$\frac{W}{W_0} = \frac{Gka_w}{(1 - ka_w)(1 - ka_w + Gka_w)} \quad \text{Eq(2)}$$

Where, k is a multilayer factor and G is Guggenheim constant in GAB equation. The GAB parameters can be determined by linear multiple regression after parabolic transformation of the equation 2.

Caurie equation (Caurie, 1981) is an improvement in BET equation and is most useful to determine properties of sorbed water in foods.

$$\ln \frac{1}{W} = -\ln \frac{1}{CW_0} + \frac{2C}{W_0} \ln \frac{(1-a_w)}{a_w} \quad \text{.....Eq(3)}$$

Where, in equation 3 is density of sorbed water in foods. Caurie's plot of $(1 - a/a_w)$ versus $\ln(1/w)$ over the a_w range was used to obtain Caurie's slope (S). The number of adsorbed monolayers was obtained by the formulae:

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

Percent bound water or non freezable water is the product of monolayer value (w_0) in the Caurie equation and number of adsorbed monolayers (N). The surface area of adsorption (A) was determined by the formula:

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

Net isosteric heat of sorption

Net isosteric heat of sorption can be calculated using Clausius-Clapeyron equation, relating the water activities and temperatures at a fixed moisture content (Bell and Labuza, 2000).

$$q_n^{st} = R \left[\frac{T_1 T_2}{T_2 - T_1} \right] \left[\ln \frac{a_{w2}}{a_{w1}} \right] \quad \text{.....Eq(6)}$$

Where, is the isosteric sorption heat (kJ/mol), R is the gas constant (0.00831434 kJ/mol K) and T_1 , T_2 are the absolute temperatures (K). This procedure assumes that heat of sorption is independent of temperature change. The plot of net isosteric heat of sorption versus moisture content gives the change in energy values to eject the molecules of water from food products.

Statistical analysis

In order to compare the precision of fit of the sorption data in selected mathematical models, statistical tools such as coefficient of determination (R^2), mean relative percent deviation modulus (P), percent root mean square error percent (%RMS) and chi square test were used (Best & Kahn, 1998).

Results and Discussion

The sorption isotherm of *rasomalai* was measured at the temperatures of 20°C, 30°C, and 40°C and in the water activity range of 0.11 - 0.97. The isotherms plotted by joining the experimental values of the equilibrium moisture content against different water activities at the temperature maintained are presented in Fig. 1.

Fig 1 Moisture sorption isotherms of *Rasomalai* at different temperatures

For *rasomalai*, part of the isotherm data was obtained by adsorption, where the water activity of the salt slurries was below initial a_w of the material and rest desorption where the water activity of salt slurries was above initial a_w of the material. The isotherm thus obtained for *rasomalai* exhibit sigmoid shape and correspond to type II as per the classification of Brunauer (Ricardo et al. 2011). The sigmoid shape of isotherm was also reported for other Indian dairy and products viz., *Khoa Peda* (Pagire et al. 2020), *Kalakand* (Deshmukh et al. 2018), *Shrikhand* (Khojare, 2018), *Bottle Guard Burfi* (Quadri et al. 2016), *Cham-Cham* (Puri & Khamuri, 2015), *Sandesh* (Khojare, 2014; Khojare & Hembade, 2016; Sahu & 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 & Cheryan, 1988) and *Chhana powder* & *Casein* (Bandyopadhyay et al. 1987).

From Fig. 1 is observed that the equilibrium moisture content in *rasomalai* isotherm rose gradually at lower water activities 0.12 - 0.44 at 20°C, 0.11 - 0.52 at 30°C and 0.11 - 0.51 at 40°C followed by an accelerated rise at higher water activities at all the three temperatures. Also at all the temperatures, as the water activity increased EMC also increased. The EMC at 20°C, 30°C, and 40°C, increased from 7.18%, 4.50 % and 2.69 % at around 0.12, 0.11 and 0.11 a_w to 76.26%, 37.90% and 37.90% at 0.94, 0.92 and 0.89 a_w respectively. *Rasomalai* isotherms showed three regions of typical isotherm reported for milk proteins (Kinsella and Fox, 1987).

Effect of temperature on sorption isotherm of *rasomalai*

In *rasomalai*, at constant water activity level, values of equilibrium moisture content decreased as an increase in temperature viz., the EMC at around a_w of 0.50 is 11.76 at 20°C compared to 8.22 and 8.01 at 30°C and 40°C respectively. The negative temperature effect on the EMC has often been observed

in foods with high protein content (Okos et al.1992). It has also been reported that the water activity shift by temperature is mainly due to changes in water binding, dissociation of water and or increase of solute solubility in water. For constant water content, the increase in water activity with temperature indicates that the sorption in *rasomalai* is an exothermic process as activity potential increased to retain constant moisture content ($\Delta G = \Delta H - T\Delta S$). However, it is observed from Fig. 1 that, in the isotherm plot of *rasomalai* at 30°C and 40°C as the water activity and temperature increased the swinging (crossing over) of isotherms took place intermittently. This inversion is due to the fact that at lower temperature water activity values sorption takes place mostly by biopolymers but as the water activity and temperature increased beyond the intermediate region, moisture begins to sorbed primarily by the sugars and other low molecular constituents leading to swinging of isotherms (Saravacos and Stinchfield,1965). *Rasomalai* contain relatively high level of sugars (27.95%) and it is reported that dissolution of sugars at high temperature offset the opposite effect of temperatures on high molecular weight compounds (Aviara,2020). Similar phenomenon was observed by Sharma et al. (2009) in ready to use *basundi* mix.

Fitting of sorption isotherm models to *rasomalai*

For *rasomalai* BET and GAB models were used to predict the experimental moisture sorption data and to understand the sorption behaviour of the product. It could be observed from Table 1 that for *rasomalai* the BET equation hold good within water activity range of 0.11 to 0.52.

Though the BET model is valid over the limited range of water activity, the BET monolayer represents tightly bound water and could be suggested as optimum water content for the stability of *rasomalai*. The chemical reactions are therefore expected to be slow in monolayer region. From Table 1 it could be observed that the BET monolayer value in *rasomalai* decreased from 5.11g of water/100g of solids at 20°C to 3.89g of water/100g of solids at 30°C and it was further decreased to 3.80 g of water/100 g of solids at 40°C. The negative temperature effect on EMC has often been observed in foods with high protein content. (Jayendra et al. 2005; Okos et al.1992; Delgado and Sun, 2002a,b).

GAB equation on the other hand is fitted to the moisture sorption data of *rasomalai* in the water activity range of 0.11-0.94. The equation holds good as indicated by the statistical values viz., R^2 , P and %RMS shown in Table 2.

Since GAB equation is useful to predict the moisture data over an entire range of water activity the graph was plotted using the GAB predicted values for *rasomalai* and shown in Fig. 2. The GAB monolayer value calculated for *rasomalai* (Table, 2) was decreases from 5.04 g of water/100 g of solids at 20°C to 4.00 g of water/100 g of solids at 30°C, it was further decreased to 3.86 g of water/100 g of solids at 40°C. For both the equations the statistical agreement between the experimental and predicted values are quite satisfactory and well within the limits of experimental error. Furthermore, it was observed from Table 1 and 2 that, there is no significant difference as inferred from unpaired t test ($P = 0.95$ and $df = 4$) in the monolayer moisture contents calculated from the BET and GAB equation.

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

Temp°C	W_0 g water/100g solids	C	R^2	P	%RMS	χ^2
20	5.11	-124.30	0.95	9.15	9.23	1.33
30	3.89	273.52	0.99	3.11	3.08	3.51
40	3.80	10.80	0.92	9.80	9.39	0.61

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

Temp°C	W_0 g water/100g solids	K	C	R^2	P	%RMS	χ^2
20	5.04	0.09	-57.86	0.95	5.57	5.11	0.39
30	4.00	0.99	65.31	0.94	7.75	8.88	2.31
40	3.86	1.03	9.39	0.92	9.54	8.31	2.37

Table 3 Properties of sorbed water in *Rasomalai* at different temperatures

Temp.°C	Density of sorbed water(C)(g/cc)	No. of adsorbed monolayers(N)	Bound or nonfreezable water($w_e \times N$)(%)	Surface area of sorption(A) m^2/g
20	1.42	3.80	20.54	103.85
30	1.97	3.11	19.15	85.03
40	2.19	2.62	15.09	71.63

Fig. 2 Predicted values of EMC of *Rasomalai* at different temperatures using GAB model

Properties of sorbed water in *rasomalai*

The Caurie equation (Eqn. 3) is highly been recommended to determine properties of sorbed water in food products (Rao et al, 2006). This model has been used to characterize sorption behaviour of *dudh churpi* by fitting an equation to an entire range of water activity (Hossain et al. 2002). Therefore, Caurie equation was used to determine properties of sorbed water in *rasomalai* viz., density of sorbed water, number of adsorbed monolayers, bound or non freezable water and surface area of sorption in *rasomalai*. The properties of sorbed water in *rasomalai* were determined at 20°C, 30°C and 40°C and presented in the Table 3.

It is observed from the table 3 that in *rasomalai* density of sorbed water increased from 1.42 g/cc at 20°C to 1.97g/cc at 30°C and it was further increased to 2.19 g/cc when temperature increased to 40°C. Similar observations also reported by Sharma et al (2009) in ready to use *basundi* mix. Bound or non-freezable water in *rasomalai* decreased from 20.54% at 20°C to 19.15% at 30°C. It was further decreased to 15.09 % when temperature rose to 40°C. The surface area of sorption decreased from 103.85 m²/g at 20°C to 85.03 m²/g at 30°C, it was further decreased to 71.63 m²/g at 30°C. This indicates that the total number of binding sites decreased through protein-protein, protein – carbohydrate and protein lipid interaction (Jayendra et al. 2005). This also reflects a reduction in exposure of charged polar groups and carboxyl function of peptide bonds that binds water.

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

Net heat of sorption of *rasomalai*

The net isosteric heat of sorption varies with the amount of water absorbed by the substrate. The Clausius-Clapeyron equation (Eqn 6) was used to calculate net isosteric heat of sorption in *rasomalai* as a function of moisture content by considering 20°C to 40°C as T_1 and T_2 . The relationship between net isosteric heat of sorption and moisture content is plotted (Fig.3).

The net heat of adsorption decreased with increased in moisture content, initially rapidly up to 19.97g of water/100 g of solids and latter approached a constant value as observed in many other food systems (Khojare & Hembade, 2016, Rao et al. 2006, Sawhney et al, 1991). The decrease in the net isosteric heat of sorption in *rasomalai* with increase in amount of water sorbed is due to the fact that initially, sorption occurs on the most active sites, giving rise to higher energy of interaction between the sorbate and the sorption sites. As these active sites become occupied, sorption subsequently occurred on the less active site in *rasomalai* giving lower heat of sorption. Therefore, the water binding in *rasomalai* is weak till the moisture content reaches to 19.97 %. It was observed that, quite a substantial amount of energy must be supplied to desorb the last traces of water from *rasomalai*. The net isosteric heat of sorption in *rasomalai* ranged from 58.24 kJ/mol at 6.63 % moisture content to 0.44 kJ/mol at 32.75 % moisture content.

Conclusions

The moisture sorption isotherm of *rasomalai* was measured at three temperatures of 20°C, 30°C, and 40°C and in the water activity range of 0.11 - 0.97. The isotherm exhibits sigmoid shape and correspond to type II. A clear cut inversion was observed in *rasomalai* isotherms at 30°C and 40°C could be attributed to solubility of sugar at high temperatures. The BET and GAB equation fitted to sorption data of *rasomalai* hold good as indicated by the statistical tools. Though the BET and GAB monolayer values reported are useful to understand sorption behaviour of water in *rasomalai*, GAB equation could be more useful as it has the capability to fit to an entire range of water activity than BET. The properties of sorbed water viz., density of sorbed water, number of adsorbed monolayers, bound or non freezable water and surface area of sorption were calculated by Caurie equation. The heat of adsorption decreased with increased in moisture content, initially rapidly up to 19.97g of water/100 g of solids and latter approached a constant value as observed in many other food systems.

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