Moisture sorption characteristics of freeze dried whey–grape beverage mix

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Abstract Moisture sorption isotherms of freeze dried whey–grape beverage powder were determined at 20, 30 and 40 °C. A gravimetric static method was used under 0.11–0.85 water activity range and the sorption isotherms were found to be Type II. Various mathematical models were fitted to experimental data and it was found that Peleg model suits best in describing the equilibrium moisture content–equilibrium relative humidity relationships of instant whey–grape beverage mix over the range of temperatures studied. The net isosteric heat of sorption varied between 5.22 and 1.12 KJ/mol at moisture level varying between 1 and 9 % db. At moisture content below 1 % (db) the isosteric heat of sorption increased sharply for freeze dried whey–grape beverage powder and value of 49.08 KJ/mol was estimated.

Keywords Freeze drying · Whey beverages · Grape juice · Powder · Modelling · Moisture sorption isotherms

Introduction

The manufacture of various value-added dairy products generates large quantities of whey with potential source of nutrients which is not being completely utilized so far in Indian dairy industry. Whey contains 20 % of protein found in milk. It is proved to be an extraordinary nutritional material. (Jindal et al. 2004). Presence of lactose, minerals, protein and water soluble vitamins makes whey a highly

nutritious product. The incorporation of paneer whey into various fruit beverages is one of the most attractive avenues for its utilisation. The main advantages associated with whey beverages is the healthful combination of the fruit based, vitamin containing components and the dairy based calcium and whey proteins. Freeze drying is one of the most sophisticated methods used for drying biological components. The attempt to find healthier foods combined with the use of whey by the dairy industry has favoured the production of whey–fruit based beverages. In the present study, whey–fruit based beverages were formulated and were subjected to freeze drying to obtain ready to reconstitute beverage mixes.

A sorption isotherm represents the relationship between equilibrium moisture content (EMC) of a material with the relative humidity (RH) of the surrounding environment at a particular temperature. These are useful in determining several product parameters related to sorption process. Sorption isotherms of several food products including whey proteins, casein, lactose, whey powder, milk powder, cheese and yoghurt have been established, but published reports on Indian dairy products are scarce (Rao 2000). There are many models to fit the moisture isotherm of food products. In some models, such as the SPS (Papadakis et al. 1993; Henderson 1952), the temperature dependence is directly expressed in the equations. Other models, such as the Guggenheim–Anderson–de Boer (GAB) and Brunauer, Emmett and Teller (BET) do not include the temperature dependence relationship directly, but this relationship is reflected in the model coefficients, which are temperature dependent. Amorphous lactose in milk powder holds more water than crystalline lactose (Rennie et al. 1999). During the sorption experiment, particularly using the static method, crystallization of the amorphous lactose occurs in the milk powder sample. This phenomenon is well known (Berlin et al. 1968; Jouppila and Roos 1994). Berlin et al. (1968) used an

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electronic recording microbalance to record gravimetric changes in a precise absorption and desorption experiment on the dehydration dairy products. Studies on hysteresis effect in sorption isotherms is also reported (Alam and Singh 2011).

Freeze-drying resulted in a highly porous product with small pores, which sorbed more water than the other dried materials (Tsami et al. 1999). Sorption isotherms of freeze dried food powders and model systems had been investigated by several research workers: instant tea (Sinija and Mishra 2008), dairy powders (Foster et al. 2005); pineapple pulp powder (Gabas et al. 2007) garlic powder (Rahman and Al-Belushi 2006) and model fruit powders (Tsami et al. 1999). Sean et al. (2005) studied desorption isotherm of milk powders at elevated temperatures and over a wide range of relative humidity using a dynamic method and generated results that can be used in spray drying simulations or spray drier designs for dairy powder production. The modified Chung-Pfost equation was found to be a good model for moisture adsorption and desorption of yoghurt powder spray (Stencl 2004). According to Kumar and Mishra (2006), both Oswin and GAB models were acceptable in describing equilibrium moisture content–equilibrium relative humidity (EMC–ERH) relationships for yogurt powder samples over the range of temperatures from 20 to 50 °C. According to Sinija and Mishra (2008), Peleg model fits best the isotherms of instant tea and green tea granules.

Yu and Li (2012) studied the sorption isotherms of colostral whey powders. Scientific information on EMC of whey–grape beverage mix at various relative humidity and temperatures are not available. There is also a need for comprehensive study of the equilibrium moisture contents of whey–grape beverage to understand its drying and storage behaviour. The present study was carried out with the objective of determining moisture sorption isotherms for ready to reconstitute whey–grape beverage mix and also to analyze the data with the help of six sorption isotherm equations available in the literature. Efforts were also made to find out the most suitable model describing the isotherms of whey–grape beverage mix and to calculate the net isosteric heat of sorption from the experimental data

Materials and methods

Preparation of freeze dried whey–grape beverage mix

Blue Grapes (*Bangalore Blue*) were washed, disinfected with Potassium permanganate and juice was extracted in a Screw type Juice extractor (Raylens, India), filtered and then pasteurized in a Sanitary 3A Stainless Steel Juice Pasteurizer (Sumukhi Associates, India) at 85 °C for 30 min. Whey was prepared from milk (Nandini Dairy, Mysore) heated to 90 °C. The milk was coagulated with 1 % citric acid solution slowly

added into it with gentle stirring. The volume of citric acid solution used was sufficient to give a concentration of 2.5 g citric acid per litre of milk. The whey was separated by filtration with a muslin cloth. Whey and grape juice were mixed in the proportion 50:50 (v/v). Compositional analyses of the whey–grape beverage showed that it contained 16 % Total solids, 0.2 % fat, 0.75 % protein, 14.87 % total sugar and 0.44 % ash. The acidity of the beverage in terms of citric acid was estimated to be 1.7 % and its pH was found to be 3.57.

The Total Soluble Solids (TSS) content was adjusted to 30° Brix using sugar and was subjected to freeze drying in a pilot scale freeze Dryer (Martin Christ, Germany). The initial freezing was done to –40 °C and during drying the temperature increase up to 60 °C. A vacuum of 100 mmHg was maintained during freeze drying. The freeze dried powder containing 2–3 % moisture was used for adsorption studies and the beverage subjected to freeze drying was used for desorption studies. The moisture content in powder was determined gravimetrically by drying in a vacuum oven at 70 °C for 17 h.

Sorption studies

The procedure suggested by Iglesias and Chirife (1982) was followed for deriving sorption isotherms at 20, 30, 40 °C. These temperatures were selected because during freeze drying the maximum temperature reached lies in this range and freeze dried powders store well under ambient conditions. The initial moisture content in powder samples were determined by drying in a vacuum oven (Ranganna 2004). Two to three grams samples of beverage and its powder filled in sterilized glass weighing dishes were placed in six separate desiccators containing saturated salt solutions for maintaining relative humidity (RH) levels from 11 to 85 %. The salt solutions used and corresponding relative humidities at different temperature are reported by several authors (Young 1967; Benado and Rizvi 1985; Gal 1975; Labuza 1984; Palipane and Driscoll 1992). The six jars were placed in an oven adjusted to a fixed temperature for 24 h so as to bring the salt solutions to a constant temperature. Triplicate samples were used (2–3 g each) for both adsorption and desorption experiments. The air inside the desiccators was sucked with the help of a vacuum pump. These desiccators were kept in an incubator maintained at 20, 30, 40 °C thermostatically. A glass dish containing 5 ml toluene was placed in desiccator with relative humidity higher than 75 % to check mold growth (Labuza 1984). The samples were weighed periodically till they attained equilibrium, after which they were analyzed for moisture content. To establish moisture sorption isotherms, the equilibrium moisture contents, determined by static gravimetric method, were plotted against water activity. The hygroscopic equilibrium of samples were reached in 7–9 days for both desorption and

adsorption. After the equilibration moisture content in samples were determined by subtraction method and expressed as g water/100 g solids. To establish moisture sorption isotherms, the equilibrium moisture contents were plotted against water activity.

Modeling of sorption isotherms

The different sorption models presented in Table 1 were chosen to fit the experimental sorption data because they are most widely used for several foods. M_e , X_m , a_w and t represent equilibrium moisture content, monolayer moisture content, water activity and temperature (C), respectively. The other symbols (A , B , C , K , K_1 , K_2 , n_1 , and n_2) are isotherm constants. Model parameters were estimated by taking the equilibrium moisture content to be the dependent variable. To calculate the equation parameters, a nonlinear regression analysis minimizing the residual sum of squares was applied within the range of 0.07–0.85 water activity, except for the BET equation which is valid only for water activity below 0.5.

The data consisting of equilibrium water contents at different temperatures and humidity levels were statistically analysed. The coefficients of various sorption equations were determined by means of standard regression technique using Origin Pro 8 software. The various sorption models were evaluated for their suitability in predicting the sorption behaviour of the sample on the basis of the coefficient of determination (R^2) and reduced chi square values and also on the basis of residual plots. The difference between the measured and predicted EMC values at various water activities were defined as residuals. The residuals were plotted against predicted values of EMC. A model is considered acceptable if the residual values fell in horizontal band centered around zero, displaying no systematic tendencies (i.e., random in nature) towards a clear pattern.

Table 1 Moisture sorption isotherm models used for analysis of ERH-EMC data of Freeze dried whey-grape beverage

Model	Mathematical expression
GAB	$Me = X_m C K a_w / [(1 - K a_w)(1 - K a_w + C K a_w)]$
BET	$Me = X_m C a_w / [(1 - a_w)(1 - a_w + C a_w)]$
Oswin	$Me = A [a_w / (1 - a_w)]^B$
Peleg	$Me = K_1 a_w^{n_1} + K_2 a_w^{n_2}$
Modified Henderson	$Me = [-\ln(1 - a_w) / (A(t + C))]^{1/B}$
Modified Halsey	$Me = [\exp(A + Bt) / \ln(a_w)]$

Me , X_m , a_w and t represent equilibrium moisture content, monolayer moisture content, water activity and temperature (C), respectively

A , B , C , K , K_1 , K_2 , n_1 , and n_2 are isotherm constants

GAB Guggenheim–Anderson–de Boer; BET Brunauer, Emmett and Teller

If residual plot indicates clear pattern, the model is not acceptable.

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

The reduced chi square is obtained by dividing the residual sum of squares (RSS) by the degrees of freedom (DOF).

Heat of sorption

The isosteric heat of sorption is a differential molar quantity derived from the temperature dependence of the isotherm, which represents the energies for water molecules binding at a particular hydration level. The net isosteric heat of sorption (q_{st}) was determined using the Clausius–Clapeyron equation (Labuza 1984):

$$\ln \frac{a_{w1}}{a_{w2}} = \frac{q_{st}}{R} \left(\frac{1}{T1} - \frac{1}{T2} \right) \quad (2)$$

where a_{w1} and a_{w2} are water activities at temperatures $T1$ and $T2$, respectively and R is the universal gas constant (8.314 J/mol K). The isosteric heat of sorption (Q_{st}) was calculated from the relationship $Q_{st} = q_{st} \pm \Delta H_v$, where ΔH_v is the latent heat of vaporization of pure water at 30 °C (43.86 kJ/mol), the average of the two temperatures used in the study (20 and 40 °C).

Results and discussion

The sorption isotherms of freeze dried whey–grape beverage mix powder, at 20, 30, 40 °C are shown in Fig. 1. At the

EMC= Equilibrium Moisture Content

Each observation is a mean of 3 replications

Fig. 1 Adsorption isotherms of freeze dried whey grape beverage mix at different temperatures

same temperature, the higher water activity, the larger was the equilibrium moisture content. This behaviour is manifested in the form of a sigmoid shaped curve, thus reflecting a Type II isotherm (Brunauer et al. 1940). Physical adsorption on micro porous solids can result in type II isotherms, and correspond to multilayer formation (Adamson 1990). Sorption isotherms of whey grape mixture are sigmoidal curves normally associated with monolayer–multilayer uptake on the non-porous or macroporous surface of a powder. The freeze-dried beverage powder has macroporous particles that permit fast uptake of water molecules by capillary action and surface water interactions. Enthalpy–entropy compensation theory was applicable for adsorption process of freeze dried colostoral whey powders, and the adsorption processes were enthalpy-driven (Yu and Li 2012).

The composition of amorphous sugars is very important in determining the sorption behaviour and stickiness of a multi component food powder. Here the mix components include lactose from whey and glucose/fructose from grape juice and added sucrose. The addition of sucrose decreased the stickiness of the juice during drying.

Figure 2 shows the adsorption and desorption curves at 30 °C for whey–grape beverage. Similar behaviour of adsorption and desorption isotherms was observed for other temperatures also. The figure clearly shows that the EMC for desorption was higher than that for adsorption, at a particular water activity. Hysteresis was more prominent at water activity values above 0.3. Some thermodynamically irreversible processes must occur during desorption or adsorption. Polar sites in the molecular structure of the material are almost entirely occupied by adsorbed water in the wet condition. Upon drying and shrinkage, the molecules and their water holding sites are drawn closely enough

Each observation is a mean of how many 3 replications(n=3)

Fig. 2 Hysteresis effect at 30 °C for instant whey–grape beverage mix. Each observation is a mean of how many three replications ($n=3$)

together to satisfy each other. This reduces the water holding capacity of the material upon subsequent adsorption. Hysteresis effect in sorption isotherms of aonla flakes has been described by Alam and Singh (2011). The freeze-dried powders show a high degree of hygroscopicity as their moisture uptake were greater than 50 % of their dry weight after storage for less than 1 week at RH of less than 90 % (Callahan et al. 1982). The phenomenon of deliquescence is important in Freeze dried powders because the exposure of solids to high RH results in the formation of a liquid phase where chemical reactions may be accelerated or physical changes catalysed. The deliquescence of the materials results from the dissolution of the particles by water adsorbed at the surface to form saturated solution at high RH (Van et al. 1983; Kontny and Zografis 1995).

The sorption curves for whey–grape beverage are drawn as EMC against the a_w (ERH). These curves are used to estimate the coefficients of the different sorption models and it was found that Peleg model where M_e and a_w represent equilibrium moisture content and water activity, respectively and other symbols are isotherm constants, gives best fit to the experimental data of freeze dried whey–grape beverage mix, with highest values of R^2 and lowest values of chi square than other models for sorption isotherms for a wide range of water activity. Values of the various coefficients,

Table 2 Estimated Parameters of the selected models fitted to sorption data for freeze dried whey–grape beverage mix

Model parameters	20 °C	30 °C	40 °C
Peleg			
k1	0.0526	0.50683	0.56888
k2	1.0194	0.0364	0.04635
n1	0.04	5.93	8.0129
n2	7.78	0.235	0.39738
R^2	0.99726	0.99722	0.99241
Reduced χ^2	1.45E-5	1.43E-5	1.42E-5
BET			
X_m	0.06731	0.03821	0.03171
C	0.5130	1.47	1.8567
R^2	0.99	0.98	0.98
Reduced χ^2	1.35 E-4	7.4 E-4	3.4E-4
GAB			
X_m	0.03489	0.03016	0.01939
C	2.0664	2.8926	12.35
K	1.0606	1.02884	1.0616
R^2	0.991	0.9923	0.9932
Reduced χ^2	4.26 E-5	1.95E-5	3.66 E-5

C, K, K_1 , K_2 , n_1 , and n_2 are isotherm constants

GAB Guggenheim–Anderson–de Boer model; BET Brunauer, Emmett and Teller model; X_m monolayer moisture content

regression coefficient (R^2) and reduced chi square (χ^2) for all the six models fitted at three different temperatures, are presented in Table 2. Figure 3 shows the residual plots for Peleg, GAB and BET models fitted to sorption data of freeze dried whey–grape beverage mix.

The Peleg model seems to be the most suitable to describe the moisture sorption isotherm of Freeze dried whey grape beverage mix at 20 °C, 30 and 40 °C. GAB followed by BET equation also showed good fittings. The parameters C and K in the GAB equation can be correlated with temperature, using Arrhenius-type equations (Van den Berg and Bruin 1981). One isotherm is well fitted by GAB equation when K is close to the unity. The fittings at all temperatures studied present K value near to 1.

In overall evaluation, the good representation of the sorption data by the GAB model is not surprising because it is a semi-theoretical, multimolecular, localized, homogeneous adsorption model and has been suggested to be the most versatile sorption model available (Al-Muhtaseb et al. 2002). The constants of the GAB equation C and K are

related to the energies of interaction between the first and further adsorbed molecules at the individual adsorption sites. The Guggenheim constant (C) was also shown to decrease with increasing temperature, indicating the expected tendency of a decrease in binding energy for the first adsorbed layer with increasing temperature. Such a decrease indicates an increasingly shorter residence time for the adsorbed water molecules in the first layer, with the character of the adsorption process becoming less strongly localized. A clear correlation of the constant K with temperature was not observed. Van den Berg and Bruin (1981) stated that the parameters C and K incorporate the temperature effect. C is more enthalpic while K is more entropic in nature. Therefore, when describing practical isotherms over limited temperature intervals, it may suffice to incorporate the temperature effect only in C. A more detailed analysis of the GAB parameters can provide further valuable information about adsorption and desorption. The difference ($H_1 - H_m$) represents the difference between the heat of condensation of water and the heat of sorption of the multi-

Fig 3 The residual plots and the degree of fit for three selected models at 40 °C

layer, which is expected to have a positive value due to the exothermic nature of moisture sorption (Van den Berg and Bruin 1981). Lower magnitudes of this difference imply the presence of less firmly bound multilayer molecules, at intermediary energy levels between those of mono-layer molecules and the bulk liquid.

Modified Halsey and modified Henderson models suit also quite well to the moisture sorption isotherms even though they present a lower adjusted R-square and higher χ^2 . However the BET equation was fitted to three water activity values only since this model is valid for a_w below 0.5. The limited number of observations may explain this poor fitting. Since BET equation is reported to be one of the most suitable models to describe the sorption behaviour of starchy foods (Sopade and Ajisegeri 1994; Al-Muhtaseb et al. 2002), it seems reasonable that it doesn't suit to describe freeze dried whey–grape powder. However Arslan and Togrul (2000) found this equation also suitable for moisture isotherms of tea.

Heat of sorption

The study of sorption isotherms at least at two different temperatures provides thermodynamic data on isosteric heat of sorption through the use of Clausius–Clapeyron equation. The isosteric heat of sorption varies with the amount of water adsorbed by the substrate. The calculation of the variation of net isosteric heat of sorption as a function of moisture content, at the mean temperature of 30 °C, was done by Eq. (2). The relationship between isosteric heat of sorption and moisture content is plotted in Fig. 4. The heat of sorption is higher at low moisture contents than at higher moisture contents. At moisture content below 1 % (db) the isosteric heat of sorption increased sharply for freeze dried whey–grape beverage powder and value of 49.08 KJ/mol was estimated. However, at moisture content above that the isosteric heat of sorption fell almost in line with the heat of

Fig. 4 Variation of isosteric heat of sorption with moisture content for instant whey–grape beverage mix

vaporization of pure water. The decrease in the isosteric heat of sorption 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 occurs on the less active sites giving lower heats of sorption (Iglesias and Chirife 1982). The net isosteric heat of sorption ranged from 5.22 kJ/mol to 1.12 kJ/mol as moisture content varied between 1 and 9 % db for freeze dried whey–grape beverage.

Conclusion

The sorption isotherms of freeze dried whey–grape beverage mix were typical type II sigmoidal curves according to BET classification. An increase in temperature resulted in lower equilibrium moisture contents at corresponding values of water activity. GAB, BET, Modified Henderson, Modified Halsey, Oswin and Peleg models were applied to analyse the data. Nonlinear regression analysis was used for the determination of the parameters in the equations. Estimated parameters and fitting ability for sorption models were evaluated. The sorption data were in good agreement with models like Peleg and GAB. However, Peleg model showed best fitting to the sorption data of freeze dried whey–grape beverage mix at all temperatures studied. The isosteric heat of sorption varied between 5.22 and 1.12 KJ/mol at moisture level varying between 1 and 9 % db. The isosteric heat of sorption curve showed a fall with increase in moisture content and approached the heat of vaporization of free water at higher moisture content. The elevated values of heat of sorption of water at low moisture contents are an indication of strong water–food component interactions by the existence of polar groups on the surface of the product. Water sorption property of freeze dried powders is important in predicting the physical state of these hygroscopic materials under various conditions, because most structural transformations and phase transitions are significantly affected by water. The deliquescence of the freeze dried materials results from the dissolution of the particles by water adsorbed at the surface to form saturated solution at high RH. Hence a highly protective laminate Polyethylene/AlFoil/Paper is recommended for the packaging of freeze dried whey–grape beverage mix and also low RH (18–20 %) should be maintained during grinding and packaging of the material.

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