Beta-glucan rich composite flour biscuits: modelling of moisture sorption isotherms and determination of sorption heat

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Abstract Moisture adsorption isotherms of beta-glucan rich composite flour biscuits were determined at 28, 37 and 45 °C. Experimental data were fitted to 12 mathematical models. A nonlinear regression analysis method was used to evaluate the constants of the sorption equations. Statistical testing of sorption models was carried out using multiple criteria such as coefficient of determination (R^2), reduced chi-square (χ^2), mean relative percent deviation modulus (P) and plotting of residuals. BET ($R^2 > 0.99$; $\chi^2 < 0.09$; $P < 7.52$; $RMS\% < 9.22$) was found suitable for predicting the M_e - a_w relationship in the a_w range of 0.10–0.53. However, in the a_w range of 0.10–0.85, although Ferro-Fontan and GAB models were found to have high R^2 values (> 0.99), Peleg model was found to meet the multiple statistical criterion ($R^2 > 0.9996$; $\chi^2 < 0.04$; $P < 3.97$; $RMS\% < 7.09$). Properties of sorbed water were also determined. BET, GAB and Caurie monolayer moisture contents ranged from 2.64 to 3.36, 1.29–2.66 and 1.88–3.38 % d.b., respectively. Second-order regression equation was found to describe the relation between monolayer moisture content, M_o and temperature, t (°C). The isosteric heat, calculated using Clausius-Clapeyron equation, was varied between 1.46 and 50.39 kJ g⁻¹ mol⁻¹ at moisture levels 1–12 % (d.b.). An exponential relationship was observed between the isosteric heat of sorption and moisture content.

Keywords Composite flour · Beta-glucan · Biscuit · Sorption isotherm · Modelling

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Introduction

Among the food processing industries, bakery segment occupies an important position of which bread and biscuits form the major products. In India, bread and biscuits accounts for over 80 % of the total bakery products produced. Among the bakery products, growth rate of biscuits is about 15 % (More 2012) and the popularity of biscuits is due to its ready-to-eat, convenience nature, unique taste and ready availability at reasonable cost in different parts of the country, including remote rural areas. Most baked products, including biscuits, are essentially produced from refined wheat flour (*maida*) which contains energy dense carbohydrates such as starch and limits in specific health beneficial components. In recent years consumer awareness has led to revitalized interests for health promoting components that can be eaten as part of daily diet. These products have a special significance in a country like India where malnutrition and infectious diseases remain a silent emergency. Health concerns are attributed to poor nutrition in low-income segments of the population, whereas among the well-off strata it is primarily due to the changing life-style and food habits. Food-based nutrition is the most important way to fight hidden hunger, under nutrition and ill health (NAAS 2012). Composite flour is a combination of wheat and non-wheat flours prepared from mixtures of flours from cereals, roots, tubers, legumes, or other raw materials (Singh et al. 2011). Blending of non-wheat flours into baked products is in fact not a new practice (Dendy 1992). Mustard protein concentrate (Rajput et al. 1988), watermelon protein isolate (Wani et al. 2012), sorghum (Rao et al. 1984), mustard cake flour (Tyagi et al. 2007), fenugreek flour (Hooda and Jood 2005), soybean (Singh et al. 2000; Gandhi et al. 2001), skim milk powder (Rao et al. 1984), sodium caseinate (Gallagher et al. 2005), whey protein concentrate (Narain and Subashini 2004; Gallagher et al. 2005; Raju et al. 2007; Jisha and Padmaja 2011), low lactose dairy powders (Nunes

et al. 2009), soy protein isolate, oat bran and chickpea flour (Dhinda et al. 2012), oatmeal (McMinn et al. 2007), processed mango kernel flour (Arogba 2001), waxy barley starch (Ghiasi et al. 1984), etc. have been incorporated into wheat flour for making a variety of baked products with a view to utilize byproducts, enhance the health attributes, etc.

Barley is one of the important food grains since ancient times used especially for making alcoholic beverages and fermented foods and is recognized as an excellent source of soluble and insoluble dietary fibre and other bioactive constituents (Newman and Newman 2008). Jood and Kalra (2001) reported that the total beta-glucan content of hull less and hulled barley grown in India was in the range of 3.37–6.23 and 2.18–4.60 g/100 g (db). However, the industrial milled barley products such as break and reductin flours contain less beta-glucan (1.28–1.35 g/100 g (db)) (Kirylyuk et al. 2000). Beta-glucans, the major fibre constituents of barley, have been credited to lower plasma cholesterol, improve lipid metabolism and reduce glycemic index (Izydorczyk and Dexter 2008). With the growing interest in the development of functional foods to combat a variety of diet-related diseases, various functional ingredients are being incorporated into foods to bestow the health benefits. Barley-based foods are intrinsically qualified to be functional foods both for reducing cardiovascular disease risk and for modifying glycemic responses for treatment and prevention of diabetes (Lazaridou and Biliaderis 2007). Several baked and other food products such as yeast bread (Gujral et al. 2003; Dhingra and Jood 2004; Jacobs et al. 2008), unleavened flatbreads or *chapatis* (Gujral and Pathak 2002; Gujral and Gaur 2005), muffins (Newman et al. 1990), cakes and cookies (Ragaei and Abdel-Aal 2006), pasta (Cleary and Brennan 2006), weaning foods (Pedersen et al. 1989), etc. have been prepared from composite flours containing wheat, barley and other flours. With the growing popularity of multigrain and composite dairy foods, attempts have been made previously in our laboratory to develop beta-glucan-rich composite flour biscuits from a blend of barley, wheat, whey protein concentrate and skim milk powder.

The water sorption isotherms of foods show the equilibrium relationship between the moisture content of foods and the water activity at constant temperatures and pressures yielding the so-called moisture sorption isotherm (MSI) curves (Rizvi 2005). Temperature affects the mobility of water molecules and the dynamic equilibrium between the vapour and adsorbed phases. In general, if the water activity is maintained constant, an increase in temperature causes a decrease in the amount of sorbed water (Al-Muhtaseb et al. 2002). The knowledge and understanding of sorption isotherms is critical for the design of drying equipment, predictions of quality, stability and shelf life of food, design of packages and for calculating moisture changes that may occur during storage (Rao et al. 2006). MSI of most food products are nonlinear,

generally sigmoid in shape and have been classified as Type II isotherms (Al-Muhtaseb et al. 2002). Several attempts have been made to describe the sorption isotherms using mathematical models and validate the effectiveness of such models using statistical techniques (Basu et al. 2006). The quality of biscuits is deteriorated due to changes in organoleptic properties especially crispness, hardness, colour and flavour and by the growth of microbes, with these deteriorative changes being increasing with increasing water activity. Biscuits are a complex combination of ingredients, with both Type II and III isotherm forms reported in the literature. Earlier researchers have studied the MSI of baked products such as biscuits (Guillard et al. 2004), oatmeal biscuits (McMinn et al. 2007; Sampaio et al. 2009), cookies (Palou et al. 1997), biscuit containing processed mango kernel flour (Arogba 2001), crackers (Tubert and Iglesias 1986), Madeira cake (Al-Muhtaseb et al. 2010), flat bread (Marzec and Lewicki 2006), ginger bread (Cervenka et al. 2008) and composite foods (Kim et al. 1998, 1999).

The underlying objective of the present study was to determine the MSI of beta-glucan-rich biscuits obtained from composite flour containing wheat and barley flours and milk solids at different temperatures (28–45 °C) and relative humidities (10–85 %). The MSI values were fitted to different two and three parameter mathematical models and their temperature dependence was evaluated using the temperature dependent Guggenheim-Anderson-de Boer (GAB) equation parameters and the Clausius-Clapeyron equation.

Materials and methods

Preparation of beta-glucan rich biscuits

Beta-glucan rich biscuits were prepared by following the method described by Raju et al. (2007) with suitable modifications. The standardized formulation consisted of barley flour 70 g, refined wheat flour 30 g, bakery shortening 35 g, whey protein concentrate (WPC) (70 g protein on dry basis) 4 g, non fat dry milk 2 g, sugar 45 g, salt 1 g, sodium bicarbonate 1 g, ammonium bicarbonate 0.85 g, lecithin 0.5 g, vanilla essence 4 mL and water 25 mL. Barley, refined wheat flour (*maida*), sugar and bakery shortening (Amrit Vanaspati Ltd., Rajpura, Punjab.) were obtained from local market. WPC and non fat dry milk (skimmed milk powder) were procured from M/s Modern Dairies Ltd., Karnal. Sodium bicarbonate, ammonium bicarbonate were obtained from M/s Sisco Research Laboratories Pvt. Ltd., Mumbai. Bakery shortening and grounded sugar were made into cream in a Hobart mixer. Barley flour, wheat flour and baking powder were sifted and added to the above mixture and mixed till a crumbly texture was obtained. Measured quantity of water was divided into two equal parts. In one part, salt was dissolved and in the

other ammonium bicarbonate was dissolved. Both were then added to the above mixture and kneaded to smooth homogeneous mass along with vanilla essence. The dough was rolled out into thin sheets of 3–5 mm thickness by means of a wooden roller. The sheets were cut into circular shape by means of a biscuit-cutter mould. The pieces were placed over perforated metallic trays of rotary rack baking oven in single layers and baked at 180 ± 10 °C for 10 min. The baked biscuits were removed from the trays and cooled to room temperature. Control biscuits were prepared similarly as described above. However, refined wheat flour (100 g) alone was used in the formulation.

Preparation of sample

Biscuits were ground using a pestle and mortar and mixed with small amounts of potassium sorbate (0.5 g/100 g biscuit) to prevent microbial growth during sorption studies. The moisture content of beta-glucan rich biscuits was about 2.3 g/100 g solids. With a view to ensure a complete adsorption isotherm, samples were dehydrated by placing them over phosphorous pentoxide in a desiccator, which gives a relative humidity environment close to 0 %, for 7 days at the desired isotherm temperature (Sawhney et al. 2011).

Chemical analysis

Chemical composition of biscuits in terms of moisture, fat, protein, crude fibre and total ash was determined using standard methods (IS 12711 1989; AOAC 1984) while beta-glucan was estimated by using a kit (Megazyme International, Ireland). The carbohydrate content was determined by calculating the difference (Table 1).

Measurement of sorption equilibrium

Adsorption isotherms of beta-glucan-rich biscuits prepared from composite flour were determined at 28°, 37° and 45 °C

over a water activity range of 0.11–0.85 using static moisture gain or loss from test samples. The standard sorption apparatus recommended by COST 90 Project and adopted by many workers (Kumar et al. 2005; Rao et al. 2006; Sawhney et al. 2011) was used in determining the sorption equilibrium of biscuits. The sorption device consists of a sorption container and support for weighing beakers in which the samples were exposed to humid atmospheres in the containers. Seven reagent grade salts, viz. Lithium chloride, potassium acetate, magnesium chloride, potassium carbonate, magnesium nitrate, sodium chloride and potassium chloride were chosen to cover the water activity range of 0.11–0.85 (Table 2). The salts were dissolved in distilled water at 100 °C and cooled to test temperatures of 28°, 37° and 45 °C for crystallization to form saturated solutions. The sorption apparatus were placed in incubators maintained at the temperatures of 28°, 37° and 45 °C and allowed to equilibrate at respective temperatures for 3 days before samples were placed in them.

Approximately 1.000 ± 0.005 g of beta-glucan rich biscuit sample was taken in each of the sorption container at respective relative humidity and temperature in triplicate making 63 samples in total. The samples were weighed at regular interval of 2 days and the equilibrium was considered to have reached when the weight differences of two successive measurements showed less than 0.001 g. The equilibrium time, however, was different for different a_w . It was about 15–19 and 23–27 days in the a_w range of 0.10–0.30 and 0.40–0.85, respectively. The moisture content was determined by gravimetric method. The average value of the triplicates was determined and expressed as equilibrium moisture content (g water / 100 g solids). The equilibrium moisture contents were plotted against water activities to obtain moisture sorption isotherm.

Modelling of sorption isotherms

A number of mathematical equations have been used for describing the sorption behaviour of different food products (Labuza and Altunakar 2007; Andrade et al. 2011). However,

Table 1 Proximate composition of beta-glucan rich biscuit made from composite flour and control biscuits

Proximates	Contents (g/100 g)	
	Beta-glucan rich biscuits	Control biscuits
Moisture	2.31	2.70
Fat	19.61	20.50
Carbohydrates	66.43	70.00
Protein	10.97	5.20
Ash	1.06	1.10
Crude fibre	1.93	0.95
Beta-glucan	1.48	–

Table 2 Salts and their corresponding water activity (a_w) at different temperatures

Salts	Temperature (°C)		
	28	37	45
Lithium chloride	0.112	0.107	0.103
Potassium acetate	0.230	0.212	0.198
Magnesium chloride	0.326	0.316	0.308
Potassium carbonate	0.441	0.435	0.430
Magnesium nitrate	0.530	0.512	0.497
Sodium chloride	0.756	0.740	0.726
Potassium chloride	0.845	0.815	0.791

in many cases the model suitable for certain food product is not suitable for the other. Hence, there is an obvious need to study more number of mathematical models for describing the sorption behaviour of formulated foods especially with multiple ingredients such as the beta-glucan rich composite flour biscuits. As per the literature, models such as GAB, BET, Oswin, Ferro-Fontan, Peleg, Henderson and Caurie were found to best describe the sorption behaviour of bakery and composite food products. Hence, in the present study, 12 mathematical models (7 two parameter, 4 three parameter and 1 four parameter) were used to fit the experimental data on moisture sorption. Model with relevant isotherm equations are shown in Table 3. The constants of the sorption models were estimated using nonlinear regression function of OriginPro 8 software.

Statistical analysis

Different methods are available to select the sorption model best fitting the experimental data. However, no single statistical parameter can be used to select the best model instead the model must always be assessed based on multiple statistical criteria (Sopade 2001; Basu et al. 2006). In the present study, the suitability of best fit model in predicting the sorption behaviour was validated on the basis of coefficient of determination (R^2), reduced chi-square (χ^2), mean relative percent deviation modulus (P), root mean square percent error ($RMS\%$) and residuals. The models with high values of R^2

(>0.95), lower values of χ^2 (<0.5), P (<5) and $RMS\%$ (<10) and irregular pattern of residuals plot are considered as best fit. The coefficient of determination, reduced chi-square, mean relative percent deviation modulus and root mean square percent error were calculated by Eqs. 1, 2, 3 and 4, respectively.

$$R^2 = \frac{\sum_{i=1}^n (M_{pre} - \bar{M})^2}{\sum_{i=1}^n (M_{exp} - \bar{M})^2} \quad (1)$$

$$\chi^2 = \frac{1}{N-n} \sum_{i=1}^N (M_{exp} - M_{pre})^2 \quad (2)$$

$$P = \frac{100}{n} \sum_{i=1}^n \left| \frac{M_{iexp} - M_{ipre}}{M_{iexp}} \right| \quad (3)$$

$$RMS\% = \sqrt{\frac{1}{n} \left[\sum_{i=1}^n \left(\frac{M_{exp} - M_{pre}}{M_{exp}} \right)^2 \right]} \times 100 \quad (4)$$

Table 3 Moisture sorption models for fitting experimental data

Name of the model	Equation	Reference
Two-parameter models		
BET	$\frac{M}{M_0} = \frac{C_b a_w}{(1-a_w)} (1-a_w + C_b a_w)$	Brunauer et al. (1938)
Oswin	$M = A \left[\frac{a_w}{1-a_w} \right]^B$	Oswin (1946)
Lewicki-I	$M = A \left[\frac{1}{a_w} - 1 \right]^{B-1}$	Lewicki and Wolf (1995)
Mizrahi	$M = \left[\frac{B a_w - A}{1-a_w} \right]$	Mizrahi et al. (1970)
Kuhn	$M = \left[\frac{A}{Ln(a_w)} \right] + B$	Kuhn (1964)
Caurie	$\frac{1}{M} = \frac{1}{C_c M_0} \left(\frac{1-a_w}{a_w} \right)^{\left(\frac{2C_c}{M_0} \right)}$	Caurie (1981)
Chung & Pfost	$Ln(a_w) = -Ae^{(-BM)}$	Chirife and Iglesias (1978)
Three-parameter models		
Lewicki-II	$M = A \left[\frac{1}{(1-a_w)^B} - \frac{1}{(1+a_w^C)} \right]$	Lewicki (1998)
Ferro-Fontan	$M = \left[\frac{B}{Ln\left(\frac{A}{a_w}\right)} \right]^C$	Ferro-Fontan et al. 1982
GAB	$M = \frac{M_0 C K a_w}{(1-K a_w)} (1-K a_w + C K a_w)$	Van-den-Berg and Bruin (1981)
Modified Mizrahi	$M = \frac{A + B a_w + C a_w^2}{(a_w - 1)}$	Mizrahi and Karel (1977)
Four-parameter model		
Peleg	$M = A(a_w)^{C_1} + B(a_w)^{C_2}$	Peleg (1993)

Determination of properties of sorbed water

Sorption properties in terms of absorbed monolayers (N), surface area of absorbent (A_s), density of absorbed water (d) and percent bound water were determined (Kumar et al. 2005). The Caurie's slope (S) was used for deriving the number of adsorbed monolayers.

$$S = \frac{2}{N} \quad (5)$$

C_c in the Caurie's equation represents density of bound water while the percent bound water or non-freezable water is obtained by multiplying BET monolayer value M_o in the equation and N . The surface area of adsorption was determined by the formula given below:

$$A = \frac{54.54}{S} \quad (6)$$

Determination of thermodynamical properties

The net isosteric heat of sorption provides an estimation of the heat required to be removed in excess of latent heat of vaporization of pure water, for extracting bound water at particular moisture content. The net isosteric heats of sorption q_{st} (KJ/kg) were calculated from the following Clausius-Clapeyron equation applied at two temperatures, T_1 and T_2 (Kumar et al. 2005).

$$q_{st} = R \left[\frac{T_1 T_2}{T_1 - T_2} \right] \left[\ln \frac{a_{w2}}{a_{w1}} \right] \quad (7)$$

where a_{w1} and a_{w2} are water activities at absolute temperatures T_1 and T_2 , respectively and R represents the gas constant (8.314 J/mol K).

Results and discussion

Moisture adsorption isotherms

The experimental equilibrium moisture content of control and beta glucan-rich biscuits in the a_w range of 0.11–0.85 is given in Fig. 1a and b respectively. The equilibrium moisture content at each water activity represents the mean value of three replications. It can be seen from the Fig. 1 that the equilibrium moisture content of beta glucan-rich biscuits increased

Fig. 1 Moisture adsorption isotherm of (a) control and (b) beta glucan-rich biscuits at various temperatures

exponentially with increasing water activity, following the form of a Type III isotherm according to the BET classification (Rizvi 2005). Foods rich in soluble components, such as sugars, have been found to show Type III behaviour, which is due to the solubility of sugars in water (Al-Muhtaseb et al. 2002). At water activities less than 0.70, a gradual increase in equilibrium moisture content with increasing water activity was observed. This reflected water sorption by the starch-based components of the beta glucan-rich biscuits made from composite flour mixture. At high water activities ($a_w > 0.70$), however, the sugar component of the biscuit rendered the most significant influence on the sorption behaviour, resulting in a high equilibrium water uptake. Ertugay and Certel (2000) observed that the sorption isotherm curve of wheat, barley, rye, oat and corn resembled the characteristics of Type II isotherm with multilayer adsorption. Type III curve is a characteristic of high-sugar containing foods which sorb relatively small amount of water at low water activities and large amounts at high water activities particularly above 0.60 (Cervenka et al. 2008). The water vapour adsorption isotherms of cereal flours such as hard and soft wheat flours

and rice flour represents a classical sigmoidal shape (Roman-Gutierrez et al. 2002; Durakova and Menkov 2004). The water vapour adsorption properties of wheat flour depend on the ability of flour components to establish direct interactions with the water molecules. Starch is the main component involved in the hydration properties of wheat flour. The contribution of the soluble and insoluble pentosans in water vapour adsorption is low even though they have highest water adsorption capacities (Roman-Gutierrez et al. 2002). However, the sorption behaviour of malted cereals is different compared to their nonmalted counterparts. Barrerio et al. (2003) reported that the equilibrium moisture content of barley malt steeply increases above 0.5 a_w which has been attributed to higher amounts of simple sugars and enzyme activity. The moisture sorption behaviour of dairy ingredients such as SMP, WPC and whey protein isolate was reported to be of classical sigmoid-shaped curve reflecting a Type II isotherm (Foster et al. 2005; Ko et al. 2008; Sawhney et al. 2013). WPC powder sorbed relatively lower water up to the a_w of 0.35. The equilibrium moisture content rose gradually between 0.35 and 0.80 a_w and larger amount of water was sorbed at a_w above 0.80 (Sawhney et al. 2013). In the regions of lower a_w , milk proteins are known to be the preferred sorption sites and at higher a_w the effect of protein is counteracted by lactose because its equilibrium moisture content increases at higher temperatures and higher a_w . At higher a_w , SMP sorbs more water as it contains more lactose content.

In the present study, the sorption behaviour of control and beta-glucan rich composite flour biscuit was found to be similar to other composite flour biscuits available in the literature with starch and sugars contributing to the greatest extent and milk proteins to the minimum. However, it can be seen that compared to beta-glucan rich biscuits, the equilibrium moisture content of control biscuits was found to be less at all a_w . Cadden (1988) reported that among several food fibers, guar gum adsorbed the most water at 4, 25 and 37 °C. Further, it was reported that the adsorption abilities of oat bran and wheat bran were similar at low and intermediate a_w . However, wheat bran was able to adsorb more moisture in the high a_w regions of the isotherm. In the present study, the higher moisture adsorption by beta-glucan rich biscuits could be attributed to higher crude fiber compared to control biscuits (Table 1). The reason for the shape of isotherm (Type III) of beta-glucan rich biscuit could be due to the presence of high amount of sugar in the product as evidenced from the proximate composition (Table 1). McMinn et al. (2007) reported that the sorption curve of microwave-baked biscuit resembled Type III isotherm with an equilibrium moisture content of 6.75 g/100 g (dry basis, db) (at 20 °C) at 0.75 a_w which increased in magnitude to 26.5 g/100 g (db) at 0.96 a_w . Guillard et al. (2004) reported that dry biscuit exhibited Type III behaviour, with an uptake of moisture of 10 g/100 g (wet basis, wb) at 0.70 a_w increasing significantly above this value to 34 g/100 g (wb) at 0.97 a_w . Similar Type III curve was

observed by Sampaio et al. (2009) in a composite flour biscuits made from wheat, oatmeal and passion fruits flour. It was reported that the equilibrium moisture content increased steeply above 0.70 a_w . Palou et al. (1997) reported that cookies exhibited sigmoid shape at 25 °C, which is described as Type II isotherm as per the classification. Similar results of exhibition of Type II isotherm were reported by Arogba (2001) in case of processed mango kernel containing biscuits. The moisture sorption behaviour of the beta glucan biscuits was temperature dependent, as indicated by a decrease in equilibrium moisture content, at a given a_w , with increasing temperature. This was indicative of a lowering of the hygroscopic nature of the biscuit. This trend is generally ascribed to a reduction in the number of active sites for water binding due to chemical and physical changes induced by temperature e.g. gelatinization of starch and denaturation of protein (Erbaş et al. 2005). The increased temperature may also have led to activation of the water molecules to higher energy levels, which caused them to become less stable and break away from the water-binding sites of the food, thus decreasing the equilibrium moisture content (Palipane and Driscoll 1992).

Sorption models and associated parameters

The parameters of the 12 sorption models applied in the study have been determined and presented in Table 4. Examination of the results indicated that, the BET model gave the best fit to the experimental adsorption data in the water activity range of 0.10–0.53. At different temperatures, the statistical comparison of the observed and predicted results revealed that the coefficient of determination was high ($R^2 > 0.9942$) and reduced chi-square ($\chi^2 < 0.098$), percent mean deviation error ($P < 7.52$), root mean square error percent ($RMS\% < 9.22$) were low. The BET constant related to the net heat of sorption C_b , was found to be 6.24, 2.85 and 2.75 at 28, 37 and 45 °C, respectively. Arogba (2001) reported that the C_b values of processed mango kernel flour fortified biscuits as 18.51 and 23.52 at 31 and 58 °C. McMinn et al. (2007) reported the C_b values of convective-baked oatmeal biscuits as 8.26 and 11.1 at 20 and 40 °C. In the entire a_w range (0.10–0.85), Peleg model gave the closest fit to the experimental data. The statistical analysis revealed R^2 values ranging from 0.9997 to 0.9998, χ^2 ranging from 0.01 to 0.04, P values ranging from 2.11 to 3.97, and $RMS\%$ values ranging from 2.73 to 7.09. Next best models to describe the experimental data were Ferro-Fontan and GAB models. The goodness-of-fit parameters for the Ferro-Fontan and GAB models indicated slightly lower R^2 values and slightly higher χ^2 values compared to Peleg model. R^2 values of Ferro-Fontan model were 0.9958, 0.9962 and 0.9958 while for GAB model they were 0.9943, 0.9954 and 0.9920 at 28°, 37° and 45 °C, respectively. The average χ^2 value of Ferro-Fontan and GAB models was found to be 0.28 and 0.40, respectively. However, the goodness-of-fit was poor for Ferro-Fontan and

Table 4 Estimated values of parameters of sorption models at various temperatures

Model	Parameter	28 °C	37 °C	45 °C
Two parameter models				
BET ($a_w=0.10\text{--}0.53$)	M_o	3.36	3.04	2.64
	C	6.24	2.85	2.75
	R^2	0.9942	0.9969	0.9968
	χ^2	0.10	0.04	0.03
	P	7.22	6.57	7.52
	$RMS\%$	9.22	7.07	5.06
Oswin ($a_w=0.10\text{--}0.85$)	A	5.41	3.64	2.62
	B	0.95	1.22	1.50
	R^2	0.9688	0.9602	0.9337
	χ^2	2.78	2.55	3.28
	P	23.27	34.38	49.12
	$RMS\%$	27.54	39.98	55.23
Lewicki-I ($a_w=0.10\text{--}0.85$)	A	5.41	3.65	2.64
	B	0.05	−0.22	−0.49
	R^2	0.9688	0.9602	0.9337
	χ^2	2.78	2.55	3.28
	P	23.25	34.28	48.88
	$RMS\%$	27.52	39.87	54.95
Mizrahi ($a_w=0.10\text{--}0.85$)	A	−0.98	0.18	0.45
	B	3.78	5.06	5.29
	R^2	0.9760	0.9489	0.9055
	χ^2	2.13	3.28	4.67
	P	10.92	22.76	32.79
	$RMS\%$	12.49	26.33	41.35
Kuhn ($a_w=0.10\text{--}0.85$)	A	−4.65	−4.74	−4.67
	B	−0.819	−1.966	−2.188
	R^2	0.9745	0.9449	0.8999
	χ^2	2.27	3.54	4.96
	P	10.95	27.68	40.14
	$RMS\%$	12.24	36.27	54.48
Caurie ($a_w=0.10\text{--}0.85$)	M_o	3.38	2.45	1.88
	C_c	1.60	1.49	1.40
	R^2	0.9688	0.9602	0.9337
	χ^2	2.78	2.55	3.28
	P	23.21	34.24	48.93
	$RMS\%$	27.48	39.83	55.02
Chung & Pfof ($a_w=0.10\text{--}0.85$)	A	1.77	1.69	1.71
	B	0.105	0.119	0.134
	R^2	0.8083	0.7646	0.7203
	χ^2	17.05	15.11	13.84
	P	61.69	94.89	111.45
	$RMS\%$	96.46	157.45	183.75
Three parameter models				
Lewicki-II ($a_w=0.10\text{--}0.85$)	A	1.66	1.01	0.61
	B	1.51	1.86	2.22
	C	−201.98	−3.99	−3.81
	R^2	0.9831	0.9799	0.9566
	χ^2	1.51	1.29	2.15

Table 4 (continued)

Model	Parameter	28 °C	37 °C	45 °C
Ferro-Fontan ($a_w=0.10-0.85$)	P	17.47	20.86	27.08
	$RMS\%$	21.23	27.74	29.08
	A	0.90	0.86	0.82
	B	6.45	3.42	2.93
	C	1.41	1.33	1.49
	R^2	0.9958	0.9962	0.9958
	χ^2	0.37	0.25	0.21
GAB ($a_w=0.10-0.85$)	P	13.36	19.44	24.41
	$RMS\%$	22.56	34.52	45.66
	K	0.9306	0.8821	0.8431
	C	16.188	14.016	38.287
	M_o	2.66	1.87	1.29
	R^2	0.9943	0.9954	0.9920
	χ^2	0.51	0.30	0.40
Modified Mizrahi ($a_w=0.10-0.85$)	P	11.88	16.60	24.52
	$RMS\%$	16.40	24.94	39.09
	A	-2.26	-1.95	-2.05
	B	1.82	4.80	6.94
	C	-4.93	-9.12	-11.74
	R^2	0.9752	0.9554	0.9151
	χ^2	2.20	2.86	4.20
Four parameter model Peleg ($a_w=0.10-0.85$)	P	19.46	32.36	46.04
	$RMS\%$	28.24	49.88	72.75
	A	11.94	359.61	7.63
	B	186.57	9.48	1037.08
	C_1	0.99	15.24	1.06
	C_2	13.80	1.11	18.19
	R^2	0.9997	0.9998	0.9998
	χ^2	0.03	0.01	0.04
	P	2.57	2.11	3.97
	$RMS\%$	3.29	2.73	7.09

GAB models in terms of P and $RMS\%$. Sampaio et al. (2009) reported that GAB model was found to be most suitable for describing the adsorption characteristics of composite flour biscuit containing wheat, oatmeal and passion fruit flour. Palou et al. (1997) reported that Peleg and GAB equations

adequately predicted the isotherms of cookies with mean relative deviation less than 7 %. Henderson equation predicted the isotherms of processed mango kernel flour incorporated biscuits better than BET with mean relative deviations less than 5 % (Arogba 2001). However, McMinn et al. (2007) reported

Table 5 Properties of sorbed water in beta glucan-rich biscuit

Temp	BET M_o	GAB M_o	Caurie M_o	Caurie's Slope	No. of adsorbed monolayers	Density of sorbed water	Bound water or non-freezable water	Surface area of sorption
°C	g/100 g db			S	N	g/cc	%	m ² /g
28	3.36	2.66	3.38	0.6810	2.94	1.144	9.87	80.09
37	3.04	1.87	2.45	0.9973	2.01	1.516	6.10	54.68
45	2.64	1.29	1.88	1.0835	1.85	1.430	4.87	50.34

◀ **Fig. 2** Experimental and predicted equilibrium moisture content with residuals (**a**, **b** and **c** indicate temperatures (28, 37 and 45 °C) and 1 and 2 indicate models (GAB and Peleg))

that Ferro-Fontan model provided the best description of sorption data in the water activity range of 0.03–0.96 followed by GAB model for convective and microoven-baked oatmeal biscuits. Also, Guillard et al. (2004) reported that Ferro-Fontan model adequately described the experimental data of the biscuit in the a_w range of 0 to 0.97 at 20 °C. In the present study, the sorption isotherm of beta-glucan rich biscuits were more accurately predicted by Peleg model than GAB model at all temperatures as can be evidenced from the highest R^2 , lowest χ^2 , P and %RMS (Tables 4 and 5) and random pattern of residuals (Fig. 2). This could be due to an extra parameter or constant in the Peleg model (Andrade et al. 2011; Palou et al. 1997; Peleg 1993). Among all the models, Chung & Pfoest model provided the worst representation of the data, with the R^2 values ranging from 0.7203 to 0.8083, χ^2 values ranging from 13.838 to 17.049, P values ranging from 61.69 to 111.45 and RMS% ranging from 96.46 to 183.75.

Properties of sorbed water

The sorption data indicates that as the temperature of sorption increased from 28 to 45 °C, the percent bound water, number of adsorbed monolayers and surface area of adsorbent decreased. The monolayer moisture (M_o) as obtained from BET, GAB and Caurie's models for 28 °C was observed to be as 3.36, 2.66 and 3.38 g/100 g solids, respectively. The corresponding values at 37 °C were determined as 3.04, 1.87, 2.45 g/100 g while at 1.29–2.66 g/100 g solids while for 45 °C were 2.64, 1.29 and 1.88 g/100 g. In general, for any temperature, M_o calculated using GAB model was found to be lower compared to BET and Caurie models. With the increase in temperature from 28 to 45 °C, the bound water content and surface area of sorption decreased from 80.09 to 50.34 % and 80.09 to 50.34 m²/g. Sampaio et al. (2009) reported that GAB M_o of composite flour biscuits containing wheat, oatmeal and passion fruit flour was 6.08 and 2.43 kg/kg at 25 and 40 °C. Palou et al. (1997) reported that GAB equation gave significantly higher M_o values for different commercial cookies and was in the range of 3.91 to 4.58 g/100 g (db). Kim et al. (1998) reported that the GAB M_o of composite food consisting of cookie/strawberry jam/cookie decreased from 4.5 to 3.4 g/100 g (db) with increase in temperature from 20 to 40 °C. Similar trend was reported by Kim et al. (1999) for composite food consisting of cookie/chocolate/cookie. Second-order regression of monolayer moisture content, M_o (% db) due to temperature, t (°C) were fitted for BET, GAB and

Caurie models (Eqs. 8, 9 and 10).

$$BET M_o = -0.0008t^2 + 0.0197t + 3.4753 \quad (R^2 = 1.0) \quad (8)$$

$$GAB M_o = 0.0009t^2 - 0.1462t + 6.0488 \quad (R^2 = 1.0) \quad (9)$$

$$Caurie M_o = 0.0019t^2 - 0.226t + 8.2285 \quad (R^2 = 1.0) \quad (10)$$

Isosteric heat of sorption

The moisture contents in the temperature range of 28–45 °C were obtained by interpolating the experimental data using GAB equation (Table 6). The sorption isosters (Fig. 3) did not show definite temperature dependence over the entire temperature range of 28–45 °C at all moisture contents (1–8 g/100 g solids). The net isosteric sorption heat of beta glucan-rich biscuit as a function of moisture content is given in Fig. 4. The curve shows that the heat of sorption decrease with increase in amount of water sorbed. It can be observed that the maximum heat of adsorption (93.79 kJ/mol) was obtained between a moisture content of 1–2 % on dry basis. However, between 2 and 5 % moisture, the isosteric heat of sorption decreased sharply and thereafter it was in line. The increase in isosteric heat at low moisture content could be due to swelling of the food polymers resulting in the exposure of sorption sites of high binding energies not previously available (Palou et al.

Table 6 Equilibrium water activity of biscuits at different moisture contents

Moisture content (g/100 g db)	Temperature (°C)		
	28 °C	37 °C	45 °C
1	0.036	0.074	0.072
2	0.116	0.242	0.337
3	0.250	0.405	0.496
4	0.379	0.511	0.581
5	0.475	0.580	0.632
6	0.544	0.628	0.667
7	0.596	0.663	0.692
8	0.636	0.689	0.711
9	0.668	0.710	0.725
10	0.693	0.727	0.737
12	0.732	0.752	0.755
14	0.760	0.771	0.767
16	0.781	0.784	0.777
18	0.797	0.759	0.784
20	0.810	0.804	0.790

Fig. 3 Sorption isosters of beta glucan-rich biscuit at different moisture contents

1997). The decrease in the isosteric heat of sorption with increase in 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 (Rao et al. 2006). As these active sites become occupied, sorption subsequently occurs on the less active sites viz. peptide binds or hydrophobic hydration sites giving lower heats of sorption (Kumar et al. 2005). The presence of fat and gelatinization of starch during baking process may have a lowering effect of the binding energy of sorbed water (Aviara and Ajibola 2002). Hence, the water-binding in beta glucan-rich biscuits is high at the product's moisture content of 2.31 %. The maximum net isosteric heats (Q_{st}) found were higher than those reported by Palou et al. (1997) for cookies (6–10 kJ/mol); by Kim et al. (1998) for cookies (~25 kJ/mol) and crackers (~42 kJ/mol); by Arogba (2001) for processed mango kernel flour containing biscuits (18.2 kJ/mol); and by McMinn et al. (2007) for convective-baked (10.8 kJ/mol) and

Fig. 4 Netisosteric heat of sorption of beta glucan-rich biscuit at different moisture contents

microwave-baked (16.1 kJ/mol) oatmeal biscuits. The comparatively higher values of beta glucan-rich biscuits could be attributed to less damage to the active sorption sites during processing and baking. An exponential equation was used to describe the relationship between the isosteric heat of sorption and moisture content (Eq. 11).

$$q_{st} = 84.857e^{-0.344M} \quad R^2 = 0.9926 \quad (11)$$

The high value of the coefficient of determination shows that exponential equation can be used to calculate the heat of sorption of beta-glucan rich biscuits for varying moisture content.

Conclusion

Results of the present study have shown that the moisture adsorption behaviour of beta-glucan rich biscuits exhibited Type III sorption characteristics. Equilibrium moisture content, at a given water activity, decreased with increasing temperature. Experimental adsorption isotherm data within the temperature range of 28–45 °C and water activity range of 0.10–0.53 were best described by BET. GAB, Ferro-Fontan and Peleg models best described the data within the water activity range of 0.10–0.85. However, only Peleg model could meet the multiple statistical evaluation criterion and emerged as the best model to describe the sorption behaviour of beta-glucan rich biscuits made from composite flour. The relation between monolayer moisture content, M_o obtained by BET, GAB and Caurie models and temperature, t (°C) was best described a second-order regression equation. The net isosteric heats of sorption for beta-glucan rich biscuits, calculated using Clausius-Clapeyron equation, showed an exponential relationship with moisture content. The results obtained from this study would be useful in designing suitable package and predict the shelf life of beta-glucan rich biscuits under different conditions.

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