International Food Research Journal 16: 561-569 (2009)

Desorption and adsorption characteristics of bael (*Aegle marmelos)* pulp and powder

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Abstract: Moisture desorption isotherm of bael (*Aegle marmelos)* pulp and adsorption isotherm of pulp powder were determined at 20, 30, 40 and 50 °C. Static gravimetric method was used by exposing the samples to controlled atmospheres maintained by saturated salt solutions. The isotherms were found to be of type II sigmoid. Experimental data were fitted to four isotherm models viz. Anderson, Guggenheim–Anderson–de Boer (GAB), Oswin and Peleg models. It was found that Peleg model fits best describing equilibrium moisture content and equilibrium relative humidity (EMC–ERH) relationships for both the bael pulp and the powder. The isosteric heat of sorption varied between 47.5 - 44.55 kJ g^{-1} mol^{-1} at moisture levels 0.5 - 3.5 g/g dry matter for bael pulp and 46.12 - 44.40 kJ g^{-1} mol^{-1} at moisture level between 0.25 - 1.125 g/g dry matter for powder.

Keywords: sorption isotherms, bael pulp, equilibrium moisture content, water activity, isosteric heat of sorption

Introduction

Bael (*Aegle marmelos)* is one of the most useful medicinal fruits of India. The common name of bael is stone apple. The tree grows wild in dry forests on hills and plains of India, Burma, Pakistan and Bangladesh; also in mixed deciduous and dry dipterocarp forests of former French Indochina. It contains many vitamins like vitamin C, vitamin A, thiamine, riboflavin, niacin, and minerals like calcium, and phosphorus (Dikshit and Dutt, 1930; Parmar and Kaushal, 1982; Roy and Khurdiya, 1995). There are innumerable references of its uses in traditional medicine (Arseculeratne *et al*., 1981; Karunanayake *et al.*, 1984; Singh, 1986; Nagaraju and Rao, 1990). The pulp contains psoralen, marmelosin and many other polyphenolic compounds. The psoralen in the pulp increases skins tolerance to sunlight and aids in the maintaining of normal skin colour. It is employed in the treatment of leucoderma. Marmelosin derived from the pulp is used as a laxative and diuretic treatment. The ripe fruit is aromatic, astringent and also have good coolant effect. The pulp is often processed as nectar or 'squash' (diluted nectar). A popular drink (called 'sherbet' in India) is made by beating the pulp together with milk/water and sugar. Due to the aroma and health benefit, the fruit can be exploited. It is very important to investigate the possibility of using various health products from this fruit. Knowledge of water activity a_w is one of the useful measurements to decide the stability of foods, select formulations and storage conditions in new products and to improve drying process and equipments (Aroldo *et al*., 2006). In this regard, equilibrium sorption data is particularly useful to design and optimization of drying process and determine the storage condition. A number of equations allow the moisture content to be related to water activity (Vazquez *et al*., 2001; Vullioud, *et al*., 2004).

Since bael is a seasonal fruit its availability is restricted through out the year. Drying is one of the most economical methods for preservation of bael fruit pulp for longer time. Accurate information on equilibrium moisture contents of bael pulp and its powder at various relative humidity and temperatures are not available. There is also a need for comprehensive study of the equilibrium moisture contents of bael fruits pulp and powder to understand its drying and storage behavior. The present paper deals with desorption isotherms for bael pulp and adsorption of its powder determined under different temperatures and relative humidity levels, the most suitable model describing the isotherms and the net isosteric heat of sorption.

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Materials and Methods

Raw material

Fresh bael (*Aegle marmelos)* fruit was purchased from local market at Kharagpur, West Bengal, India. Fully ripen bael fruits (Figure 1) were chosen for this study. The fruits were washed, graded and their hard rind was broken manually. Pulp was scooped out along with seeds, gums and mixed with equal amount of (w/w) water and citric acid at the rate of 5g per kg of pulp. The mixture was passed through a nylon sieve (20 mesh) to remove seeds and fibre and its concentration was adjusted to (18 °Brix) by addition of requisite amount of water. The pulp was collected in glass bottles and sterilized by placing the crown capped bottles in boiling water bath for 15 min. and stored at ambient condition for further studies. The same pulp with 18 °Brix was freeze dried to obtain bael pulp powder. Eighty grams of pulp sample taken in circular tray (22 cm diameter) was frozen by putting the sample in deep freezer over night. The frozen sample was dried in a LYODEL (Delvac, India) freeze drier. It takes about 10 hr for drying of 80 g of 2 mm thick sample. After drying the dried flex collected and kept in desecrator and then ground in a mixer-grinder (Kenstar, India). The ground powder was sieved in 500 μ S.S. sieve. The moisture content and the proximate analysis of pulp were determined by standard procedure (AOAC 1990). It contained 80.82 g of water, 1.26 g of protein, 0.23 g of fat, 0.87 g of minerals 16.82 g of carbohydrate per 100 g. The moisture content of freeze dried powder was 3.48 percent wet basis. The bael pulp and its freeze-dried powder were used for sorption studies.

Experimental method

Procedure adopted by Iglesias and Chirife (1982) was followed for deriving desorption and adsorption isotherms of both the products at 20, 30, 40 and 50 °C. Approximately one to two grams samples of pulp and pulp powders filled in sterilized glass weighing bottles were placed in six separate vacuum desiccators containing saturated salt solutions (LiCl, $MgCl_2$, $Mg(NO_3)_2$, NaCl, KCl and KNO_3) for maintaining RH levels from 10 to 95% (Young, 1967; Greenspan,1977; Palipane and Driscoll, 1992). The air inside the desiccators was sucked partially to maintain a partial vacuum with the help of a vacuum pump.

All six desiccators were kept in an incubator thermostatically controlled at 20 °C and the gain or loss in weights of all the samples in each desiccator were taken at two days interval until the sample attained EMC. The attainment of EMC was ascertained when three consecutive weight measurements showed a difference of less than 0.001g. Depending upon the nature of the samples and the temperature inside the incubator the equilibrium took about 2-3 weeks. Samples were kept in triplicate and the average values of EMC have been reported. The EMC of each sample was determined with the help of a vacuum oven at 70±1 °C temperature and pressure less than 100 mm Hg for 24 hr (AOAC 1990). The same experiment was conducted for sorption process at 30, 40 and 50°C by changing the temperature of the incubator. A glass bottle containing 5 ml toluene was placed in those desiccator whose relative humidity higher than 75 % to prevent mold growth (Labuza, 1984). When moisture content of the bael samples reached equilibrium with the relative humidity of the atmosphere maintained inside the desiccator, the water activity a_w of the sample was equal to the relative humidity (fraction) of the atmosphere inside the desiccators.

The monolayer moisture content of a food material provides the maximum moisture content upto which the product is more stable. It is a crucial parameter in the determination of the surface potential of moisture sorbed in food. It was a satisfactory specification for the lower limit of moisture in food. The monolayer moisture values were obtained by fitting the GAB equation (Table 1).

Analysis of sorption data

A large number of models have been proposed in the literature for the sorption isotherms. The experimental data were fitted to four sorption models viz., Anderson, GAB, Oswin and Peleg models. The different models are presented in Table 1. The coefficients of individual models were determined by means of standard regression technique using Origin 7.5 software.

Validation of sorption models

The various sorption models were evaluated for their suitability in predicting the sorption behavior of the sample on the basis of coefficient of determination (R^2), reduced Chi-square (χ^2) values and residual plots. The residuals were plotted against predicted values of EMC. A model is considered acceptable, if the residual values falls in horizontal band centered around zero, displaying no systematic tendencies (i.e. random in nature) towards a clear pattern. If residual plot indicates clear pattern, the model is not considered acceptable.

Heat of sorption

The net isosteric heat of sorption can be used to estimate the energy requirements of drying and provides important information on the state

Table 1. Sorption models used for fitting the experimental data

Model	Equation
Anderson	$X_e = \frac{A.B.C.a_w}{1+(B-2).C.a_w+(1-B).C^2.a_w}$
GAB	$X_e = \frac{(A-1).X_m.B.a_w}{1+(A-1).B.a_w} + \frac{X_m.B.a_w}{1-a_w.B}$
Oswin	$X_e = A.\left(\frac{a_w}{1-a_w}\right)^B$
Peleg	$X_e = A.(a_w)^C + B.(a_w)^D$

Sources: (Peleg, 1993; Wang and Brennan, 1991)

Figure 1. Pictorial view of bael fruit

of water in food products. The net isosteric heat of sorption q_{st} (kJ g^{-1} mol^{-1}) was determined using the Clausius–Clapeyron equation (Eqn. 1) (Labuza et al., 1985, Rao et al., 2006, Zhengyong et al., 2008):

$$\ln\left(\frac{a_{w2}}{a_{w1}}\right) = \frac{q_{st}}{R}\left(\frac{1}{T_1} - \frac{1}{T_2}\right) \quad (1)$$

where, a_{w1} and a_{w2} are water activities (decimal) at temperatures T_1 and T_2 (K), respectively. The isosteric heat of sorption Q_{st} (kJ g^{-1} mol^{-1}) was calculated from the relationship

$$Q_{st} = q_{st} + \Delta H_v \quad (2)$$

Where, ΔH_v (kJ mol^{-1}) is the latent heat of vaporization of pure water at any temperature. Here temperature was taken 35°C (ΔH_v = 43.53 kJ mol^{-1}), the average of the two temperatures (20⁰C and 50⁰C) for the study.

Results and Discussion

Sorption isotherm

The desorption isotherm of bael pulp (18 °Brix) and adsorption isotherm of bael pulp powder at 20, 30, 40 and 50 °C are presented in Figure 2 and 3, respectively. It can be evident from the figures that the relationship between ERH and EMC data is type II sigmoid. The EMC increased with an increase in ERH at any temperature, while it decreased with the increase in temperature at the same a_w (ERH). The result may be explained that at higher temperatures the kinetic energy of the water molecules was high and water absorption at a given ERH was low (Demertzis *et al*., 1989; Mohamed *et al*., 2004). Similar results have been reported in the literature for the sorption isotherm (Stencl *et al*., 1999; Basunia and Abe, 2001; Lahsasni *et al*., 2003; Mohamed *et al*., 2004; Ghodake *et al*., 2007; Sinija and Mishra, 2008).

Three regions could be identified on the isotherms viz., region I (a_w : 0.0–0.45), region II (a_w: 0.45–0.85) and region III (a_w: 0.85–1.0). The moisture uptake was slow in the region I, followed by a linear and steady

Table 2. Constants of different models fitted to the desorption data of bael pulp

Models	Constants	Temperature (⁰C)			
		20	30	40	50
Anderson	A	2.8564	1.675	1.509	1.381
	B	0.1055	0.1350	0.1105	0.1027
	C	0.8736	0.9053	0.9318	0.9246
	R^2	0.997	0.998	0.998	0.997
	χ^2	0.0134	0.0069	0.0063	0.0069
GAB	M_w	0.1637	0.1192	0.076	0.0364
	A	4.2848	4.6643	3.0378	9.9143
	B	1.021	1.0532	1.1008	1.166
	R^2	0.998	0.999	0.997	0.994
	χ^2	0.0072	0.0031	0.0082	0.0153
Oswin	A	0.3226	0.2481	0.1821	0.1579
	B	0.7509	0.7921	0.8576	0.8429
	R^2	0.996	0.998	0.999	0.998
	χ^2	0.0168	0.0062	0.0035	0.0037
Peleg	A	0.2515	0.2644	0.3121	0.2532
	B	5.8639	5.3596	5.2331	4.2838
	C	0.3029	0.3760	0.5395	0.3958
	D	9.6717	10.9080	13.3542	13.2818
	R^2	0.999	0.996	0.999	0.998
	χ^2	0.0001	0.0023	0.0056	0.0037

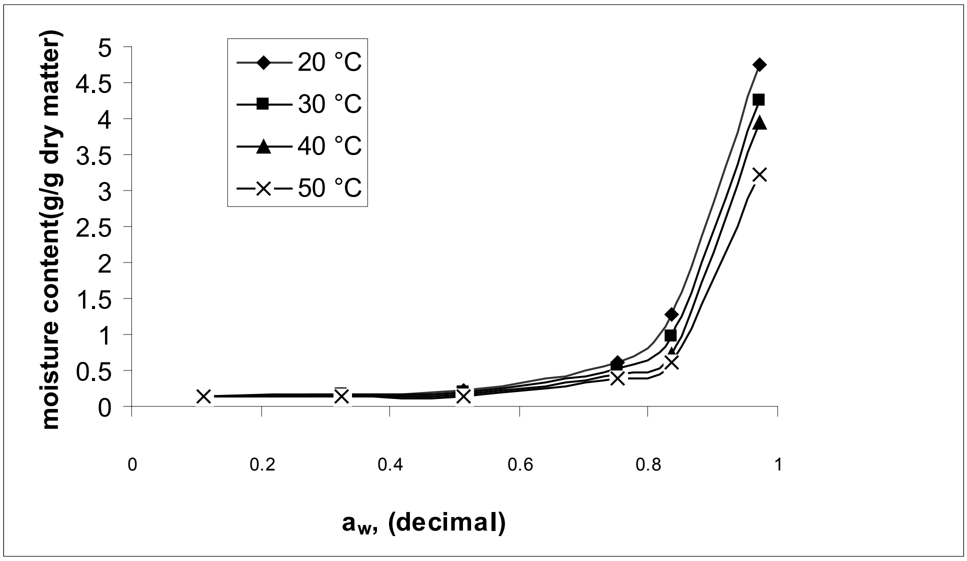


Figure 2. Desorption isotherms of bael pulp at different temperatures

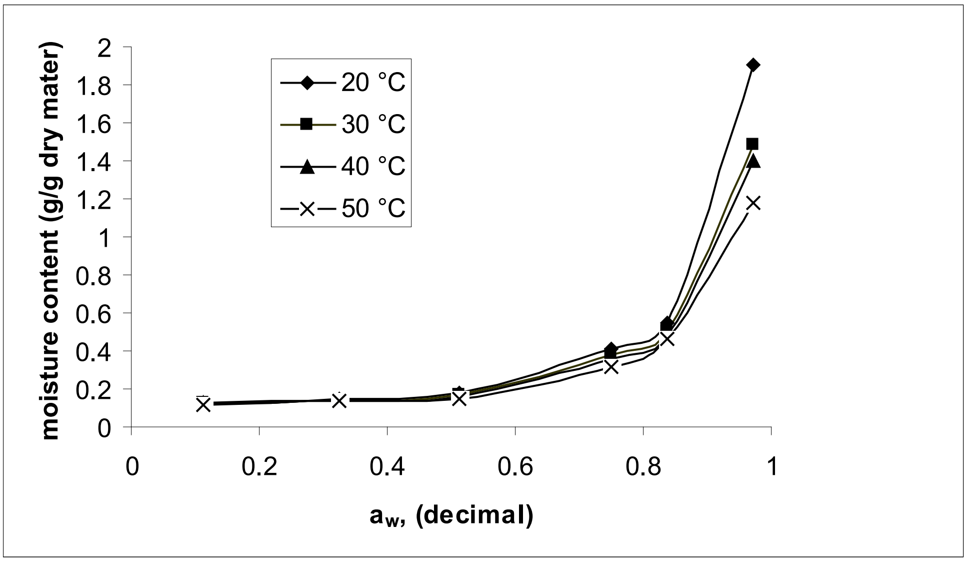


Figure 3. Adsorption isotherms of bael pulp powder at different temperatures

rise in the region II and a subsequent accelerated rise in the region III. This trend is similar to the work reported by previous researcher for sugar-rich products like dried fruit and dairy products (Kinsella and Fox, 1987; Ayranci *et al*., 1990; Tsami *et al*., 1990; Rao *et al*., 2006).

The monolayer moisture content values for bael pulp and its powder are obtained from GAB model are shown in Table 2. As temperature increased from 20 to 50 °C, the monolayer moisture value of pulp decreased upto 3.64% d.b. This may be due to the higher water vapour pressure in pulp at higher temperature. The similar effect of temperature on monolayer moisture content on Murici fruit (Byrsonima sericea) and Inga fruit (Inga edulis) was reported by Aroldo *et al.* (2006). The monolayer moisture content of dried pulp powder at different temperature also shown in Table 3.

Fitting of sorption models

Four sorption models were fitted to the experimental data of bael pulp (18 °Brix) and its freeze-dried powder. Table 2 and 3 represent the values of different constants of models, the coefficient of determination (R^2) and reduced chi-square (χ^2) values at different experimental temperatures for desorption of bael pulp and for adsorption of bael pulp powder, respectively. It was found that Peleg model gives best

Table 3. Constants of different models fitted to the adsorption data of bael pulp powder

Models	Constants	Temperature (^{0}C)			
		20	30	40	50
Anderson	A	1.2964	1.1926	1.2019	0.8926
	B	0.1364	0.1575	0.14695	0.2027
	C	0.8277	0.7863	0.7845	0.7678
	R^2	0.991	0.986	0.985	0.981
	χ^2	0.0073	0.0064	0.00609	0.00526
GAB	M_w	0.0875	0.06651	0.0917	0.08765
	A	30.7972	38.4817	12.0813	12.0131
	B	1.0077	1.03571	1.05695	1.0998
	R^2	0.997	0.989	0.932	0.811
	χ^2	0.0022	0.005	0.0273	0.0526
Oswin	A	0.2038	0.2058	0.1936	0.1908
	B	0.6235	0.5508	0.5523	0.5095
	R^2	0.997	0.996	0.995	0.992
	χ^2	0.0015	0.0013	0.0014	0.0015
Peleg	A	0.295	0.2274	0.2017	0.1597
	B	2.1759	1.5992	1.5236	1.2555
	C	0.4973	0.3291	0.2707	0.1432
	D	10.8997	8.8752	8.7389	7.516
	R^2	0.997	0.996	0.997	0.999
	χ^2	0.0032	0.0022	0.0019	0.0003

fit to the experimental data for both the products at almost all temperatures with high R^2 values and low χ^2 values (Table 2 and 3). Figure 4 shows desorption and adsorption behavior of bael pulp and powder at 30°C with Peleg model. Figure 5 shows some residual plots for bael pulp and its powder at 30°C. It can be seen from the residual plots that the values fell in horizontal band centered on zero with no systematic tendencies, which indicates a best fit.

Heat of sorption

The calculation of the variation of net isosteric heat of sorption as a function of moisture content, at the mean temperature of 35 °C, was done by Eqn (1). For example, in case of bael pulp at moisture content of 0.5 g/g dry matter and a_w values of 0.71 and 0.826 at 20°C and 50°C, respectively, the net isosteric heat of sorption was 3.97 kJ mol^{-1} while the corresponding heat of sorption was derived as 47.50 kJ mol^{-1}. The relationship between isosteric heat of sorption of pulp and powder with moisture content is plotted in Figure 6 (a) and (b), respectively. At moisture content below 1.25 g/g dry matter the isosteric heat of sorption increased sharply for bael pulp and for bael pulp powder it increased sharply below moisture content 0.5 g/g dry matter. These values were estimated as 47.5 kJ mol^{-1} and 46.12 kJ mol^{-1} for pulp and powder, respectively. However, at moisture content above those the isosteric heat of sorption fell almost in line with the heat of vaporization of pure water for both the products. 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 site giving lower heats of sorption (Iglesias and Chirife, 1982). The net isosteric heat of sorption ranged from 3.97 kJ mol^{-1} at moisture content of 0.5 g/g dry matter to 1.0 kJ mol^{-1} at a moisture content of 3.5 g/g dry matter for bael pulp and the corresponding value for bael pulp powder was 2.6 kJ mol^{-1} at moisture content of 0.25 g/g dry matter and 0.87 kJ mol^{-1}at moisture content of 1.125 g/g dry matter. The isosteric heat of sorption was found to be a good measure of the interaction of water vapour with the food substrate.

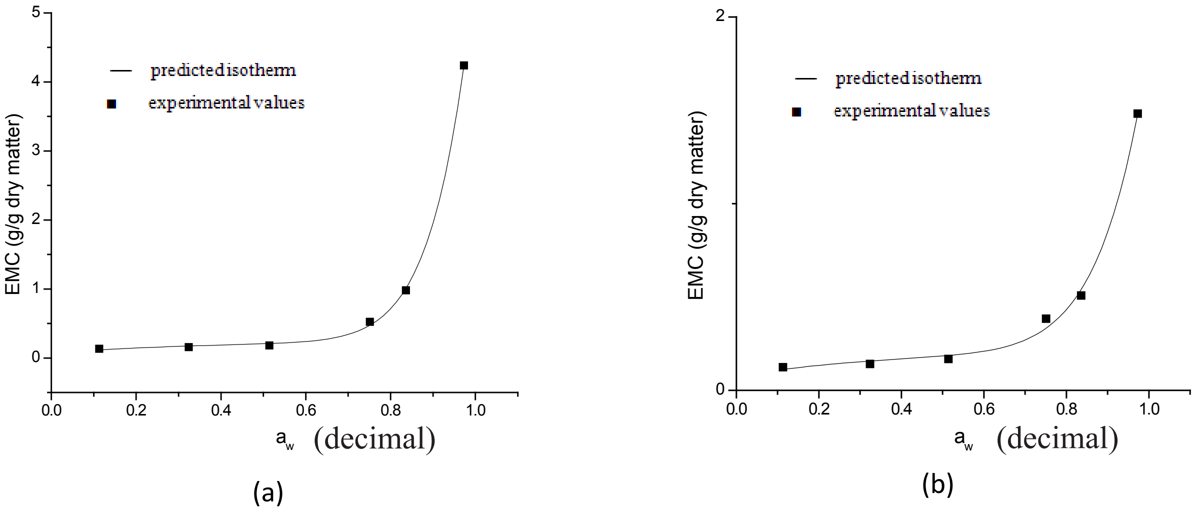
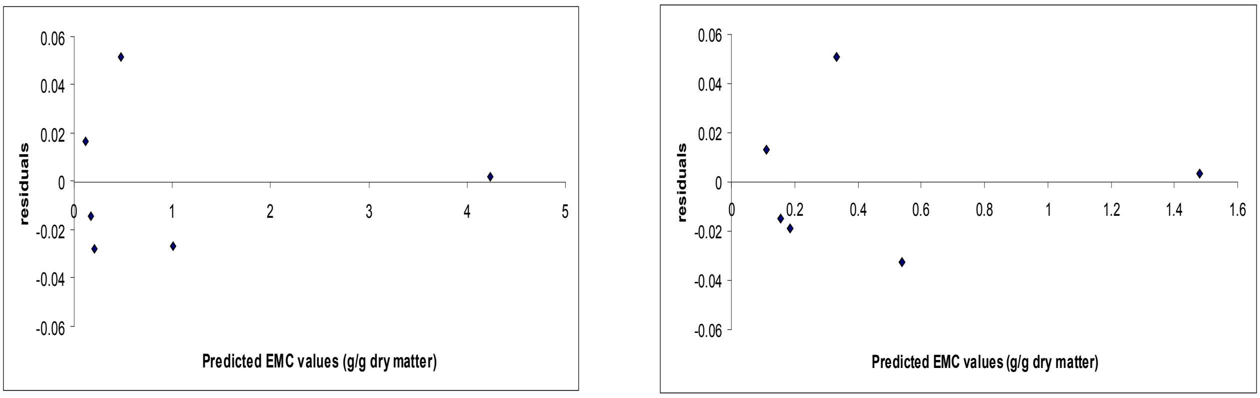
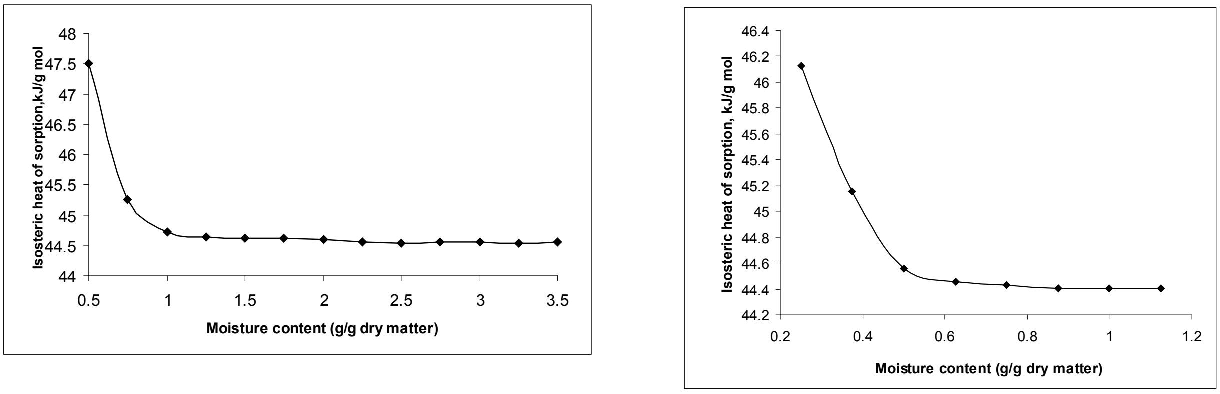


Figure 4. Desorption (a) and adsorption (b) curves for bael pulp and powder at 30 ºC with Peleg model.



(a) (b)

Figure 5. Residual plots for Peleg's Model at 30 °C a) bael pulp and b) powder



(a) (b)

Figure 6. Variation of isosteric heat of sorption with moisture content for a) bael pulp and b) powder

Conclusions

The moisture sorption isotherms of pulp and its powder samples showed a sigmoid shape and belong to type II classification. The moisture sorption of the product increased steeply above 0.85 a_w. The experimental data illustrated that the equilibrium moisture content (EMC) increased with decreasing temperature, at constant equilibrium relative humidity (ERH). A reduction in moisture sorption and water-binding capacity was observed as the temperature of sorption increased from 20°C to 50°C. Furthermore, at constant temperature, the EMC increased with increasing ERH. Four sorption models were tested for their ability to fit the experimental data. The Peleg model fitted best to describe the sorption isotherm at all the temperatures studied for both bael pulp and bael pulp powder. The isosteric heat of sorption curve showed a regular fall with increasing moisture content and approached the heat of vaporization of free water at higher moisture content.

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