Moisture sorption studies on vacuum dried coconut presscake

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Abstract Moisture sorption isotherms of vacuum dried coconut presscake were determined at 25 °C, 30 °C, and 38 °C using a gravimetric technique. Sorption isotherms exhibited Type II behaviour. The effect of temperature on equilibrium moisture content was not significant ($p>0.05$). Three models available in the literature viz., GAB, BET, and Halsey models were fitted to the sorption data. Out of the three tested models, Halsey model best fitted the experimental data with a relative deviation percent below 10%. The net isosteric heat of sorption was determined from the equilibrium adsorption data using the Clausius–Clapeyron equation. Isosteric heats of sorption were found to decrease exponentially with increasing moisture content.

Keywords Sorption isotherm · Coconut presscake · Equilibrium moisture content · Isosteric heat of sorption

Introduction

Coconut has been a traditional food in almost all South Asian countries. In the aqueous processing of coconuts, extraction of coconut milk is a major step, which is used for many purposes viz., recovery of coconut oil, conversion to spray dried coconut milk powder and preparation of

coconut skim milk and cream. One of the by-products after coconut milk extraction is the coconut solid residue known as presscake. Coconut presscake poses a tremendous disposal problem to processors of coconut milk owing to its high nutritional content. One of the novel uses of this by-product is drying of the presscake (vacuum drying, tray drying) and making it into flour, which can be used as ingredients in the preparation of curries, chutney etc. (Hagenmier 1983; Gonzalez 1986; Arumughan et al. 1993; Jena and Das 2007). Dried defatted coconut presscake can also be used as dietary fibre in weight control foods (Raghavendra et al. 2004; Trinidad et al. 2001).

Coconut presscake, being a nutrition rich product is susceptible to deteriorative reactions such as browning, lipid oxidation and microbial growth during long term storage. Packaging and storage systems for such products need to be carefully designed. In this regard, moisture sorption isotherms serves as useful hydrodynamic tool for designing suitable packaging and storage systems to optimize retention of product quality and biological stability (Debnath et al. 2002). Sorption behavior of various coconut products viz., fresh coconut and copra has been studied by various researchers (Diamante 1995, Nelson and Okos 2005). Nelson and Okos (2005) studied the sorption behaviour of fresh coconut at temperatures of 25, 35, 45, 55, and 60 °C and humidities up to 80%. However, little information exists on sorption characteristics of coconut presscake.

Formulating a unique universal mathematical model, either theoretical or empirical, for accurate prediction of sorption isotherms in the whole range of water activity for different types of foods is difficult (Chen 2000). Taking into account that food materials with similar chemical composition but different physical characteristics may give different sorption isotherms, it is necessary to obtain

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experimental data and then determine specific values for the adequate model (Vulliou et al. 2004). In light of these facts, it is necessary to investigate the sorption behavior of coconut presscake in order to find out the best fit model.

Knowledge of the temperature dependence of sorption phenomena provides valuable information about changes related to the thermodynamics of the system. The Clausius–Clapeyron equation is used to predict the temperature effect on water activity and has the advantage that it gives the heat of adsorption and desorption for food materials (Arslan and Toğrul 2005). The isosteric heat of sorption is used as an indicator of binding energy of absorbed water by the solid particles (Fasina et al. 1997). As a theoretical prediction of heat of sorption of water is not feasible due to the complexity of the physicochemical structure of foodstuffs, it is necessary to determine the heat of sorption experimentally, either by direct calorimetric measurements or from moisture sorption data (Mulet et al. 1999).

The purpose of the present study was to determine moisture sorption isotherms of vacuum dried coconut presscake at various temperatures, to evaluate the applicability of various existing models for fitting the sorption data and also to calculate the heat of sorption.

Material and methods

Coconut presscake preparation Dehusked mature coconuts were purchased from the local market. Coconut was steamed in an autoclave at 338.43 kPa (3.34 kg.cm^{-2}) pressure for 12.5 min to remove the shell and testa (Jena and Das 2004). The steamed coconut meat was washed, cut into small pieces and comminuted by a hammer mill. Particle size of coconut gratings was fixed at $2000 \mu\text{m}$ (Zicarelli et al. 1981). Grated coconut was mixed with water in the ratio of 5:1 and coconut milk was extracted by using a hydraulic press at a pressure of 219.3 kPa (2.16 kg.cm^{-2}) for 10 min. The coconut presscake obtained after the extraction of coconut milk was analyzed for moisture, fat, protein, carbohydrate, crude fibre, and ash content as per the standard methods prescribed in AOAC (1997). The composition of raw coconut presscake was: moisture $50.93 \pm 1.31\%$, fat: $35.15 \pm 1.54\%$, protein: $4.04 \pm 0.5\%$, fiber: $1.80 \pm 0.2\%$, ash: $1.15 \pm 0.25\%$ and carbohydrates: $6.92 \pm 1.1\%$. The presscake obtained after the extraction of coconut milk was used for vacuum drying.

Drying of coconut presscake A laboratory scale vacuum dryer was used for the drying coconut presscake at a temperature of 70°C . Absolute pressure maintained inside the dryer was $62 \pm 3 \text{ mm Hg}$. The presscake was placed in a tray at a thickness of 3 mm and drying was carried out for 2.5 h, during which moisture content of the presscake reduced below $0.02 \text{ kg water. kg dry solid}^{-1}$. The dried

product was in the form of flakes, which was ground by mortar and pestle after cooling to room temperature ($29 \pm 1^\circ\text{C}$). The moisture content of vacuum dried presscake was $0.016 \pm 0.002 \text{ kg water. kg dry solid}^{-1}$. Vacuum dried coconut presscake was used for sorption studies.

Sorption studies Equilibrium Moisture Content (EMC) method was followed to obtain adsorption isotherms of vacuum dried coconut presscake. Seven saturated salt solutions (LiCl , $\text{KC}_2\text{H}_3\text{O}_2$, $\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$, K_2CO_3 , $\text{Mg}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$, NaCl and KCl) giving a range of relative humidities of 0.114, 0.237, 0.329, 0.443, 0.536, 0.762, and 0.855 respectively were used. Each solution was transferred to airtight glass desiccators. Duplicate samples of about 1 g of the vacuum dried coconut presscake were weighed in small petri dishes and placed above the saturated salt solutions inside the desiccators, and tightly closed. The desiccators containing the samples were placed in an incubator maintained at a selected temperature for equilibration. Three temperatures of 25°C , 30°C , and 38°C were investigated. The temperatures were selected based on the possible range of temperatures that may be encountered during storage of coconut presscake.

Weight of each of the samples was measured every day. The total time for removal, weighing and replacing the samples was less than 10 s. This minimizes the atmospheric moisture sorption during weighing. The experiment was completed when less than 1% weight change was found between two successive readings. No visible mould growth was observed during the experiments. Equilibrium moisture content of the samples was measured by vacuum oven method (AOAC 1997). Average reading of the replicated samples was taken for development of isotherms. Water activity of the samples was measured in Aqua lab water activity meter (Model CX2, USA).

Fitting to existing mathematical models Equilibrium moisture content data was fitted using the Guggenheim–Anderson–de Boer (GAB), Brunauer–Emmett–Teller (BET) and Halsey models. The three models are presented in Table 1. The constant C is related to the first layer heat of sorption and K is a factor correcting the properties of multi layer molecules with respect to the bulk liquid.

Experimental data up to water activity values of 0.5 were used to fit BET model as this model is known to be valid below this value of water activity (Chirife and Iglesias 1978). The constants of the selected models were estimated by non-linear regression analysis using a software package (Origin 6.0). The goodness of fit of the selected models was judged from the relative deviation percent modulus, R_d . A model with R_d value of less than 10% is considered a good

Table 1 Equilibrium moisture sorption models fitted to experimental data

Name of model	Equation	Reference
GAB	$X = CKMa_w / [(1 - Ka_w)(1 - Ka_w + CKa_w)]$	Ayranci et al. (1990)
BET	$X = C_B Ma_w / [(1 - a_w)(1 - a_w + C_B a_w)]$	Chirife and Iglesias (1978)
Halsey	$\ln X = (1/r) \ln A - (1/r) \ln[\ln(1/a_w)]$	Ayranci et al. (1990)

X and M are the equilibrium and monolayer moisture content respectively, kg water. kg dry solid⁻¹; a_w is the water activity, fraction; C and K are GAB model constants; C_B is BET model parameter; A and r are Halsey model constants.

fit (Wang and Brennan 1991). Residual plots were also used to assess the curve fitting agreement of selected models. The relative deviation percent modulus was calculated from Eq. 1.

$$R_d = \frac{100}{N} \left[\sum_{i=1}^N \left(\frac{|X_{pi} - X_{ei}|}{X_{ei}} \right) \right] \quad (1)$$

where, X_{ei} and X_{pi} are the experimental and predicted equilibrium moisture content respectively. N is the total number of observations.

Net isosteric heat of sorption Since sorption data was obtained at different temperatures it is possible to evaluate the net isosteric heat of sorption at various moisture contents calculated using the best-fit isotherm. The study of sorption isotherms at two different temperatures provides the thermodynamic data on net isosteric heat of sorption through use of the integrated form of Clausius-Clapeyron

equation (Labuza et al. 1985; Bianco et al. 2001); which is given by,

$$\ln(a_{w1}/a_{w2}) = (q_{st}/18R)[(1/T_2) - (1/T_1)] \quad (2)$$

where, a_{w1} and a_{w2} are the water activities (fraction) at temperatures T_1 and T_2 respectively. q_{st} =Net isosteric heat of sorption (kJ. kg water⁻¹), and R =Universal gas constant (8.314 kJ. kmol⁻¹ K⁻¹).

Results and discussion

Moisture sorption isotherms Sorption isotherms of dried coconut presscake at 25 °C, 30 °C, and 38 °C are shown in Fig. 1a, b and c respectively. The deviations in experimental results have also been shown in the figure in the form of error bars. The isotherms were sigmoid in shape of the Type II isotherm. A slow increase in equilibrium moisture content was observed between the water activity values of 0.1 and

Fig. 1 Moisture sorption isotherms of dried coconut presscake at different temperatures. **a** 25 °C **b** 30 °C **c** 38 °C

Table 2 Models with their respective constants and relative deviation percent

Model	Model parameters					
Temperature:25 °C	<i>C</i>	<i>K</i>	<i>M</i>	<i>A</i>	<i>r</i>	<i>R_d</i> %
GAB	9.294	0.986	0.019	–	–	10.5
BET	34.037	–	0.015	–	–	9.62
Halsey	–	–	–	0.013	1.17	8.33
Temperature:30 °C						
GAB	4.50	0.978	0.020	–	–	9.03
BET	6.791	–	0.018	–	–	9.48
Halsey	–	–	–	0.015	1.10	4.17
Temperature:38 °C						
GAB	3.496	1.00	0.021	–	–	10.1
BET	3.924	–	0.020	–	–	13.02
Halsey	–	–	–	0.022	0.99	6.19

0.6 followed by a steep increase beyond 0.6 at all the temperatures. Increase in temperature resulted in slight decrease in equilibrium moisture content. At increased temperatures, some water molecules may have been activated to energy levels that allow them to break away from their sorption sites, thus decreasing the equilibrium moisture content (Palipane and Driscoll 1992). However, the effect of temperature on equilibrium moisture contents of dried coconut presscake was found to be insignificant from the analysis of variance (ANOVA) of experimental data (probability $p > 0.05$). Same type of result was also observed by Maskan and Göğüş (1997) for pistachio nut paste.

Fitting of moisture sorption data to existing models GAB, BET and Halsey models were used to fit the experimental moisture sorption data. Constants and relative deviation percent of these fitted models have been presented in Table 2. From Table 2 it can be observed that relative deviation percents of all the fitted models except BET model were lower than 10%. This describes the adequacy of prediction of equilibrium moisture content values by Halsey and GAB

models. R_d values of the Halsey model (8.33%, 4.17%, and 6.19% for 25 °C, 30 °C, and 38 °C respectively) were lower than the corresponding R_d values of GAB and BET model. Lower the values of R_d , better is the goodness of fit of the model. Moreover, the applicability of BET model was only up to water activity of 0.5. Figure 2 show the deviations in experimental equilibrium moisture contents and corresponding predicted values by Halsey model respectively. Based on the above observations and Fig. 2 it can be ascertained that Halsey model fitted best to the experimental moisture sorption data for dried coconut presscake. The uniformly scattered points in residual plots of Halsey model indicated the acceptability of this model as the best and more suitable model to predict equilibrium moisture content of dried coconut presscake in the temperature range studied.

Monolayer moisture content Monolayer moisture content corresponds to the amount of moisture adsorbed by a single layer to the binding sites in the food material. Monolayer moisture content, as obtained by the GAB model was 0.020 ± 0.001 kg water. kg dry solid⁻¹. The effect of temperature on GAB monolayer moisture content values was not much

Fig. 2 Moisture sorption isotherm predicted by Halsey model at different temperatures **Fig. 3** Variation of net isosteric heat of sorption with equilibrium moisture content

pronounced. BET monolayer moisture content values (0.015 and 0.018 kg water. kg dry solid⁻¹) were found to be slightly lower than corresponding GAB values (0.019 and 0.020 kg water. kg dry solid⁻¹) at low temperatures. However, at higher temperatures, both GAB and BET monolayer moisture contents were almost equal (0.020 and 0.021 kg water. kg dry solid⁻¹). The values of K of GAB model were less than or equal to unity.

Net isosteric heat of sorption The evaluation of net isosteric heat of sorption, q_{st} as a function of equilibrium moisture content was done by applying Eq. 2 to the isotherm data at 25 °C and 38 °C by using the best fit Halsey model. Figure 3 represents the variation of net isosteric heat of sorption with equilibrium moisture content. From Fig. 3 it is observed that net isosteric heats of sorption decreased exponentially as the moisture content increased. Large endothermic values at low moisture contents indicated weaker interactions between water vapor and the dried presscake. These interactions became stronger as the moisture content is increased. This type of behavior has been observed by Maskan and Göğüş 1997 and Hayoglu and Gamli 2007 for pistachio nut paste.

Conclusion

Moisture sorption characteristics of vacuum dried coconut presscake could be predicted agreeably with both GAB and Halsey model. However, Halsey model was found to be more suitable for accurate prediction of sorption isotherm at different temperatures with a relative deviation percent below 10%. The model developed in the present study may be utilized in predicting the equilibrium moisture content of dehydrated coconut presscake at various temperatures and relative humidity. This study will help in designing packaging systems of a nutritious product like coconut presscake so that it can be stores for a longer period for preparations of value added products. No significant effect of temperature on the equilibrium moisture content of coconut presscake was observed. Net isosteric heat of sorption for dried coconut presscake decreased with an increase in moisture content suggesting endothermic reaction at low moisture contents.

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