

## Sorption isotherm study on vacuum and freeze-dried jamun pulp

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### Abstract

Fruits play the most important role in nutrition. In India many underutilized medicinal fruits are available, and the most important fruit is jamun which is otherwise called black plum. Drying of underutilized jamun fruits helps in minimizing their post-harvest loss which occurs during harvesting season. The freeze-dried powder results with polar sites in molecular structure material which is occupied with water in wet conditions. During drying the molecules which were holding the water particles are attracted closely to each other. The study on sorption isotherms was carried out on the dried powder. These results gave a suitable model to fit sorption behaviour. The empirical equations frequently used to model drying kinetics are Newton, Henderson–Pabis, Page, Modified Page, Wang–Singh, Logarithmic, Two-Terms, Modified Henderson–Pabis and many mathematical models to describe the water sorption behaviour of foods. The GAB equation was fitted as the best fit for vacuum-dried jamun pulp powder stored at 27°C and 37°C.

## 1. Introduction

The important thermodynamic tool used for the interaction between a food component and water is the sorption isotherm. This sorption isotherm gives information for food processing operation based on the equilibrium moisture content of that product with relation to the relative humidity of that particular surrounding maintained at a constant temperature. The sorption isotherm models can predict the amount of water that can be held by the food material if it is exposed to air at a particular relative humidity.

This moisture absorption of the equilibrium moisture content obtained during the study mainly depends on the environmental relative humidity with the composition of the food. In general, the consumption of berries has increased during the last decade and berries of different kinds are widely consumed in many countries (Heinonen, 2007; Szajdek and Borowska, 2008). Dried jamun fruit has been used as food ingredients on fruit infusion. Over recent decades the post-harvest technology used for preserving berry fruit is drying. Drying will prolong the shelf life of the fruit with its

quality. By lowering the moisture content through drying there will be a decrease in spoilage and contamination during storage reported by the authors (Akpınar and Bicer, 2005). Drying of jamun fruit is considered as an important source of phenols, antioxidants, flavonoids minerals and is used as an ingredient in functional foods.

Simulation of the drying process through mathematical modelling is an important tool used to minimize operative problems such as product damage and excessive consumption of energy, among others (Stamatios and Vassilios, 2004) The empirical equations frequently used to model drying kinetics are Newton, Henderson–Pabis, Page, Modified Page, Wang–Singh, Logarithmic, Two-Terms, Modified Henderson–Pabis and many mathematical models to describe the water sorption behaviour of foods can be found in the literature (Ahrne, 2004). The Guggenheim-Anderson-de Boer (GAB) model is considered to be the most versatile and the best one for fitting the sorption data for the majority of food products in a water activity range of 0 – 0.85 (Lomauro *et al.*, 1985; Prothon and Ahrne, 2004). A common way of presenting the relationship between *aw* and water content is a sorption isotherm (Gondek and

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Lewicki, 2005) however, there is no direct relationship between these two parameters. The shape of the curves depends, above all, on the composition and structure of the material, temperature, and pressure. Knowledge of the moisture sorption characteristics is crucial for shelf-life predictions and determination of critical moisture and water activity for acceptability of products that deteriorate mainly by moisture gain and are important in drying, packaging and storage (Bianco *et al.*, 2001).

## 2. Materials and methods

### 2.1 Sample preparation

Mature Jamun fruits were directly obtained from producers in the region of Pollachi. The fruits were sorted by their maturity and the fully ripped fruits were washed in normal tap water. The free water in the fruit was removed using a hairdryer and wiped down using tissue papers. Pre-weighed 100 g of jamun fruit was packed in each polypropylene zip lock bag and kept in a deep freezer at -30°C for further use.

### 2.2 Drying conditions

The stored jamun fruits were taken from the deep freezer and kept at room temperature to reach their normal state. Jamun pulp was extracted manually by separating the pulp from the seed. Approximately 500 g of pulp was taken for the drying experiment.

### 2.3 Vacuum shelf drying

The pulp of jamun fruits was kept in stainless steel trays and arranged in a vacuum shelf drier. The process parameters in vacuum drying of jamun pulp were vacuum pressure, the temperature of drying and loading weight.

### 2.4 Freeze drying

The selection of the freezing conditions is very important to optimize the efficiency of the freezing process and to preserve the biological microstructure of the samples. Freeze drying was carried out using a laboratory freeze dryer (Freeze Mobile 24, Virtis Company, Inc., Gardiner, NY). The thawed jamun pulp was poured into a stainless pan to form a layer of 10 mm. The samples were placed at 25°C for 24 hrs before transferring to the freeze dryer. The vacuum pressure of the dryer was set at 20 Pa, the plate temperature was -20°C, -30°C and -40°C, and the condenser was at 60°C. The residence time needed to dry the jamun pulp to below 0.05 kg water/kg dry solids was determined when the vacuum pressure had dropped to 30 mTorr (4 Pa).

### 2.5 Adsorption isotherm determination

An adsorption isotherm study was done based on Gondek and Lewicki (2005) methods. A known weight of 5 g of samples was taken and placed in desiccators which contains nine different saturated salt solutions in each desiccator. The saturated salt solutions are LiCl ( $a_w = 0.113$ ), potassium acetate ( $a_w = 0.225$ ), MgCl<sub>2</sub> ( $a_w = 0.329$ ), K<sub>2</sub>CO<sub>3</sub> ( $a_w = 0.438$ ), Mg(NO<sub>3</sub>)<sub>2</sub> ( $a_w = 0.529$ ), NaNO<sub>2</sub> ( $a_w = 0.648$ ), NaCl ( $a_w = 0.753$ ), (NH<sub>4</sub>)<sub>2</sub>SO<sub>4</sub> ( $a_w = 0.810$ ), Thymol was placed in the desiccators containing NaCl, (NH<sub>4</sub>)<sub>2</sub>SO<sub>4</sub> in order to prevent the growth of mould. The samples were weighed periodically till they attained equilibrium, after which they were analysed for moisture content. To establish moisture sorption isotherms, the equilibrium moisture contents determined by the static gravimetric method, were plotted against water activity. For the study the samples were kept in both 37°C and 27°C.

### 2.6 Sorption isotherm model fitting

The data obtained corresponding to  $a_w$  and moisture content at the temperatures studied was adjusted to Henderson, Hasley, Iglesias and Chirife and GAB.

$$\text{Henderson} \quad M = a(-\ln(1 - a_w))^n \quad (1)$$

$$\text{Hasley model} \quad M = a(-\ln(a_w))^n \quad (2)$$

$$\text{Iglesias and Chirife} \quad \ln(M + (M^2 + M_{0.5})^{\frac{1}{2}}) = ba_w + c \quad (3)$$

$$\text{GAB} \quad M = \frac{M_0 C K a_w}{(1 - K a_w)(1 - K a_w + C K a_w)} \quad (4)$$

$$C = C_0 \exp(\Delta H / RT) \quad (5)$$

$$K = K_0 \exp(\Delta H_k / RT) \quad (6)$$

where M is the equilibrium moisture content,  $a_w$  water activity, C, K are GAB model constant, a, b, c fitting model constant, M<sub>0</sub> monolayer moisture content, C is related to first layer heat of sorption factor, K is factor correcting c and are related to monolayer and multilayer properties (Bajpai and Tiwari, 2013)

### 2.7 Model validation

In our study the Henderson, Hasley, Iglesias and Chirife and GAB equations are used to model the moisture isotherm for jamun pulp powder dried under both vacuum and freeze dryer. The models were validated with the experimental data using nonlinear regression. The calculations were analysed using the MATLAB Version 5.3 (Mathworks, Inc., Natick, MA). The statistical validity to fit the models were evaluated using statistical parameters such as the root mean square error (RMSE) and determination of co-efficient (R<sup>2</sup>). Models are used to show the relationship between the equilibrium moisture content, water activity and temperature. Best fit for both the samples were evaluated

based on the model run with high R<sup>2</sup>, lowest Chi-square and RMSE value which is close to zero.

$$RMSE = \sqrt{\frac{\sum_{i=1}^N (M_{exp} - M_{pre})^2}{N}} \times 100 \tag{7}$$

$$\lambda^2 = \frac{\sum_{i=1}^N (M_{exp} - M_{pre})^2}{(N-z)} \tag{8}$$

### 3. Results and discussion

#### 3.1 Sorption isotherm

The sorption isotherm for jamun pulp obtained by vacuum-dried and freeze-dried powder were shown in Figure 1 and Figure 2. The equilibrium moisture content tends to decrease with respect to an increase in temperature on given particular water activity. Similar results were obtained by Catelam *et al.* (2011). It was observed that the increase in temperature has a very evident effect on the equilibrium moisture sorption isotherm on both the vacuum and freeze-dried samples. Garrone *et al.* (2010) found that the isotherms of pineapple, mango and guava dried under vacuum, at temperatures of 25 and 50°C, showed the same behaviour observed by Silva *et al.* (2010). In Figure 1, a

slight variation was observed on the equilibrium moisture content of the vacuum-dried sample. These results might be due to the hygroscopicity of the powder. These results suggest that the absorption on the porous microparticles will have resulted with type III isotherms (Admson, 1990). The composition of amorphous sugars is very important in determining the sorption behaviour and stickiness of a multi-component food powder. This pattern was observed commonly on high sugar content food products. According to the study of Garrone *et al.* (2010), the interaction points on the isotherm model depends mainly based on the type of sugar present and sugar size distribution in food samples.

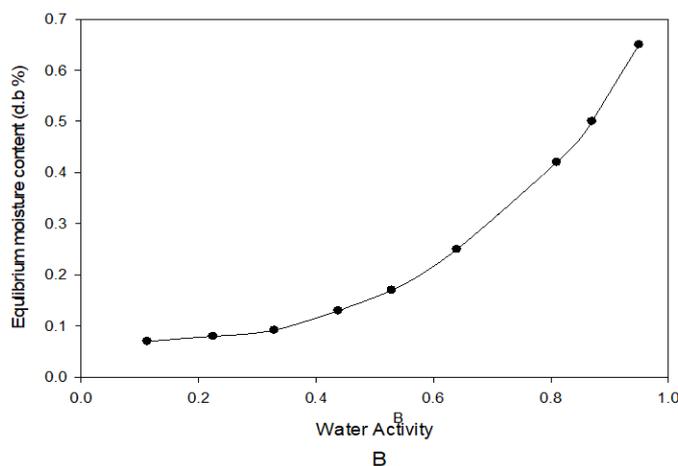
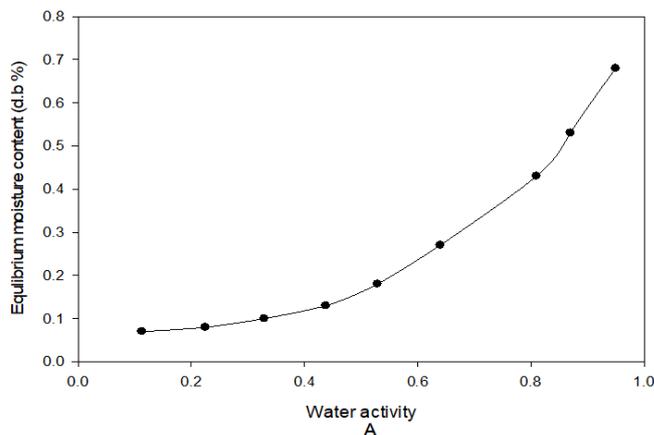


Figure 2. Moisture sorption isotherms of freeze-dried jamun pulp powder A: (37°C) B: (27°C)

On top of that, the freeze-dried powder results with polar sites in molecular structure material which was occupied with water in wet conditions. During drying the molecules which were holding the water particles were attracted closely to each other. This significantly reduces the water holding capacity of the material on subsequent adsorption. The freeze-dried jamun pulp powder has a high degree of hygroscopicity as it intends to take the moisture greater to 50% on its dry weight. This moisture absorption occurs during the storage of freeze powder for one week under certain conditions (RH is less than 90%). 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

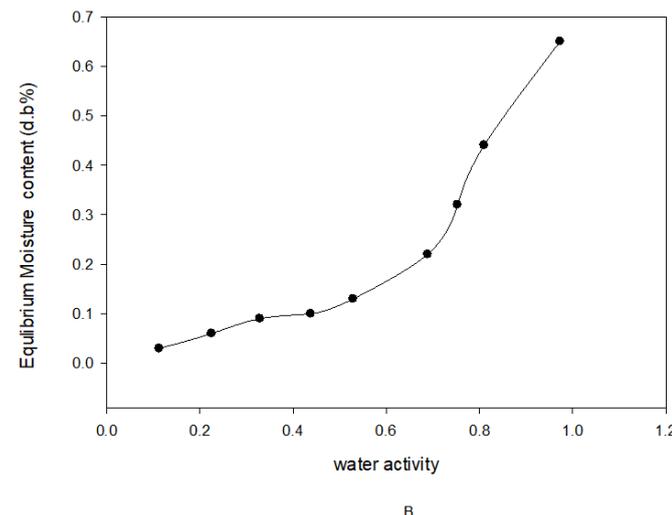
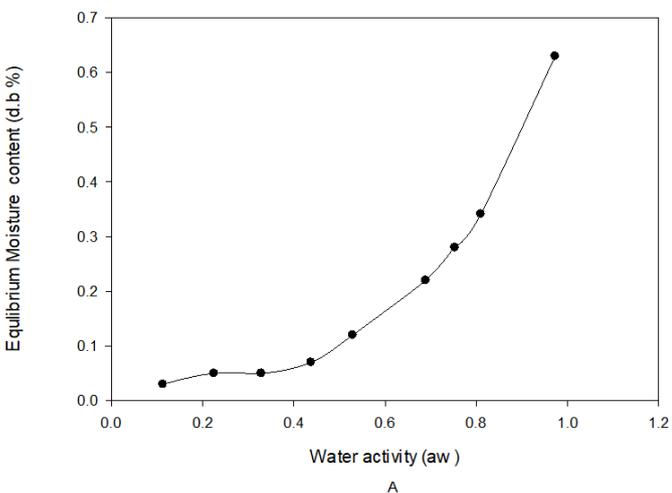


Figure 1. Moisture sorption isotherms of vacuum-dried jamun pulp powder A: (37°C) B: (27°C)

chemical reactions may be accelerated or physical changes catalysed (Callahan *et al.*, 1988).

### 3.2 Moisture sorption isotherm modelling

The experimental moisture content on sorption study for freeze and vacuum-dried jamun pulp at 27°C and 37°C were fitted to Henderson, Hasley, Iglesias and Chirife and GAB equation using nonlinear regression analysis are shown in the Table 1. Models are used to show the relationship between the equilibrium moisture content, water activity and temperature VD and FD jamun pulp powder. On most of the food and biological substances, the adsorption curves will be sigmoid as suggested by Sopade *et al.* (2010). The coefficient ranges between 0.8153 to 0.9592 on 27°C and 0.8515 to 0.9632 on 37°C on the adsorption model for freeze-dried jamun pulp powder. The adsorption model coefficient ranges between 0.7739 to 0.9533 on 37°C and 0.7811 to 0.9114 on 27°C of vacuum-dried jamun pulp powder.

To determine the goodness of fit, the models have to be suggested with coefficient determination, RMSE and Chi-square values (Kaymak *et al.*, 2004). The data obtained in Table 1 and Table 2 suggests that comparing the value models run for sorption isotherm with high  $R^2$  which is close to one, lowest values of Chi-square and RMSE close to zero value of Henderson model describes the goodness of fit in adsorption study in the water activity and temperature range study of freeze-dried jamun pulp powder. However, the GAB equation was fitted as the best fit for vacuum-dried jamun pulp powder on both 27°C and 37°C sorption studies are shown in Table 3 and Table 4. In overall evaluation on vacuum-dried samples. The GAB model is not so surprising based on its semi theoretical and homogenous adsorption has been suggested as a versatile sorption model available (Al-Muhtaseb *et al.*, 2008). According to Van der Berg and Bruin (1980), it is clear that the C and K parameters incorporate the effect of temperature. Whereas this C and K are enthalpy and entropic in nature which suffices temperature effect on sorption models.

## 4. Conclusion

The study on sorption isotherms was carried out on freeze-dried and vacuum shelf dried powder. The important thermodynamic tool used for the interaction between food components and water is the sorption isotherm. These results gave a suitable model to fit sorption behaviour. Temperature affected the sorption behaviour, with equilibrium moisture content decreased with increasing temperature at constant water activity. On comparing the value models run for sorption isotherm with high  $R^2$  which is close to one, lowest values of Chi-square and RMSE close to zero value of

Henderson model describes the goodness of fit in adsorption study in the water activity and temperature range study of freeze-dried jamun pulp powder. The GAB model is not so surprising based on its semi theoretical and homogenous adsorption has been suggested as a versatile sorption model. In general, it can be concluded that among the dried pulp powder freeze-dried powder can be recommended for long term storage of jamun pulp powder.

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Table 1. Moisture sorption (27°C) fitted data of freeze-dried jamun pulp powder

Model	Equation	Parameter values	R <sup>2</sup> value	Standard Error Estimate	RMSE	Chi-square
Henderson	$M = a(-\ln(1 - a_w))^n$	a = 21.9637, n = 0.9626	0.9592	5.0648	2.46	7.70
Halsey	$M = a(-\ln(a_w))^n$	a = 13.8795, n = -0.4747	0.8737	8.9152	1.14	10.47
Iglesias and Chirife	$\ln(M + (M^2 + M_{0.5})^{\frac{1}{2}}) = ba_w + c$	b = 73.5080, c = -16.3161	0.8153	10.7803	1.99	6.98
GAB	$M = \frac{M_0CKa_w}{(1 - Ka_w)(1 - Ka_w + CKa_w)}$	M <sub>0</sub> = 8.8444, C = 161.16 × 10 <sup>6</sup> , K = 0.9065	0.9153	7.8866	1.85	9.80

Table 2. Moisture sorption (37°C) fitted data of freeze-dried jamun pulp powder

Model	Equation	Parameter values	R <sup>2</sup> value	Standard Error Estimate	RMSE	Chi-square
Henderson	$M = a(-\ln(1 - a_w))^n$	a = 18.4601, n = 0.9469	0.9632	3.6643	1.63	1.03
Halsey	$M = a(-\ln(a_w))^n$	a = 11.2503, n = -0.4987	0.8763	6.7215	0.66	3.21
Iglesias and Chirife	$\ln(M + (M^2 + M_{0.5})^{\frac{1}{2}}) = ba_w + c$	b = 57.6018, c = -11.8104	0.8515	7.3649	2.18	7.30
GAB	$M = \frac{M_0CKa_w}{(1 - Ka_w)(1 - Ka_w + CKa_w)}$	M <sub>0</sub> = 7.3446, C = 32.67 × 10 <sup>6</sup> , K = 0.9041	0.9102	6.1847	1.38	1.80

Table 3. Moisture sorption (37°C) fitted data of vacuum-dried jamun pulp powder

Model	Equation	Parameter values	R <sup>2</sup> value	Standard Error Estimate	RMSE	Chi-square
Henderson	$M = a(-\ln(1 - a_w))^n$	a = 29.5716, n = 0.7208	0.9070	7.4274	0.266	4.38
Halsey	$M = a(-\ln(a_w))^n$	a = 19.949, n = -0.3703	0.7739	11.58	0.657	9.37
Iglesias and Chirife	$\ln(M + (M^2 + M_{0.5})^{\frac{1}{2}}) = ba_w + c$	b = 74.2718, c = -11.2207	0.8833	8.3189	0.544	6.92
GAB	$M = \frac{M_0CKa_w}{(1 - Ka_w)(1 - Ka_w + CKa_w)}$	M <sub>0</sub> = 9453.5, C = 0.0066, K = 0.4173	0.9533	5.6822	0.22	5.94

Table 4. Moisture sorption (27°C) fitted data of vacuum-dried jamun pulp powder

Model	Equation	Parameter values	R <sup>2</sup> value	Standard Error Estimate	RMSE	Chi-square
Henderson	$M = a(-\ln(1 - a_w))^n$	a = 28.2792, n = 0.7371	0.9114	6.7248	0.281	10.26
Halsey	$M = a(-\ln(a_w))^n$	a = 18.3910, n = -0.4025	0.7811	10.5685	0.707	3.3
Iglesias and Chirife	$\ln(M + (M^2 + M_{0.5})^{\frac{1}{2}}) = ba_w + c$	b = 70.0883, c = -10.2875	0.9022	7.0644	0.538	4.39
GAB	$M = \frac{M_0CKa_w}{(1 - Ka_w)(1 - Ka_w + CKa_w)}$	M <sub>0</sub> = 12.1167, C = 329 × 10 <sup>5</sup> , K = 0.8517	0.8511	9.4149	0.600	9.40

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