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Research Article

Moisture Sorption Characteristics of Locust Bean Pulp Flour

Peter I. Akubor

Department of Food Science and Technology; Federal University Wukari, Taraba State, Nigeria

Abstract Flour was prepared from African locust bean pulp (ALBPF). The equilibrium moisture contents of the African locust bean pulp flour were determined at 30, 35 and 40 ^{o}C and fitted into Brunauer, Emmet and Teller (BET), Smith, Oswin, Henderson, Iglesias and Chirife; and Guggenheim-Anderson –de Boer(GAB) models. The goodness of fit of the models was evaluated using Coefficient of determination (R^2) and root mean square error (RMSE). The moisture sorption isotherms of the flour at the three temperatures exhibited sigmoldal shape. The equilibrium moisture content (EMC) of the ALBPF at the three temperatures increased slowly at low water activities (a_w 0.1- 0.3) but increased rapidly at high water activities (a_w 0.5-0.9). The EMC of the ALBPF up to a_w 0.6 decreased with increase in temperature but increased sharply with increase in temperature at high water activities (0.7-0.9). The Oswin, Henderson models and GAB models described adequately the relationship between water activity and the water content of the ALBPF by their R^2 and the RMSE. The Iglesias and Chirife ($R^{2\,=}$ 0.9778-0.9913) and Smith ($R^{2\,=}$ 0.9273-0.933) models also indicated good fitting but with relatively higher RMSE values. Oswin and Henderson equations gave R^2 values of 0.9798-0.9900 and 0.9892- 0.9946, respectively and the RMSE values of 1.22-0.09 and 0.10-1.42, respectively. Oswin described the ALBPF isotherm better at high than at low temperatures. On the other hand, the Henderson model described the ALBPF isotherm well at 30 ^{o}C and 35 ^{o}C and less satisfactory at 40 ^{o}C. Both the R^2 and RMSE for the Henderson model increased with increase in temperature. The GAB model gave the best fit over the wide range of a_w (0.1- 0.9) evaluated in the present study. The GAB model had the lowest RMSE with values ranging from 0.04 to 0.10 and the highest R^2 values at the three temperatures studied. The GAB parameters (Wm, C and K) decreased with increase in temperature.

Keywords Sorption, isotherms, Models, pulp flour, African locust bean, Goodness of fit

Introduction

In Nigeria, African locust bean (*Parkia biglobosa*) tree grows widely throughout the savanna. The tree produces 25-52 kg of pods [1]. A mature pod contains yellow, dry and powdery pulp in which dark brown seeds are embedded [1]. The pulp is licked for its sweet taste but only to a small extent. The pulp is usually washed away when the seeds are processed into a condiment called *dawadawa* or *iru* which is a source of protein intake among the low income groups and rural populations of West Africa [2]. In West Africa, the pulp is prepared as flour and used in soups and stews or eaten with cereals as porridge [1]. A traditional drink is prepared from the fruit by infusing the dried ground fruit pulp in hot water [3]. This drink is widely consumed as health tonic and is valued for its many medicinal properties [3]. Products processed from locust bean pulp on experimental basis include jam and syrup [3, 4].

There growing interest in the utilization of locust bean pulp is due to its nutraceutical potential which has led to the extensive studies on the pulp [5]. The chemical composition and functional properties of locust bean pulp flour have been determined [1]. Locust bean pulp was reported to be rich in dietary fiber, essential vitamins and minerals and phytochemicals [5, 6]. The functional properties and the effects of pH and NaCl concentration on the functional properties of the pulp flour were recently reported by Akubor and Adedeji [6]. Akubor [4] reported the changes in the physicochemical, sensory and microbiological properties of syrup and jam prepared from locust bean pulp during storage. The previous studies showed that the flavonoids, carotenoids and vitamin C in the locust bean pulp would exert health promoting effect in addition to those of the dietary fiber [1, 7]. The potential of locust bean pulp flour as useful functional ingredient in diverse food product applications such as baked products was suggested by the previous studies [8, 9].

Packaged food powders absorb water during storage which causes changes in the chemical composition and functional properties [10]. Knowledge of the moisture sorption behavior of sugar containing foods like locust bean pulp flour is very important in selecting packaging materials as well as predicting the stability and moisture changes during storage [11]. Moisture sorption isotherm is used to investigate structural features of such as specific surface area, pore volume, pore size distribution and crystallinity of a food product [12]. Such data are used for selecting appropriate storage conditions and packaging systems that optimize or minimize retention of aroma, color, texture, nutrients and biological stability [13]. Moisture sorption isotherms are characteristic of a food and vary with different foods, processing methods and temperature [10, 14, 15]. Moisture sorption isotherm equations give insight into the moisture binding characteristic of foods [16]. Therefore, it is useful to determine the sorption characteristic of flours and the suitability of sorption models checked. Such models allow the understanding of the different aspects of the food-water interaction [17].

Therefore, the objectives of the study were to determine the moisture sorption characteristics of locust bean pulp flour and to assess the influence of temperature on the moisture sorption characteristics of locust bean pulp flour.

Materials and Methods

Source of Raw Materials

Mature and ripe African locust bean (*Parkia biglobosa*) fruit pods were plucked from locust bean trees in a local farm in Ugwaka –Ollah Township, Kogi State, Nigeria. Commercial wheat flour was purchased from a local shop in Idah Township, sieved through 60 mesh sieve (0.05 mm) (British standard) and stored in high density polyethylene bag in a refrigerator prior (10 ^{o}C) to use.

Preparation of Locust Bean Pulp Flour

Locust bean fruit pods were cleaned and split open manually. The yellow pulp along with the attached seeds were removed from the hulls, sun dried (48h) and pounded lightly in a mortar with pestle. The pulps were separated from the seeds, milled in a hammer mill and sieved through 60 mesh sieve (British standard).

Moisture Sorption Studies

The moisture sorption properties of locust bean pulp flour were determined using the static gravimetric technique as described by Ariahu *et al*. [12]. Saturated salt solutions of lithium chloride, potassium acetate, magnesium chloride, potassium carbonate, magnesium nitrite, sodium nitrate, sodium chloride, ammonium sulphate and barium chloride were used to obtain different relative humidity environments having water activities of 0.11, 0.21,0.33, 0.43, 0.50, 0.67, 0.76, 0.86 and 0.90, respectively in desiccators. The triplicate flour samples (2 g) were placed over the saturated salt solutions in the desiccators and kept in the temperature controlled cabinet (Gallenkamp, Uk) at 30, 35 and 40 ^{o}C, respectively. The samples were weighed daily using a digital weighing balance until they attained constant weight. The equilibrium moisture content (EMC) (dry basis) of each sample was determined by oven drying at 105 ^{o}C to constant weight [18]. The initial moisture content of the locust bean pulp flour was also determined by oven drying method [18]. The moisture sorption isotherms were obtained by plotting the EMC (% H_2O/100g, dry basis) against water activity.

Fitting Sorption Isotherm Models

The equilibrium moisture contents were fitted into Brunauer Emmet and Teller (BET), Oswin, Iglesias and Chirife, Henderson, Smith and GAB equations. The equations except the Gugeheim-Anderson DeBoer (GAB) were rearranged to linear forms to determine sorption models and their linear forms used for this study are presented in

Table 1.For BET, a_w = water activity, Mo =monolayer moisture content (dry basis); m=equilibrium moisture content, C=constant related to the net heat of sorption. In the other models, k,n,b,B, c are constants.

Table 1: Moisture models and their linear forms used for describing the moisture sorption isotherms of African locust bean pulp flour

Model	**Equation**	**Linear form**
BET	$Aw/(1-a_w)m \quad +a_w(C-1)/Mo \quad C.aw$ 0.05-0.45	$1/(1-a_w)m=1/mo +1/Cmo -1-a_w/a_w$
Oswin	$M=K\ a_w/1-a_w.a_w$ upto 0.50	
Iglesias and Chirife	$Ln(m+\sqrt{m2}+Mo_{0.05} =ba_w +P$	$Ln\ (m ++\sqrt{m2}+Mo_{0.05=}ba_w +P$
Henderson	$1-a_w =exp\ (-BM^c)$	$In\ \{-b\ (1-a_w =Clnm +ln\ B$
Smith	$M =B-C\ ln\ (1-a_w)$	$M =B- C\ ln(1-a_w)$

For BET, aw =water activity. Mo, Monolayer moisture content (dry basis).m,equilibrium moisture content. C, constant related to the heat of sorption. In the other models, k,n,b,p,B,c are constants.

GAB Model

The GAB model used for the study was given as:

$W/Wm = Ck_{aw}/(1-k_{aw})(1-k_{aw} +(k_{aw}))$…………Equation 1

Where a_w = water activity, w =equilibrium moisture content on a dry weight basis ($gH_2O/100g$, dry matter). Wm=moisture content on dry weight basis (g $H_2O/100g$, dry matter) with one molecule per active sorption site (formerly called monolayer in BET Model). C=GAB sorption constant. C (T) = C exp $(H_1 - Hm)/RT$.H_1 = total heat of sorption of the first layer on primary sites. Hm = total heat of sorption of the multilayer which differs from the heat of condensation of pure liquid water. HL= heat of condensation of pure water vapor.

The GAB model may be written as [12]:

$a_w/W = C_1 + C_2a_w + C_3\ a_w^2$ ……………..Equation 2.

$C_1 = 1/\ Wmck$

$C2 = 1/Wm\ (1-2/c)$

$C_3 = K\ (1/Wm\ c-1)$ …………………Equation 3.

Where Wm =monolayer moisture content. C =GAB sorption constant. K =GAB sorption constant related to multilayer properties. The parameters C_1, C_2 and C_3 were calculated using a quadratic regression [19].With these values, the GAB constants were determined by solving the system of equation 3.

Evaluation of the influence of temperature on GAB constants

The influence of temperature on all the three GAB constants was determined using the following equations [12]:

$Wm\ (T) = Wm^1 \exp\ (\Delta H/RT)$

$C^1\ (T) = C^1 \exp\ (H_1–Hn)/RT$…………Equation 4.

$K^1\ (T) = K^1 \exp\ (HL – Hm)/RT$

These equations are similar to the well known Arrhenius equations in reaction kinetics. The constants Wm^1, C^1, K^1 and the corresponding exponent in equation 4 were obtained from the GAB constant determined for several temperatures (30, 35 & 40 °C) by means of a least square analysis.

Results and Discussion

Sorption Isotherms

The adsorption moisture sorption isotherms of locust bean pulp flour at 30, 35 and 40 °C are shown in Fig. 1.Each point on the isotherm curves represents the average of three replications. The three sorption isotherms exhibited a sigmoid shape, which is described as type 11 isotherm according to the classification of Brunauer [12]. Most biological products have sigmoid isotherm curve [20]. At low and intermediate water activities, the equilibrium moisture content (EMC) of African locust bean pulp flour at the three temperatures investigated in the present study increased slowly. However, there was steep rise of EMC at high water activities. This behavior was reported as typical of sugar containing products [21,22]. Similar results were reported for sultana rasins [23] and dry fura powder [24]. This behavior of locust bean pulp flour could be attributed to physical adsorption of water on polymeric molecules (e.g. starch, protein) at low water activities and dissolution of sugars at high water activities [25]. In physical adsorption of water, the forces involved are mainly those of molecular interactions which embrace permanent dipole, induced dipole and quarapole attraction [25]. They are the type of forces responsible for non ideal behavior of gases, cohesion of molecules, Van der waals forces [25]. This behavior reflects the moderately high sugar content of locust bean pulp flour as well as the effect of water on the physicochemical state of sugars [21]. Gabas *et al*. [21] reported that most sugars may be present in the form of crystalline solids, amorphous solids (bound to other food components) and aqueous solution. In the locust bean pulp flour, the sugars were probably in the amorphous form where the sugars are very hygroscopic and unstable [25]. The adsorption of water imparted mobility to the sugar molecules and which resulted in their transformation from the metastable amorphous state to the more stable crystalline state [25]. In this form, glucose and sucrose began to dissolve (Fig.1).As the solubility of the sugars increased, the EMC of the locust bean pulp flour also increased. The time taken for the African locust bean pulp flour to reach equilibrium varied from 6 to 21 days and was shorter at lower water activities than at higher temperatures.

Influence of Temperature on Moisture Sorption Isotherms

The Fig .1 also showed that the EMC of the African locust bean pulp flour up to water activity of 0.6 decreased with increase in temperature. However, at higher water activities (a_w 0.7- 0.9), the EMC slightly increased as the temperature increased.

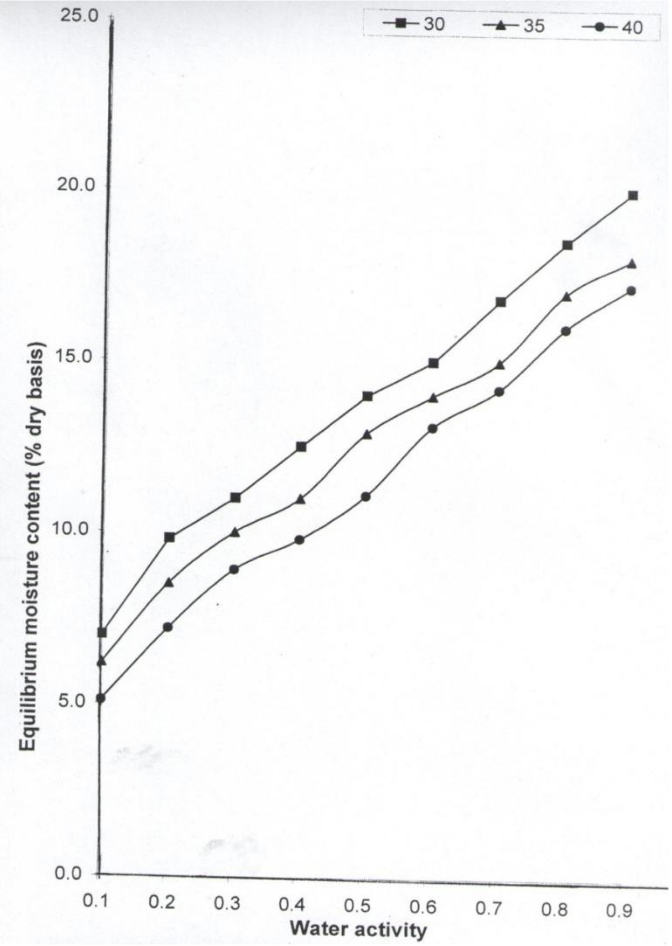

Figure 1: Adsorption moisture sorption isotherms of locust bean pulp flour at 30, 35 and 40 ºC

This resulted in crossing of the isotherm curves. Similar results were reported for millet fura powder containing 10g sugar [24].This indicated that the locust bean pulp flour within the water activity range of 0.1-0.6 became less hygroscopic with increase in temperature. This was in agreement with the results reported for corn and fish flours [26], dry fura [24] and skimmed powder [27]. At low water activities, adsorption of water on only crystal surfaces takes place [28]. This according to Crapiste and Rotstein [27], this behavior is necessitated by the thermodynamic relationship ΔG-ΔH=$T\Delta S$ where ΔG, ΔH and ΔS are changes in the free energy, enthalpy and entropy, respectively and T is the absolute temperature. Since $\Delta G < 0$ (sorption is a spontaneous process) and $S\Delta < 0$ (the sorbed water molecule has less freedom), then $\Delta H < 0$. Therefore, increase in temperature represents a condition unfavorable to water sorption [24]. Similar temperature effects have been reported by Smith et al. [29] for glucose and sucrose at low water activities. However, at high water activities ($a_w > 0.7$), sugar dissolution takes place and increase in temperature increases the water sorption for high sugar containing foods [28]. This results in the cross over of the isotherms at higher water activities.

This has been demonstrated by for sultana raisins which are high in sugars [23]. This observation contradicted the results obtained for African locust bean pulp flour at high water activities ($a_w > 0.67$) as the EMC slightly decreased ($p > 0.05$) with increase in temperature. The reasons for these observations were probably that the locust bean pulp flour contained 7.5 % total sugar [1] and when compared to fruits such as rasins that contained 20-30 % total sugar [24] cannot be classified as very high sugar product. Also, the equilibrium moisture contents of the African locust bean pulp flour were obtained at 30, 35 and 40°C. Eqwuje and Ariahu [30] studied the adsorption isotherms of velvet bean seeds at 278.1, 298.1 and 318.1 °K and reported no significant dependence of the EMC on the temperature. Thus, they concluded that the dependence of sorption data on temperature is clearer at higher temperature and when a wide range of temperatures are studied.

Analysis of sorption models

The selected six isotherm equations used for fitting the experimental equilibrium moisture content (EMC) of the African locust bean pulp flour, their linearized forms and constants are shown in Table 2. The parameter values of the six models used to describe the moisture sorption isotherms of locust bean pulp flour at 30, 35 and 40 ^{0}C are presented in Table 2. Although, the BET model holds for a limited range of water activity (0.1- 0.5)[10] , two constants namely monolayer (Mo) constant and energy content (C) were obtained. The BET equation is widely used to determine the monolayer moisture content [10]. The monolayer values shown in Table 2 were calculated from the BET equation using data for low water activity (0.1 -0.5). The Mo and C constants of the BET model slightly decreased with increase in temperature. The Mo of locust bean pulp flour decreased from 0.0752 Kg/Kg at 30°C to 0.0608 Kg/Kg at 40 °C. Similar observations were made for corn snacks and coffee [14,15]. This indicated that the shelf life of locus bean pulp flour would not decrease to a great extent with increase in temperature [14]. Monolayer values that varied from 3.2 to 16 g H_2O/100g DM had been reported for starchy foods [11]. The values obtained for locust bean pulp flour in the present study from the BET model (0.0608-0.0752 Kg DM) were comparable to 5.05 H_2O DM reported for wheat flour [22]. Moisture gained by a food in excess of the monolayer value represents free water, which promotes browning, hydrolysis, caking and other defect s[26]. The surface area of the LBPF obtained from the monolayer value was 1.089 m^2g^{-1}. This value was lower than 5.00 m^2g^{-1} reported for raffinose [28]. The sorption analyses of the different models showed that the Oswin and Henderson with two parameters (simple models) and the Guggenheim-Anderson –de Boer GAB models with three parameters described adequately the relationship between water activity (a_w) and the water content of the African locust bean pulp flour as determined by their R^2 (Coefficient of determination) and the RMSE (root mean square error) (Table 3). The values of the fit statistics for each of the models were satisfactory, taking into account the wide range of applications of these models. The Iglesias and Chirife ($R^{2\,=}$ 0.9778-0.9913) and Smith ($R^{2\,=}$ 0.9273-0.933) models also indicated good fitting but with relatively higher RMSE values. Oswin and Henderson equations gave R^2 values of 0.9798-0.9900 and 0.9892- 0.9946, respectively and the RMSE values of 1.22-0.09 and 0.10-1.42, respectively.

Table 2: Parameter values for the models used to describe the moisture sorption isotherm of African locust bean pulp flour

Model	**Range of a_w**	**Temperature (°C)**	**Constants**		**R^2**	**RMSE**
	0.05-0.45		Mo	C		
BET		30	0.0752	49.10	0.4529	0.48
		35	0.0680	40.84	0.6236	0.22
		40	0.0608	26.40	0.8457	0.20
Oswin	0.1-0.5		K	N		
		30	0.1390	0.3140	0.9798	1.22
		35	0.1243	0.3200	0.9871	0.12
		40	0.1119	0.3665	0.9900	0.09
Iglesias and Chirife	0.1-0.9		P	B		
		30	-0.8178	0.3682	0.9802	0.30
		35	-0.8435	0.3578	0.9778	1.42
		40	-0.8755	0.36.24	0.9913	13.14
Henderson	0.1-0.9		B	C		
		30	276.91	2.9703	0.9892	0.10
		35	264.49	2.8101	09902	0.11
		40	249.86	2.5058	0.9946	1.42
Smith	0.1-0.9		B	C		
		30	0.0872	-0.0524	0.9273	1.45
		35	0.0764	-0.0506	0.9333	1.45
		40	0.0650	-0.0504	0.9332	2.10
GAB		Wm	C	K		
		30 0.3165	1.0158	0.7753	0.9909	0.09
		35 0.2833	1.0214	0.8055	0.9849	0.10
		40 0.0980	0.5710	0.5558	0.9934	0.04

R^2, Coefficient of determination. RMSE, root mean square percentage error. a_w, water activity. Mo, monolayer moisture content (% dry basis).m, equilibrium moisture content. C, constant related to the net heat of sorption. K,n,b,P,B ,C are constants.

The constants derived from these sorption isotherms are used to construct moisture sorption isotherms which are helpful in describing moisture changes during ingredient mixing, packaging and storage [10]. Linear models with high R^2 and low RMSE are satisfactorily acceptable [21]. Among the two parameter equations, Oswin described the isotherms of the African locust bean pulp flour better at high than at low temperatures. While the R^2 increased from 0.9798 at 30 °C to 0.9000 at 40 °C, the RMSE decreased from 1.22 at 30 °C to 0.99 at 40 °C. On the other hand, the Henderson model described the African locust bean pulp flour isotherm well at 30 °C and 35 °C and became less satisfactory at 40°C. Both the R^2 and RMSE for the Henderson model increased with increase in temperature. The fitting precision is about the same for Oswin and Henderson models (Table 2). The Henderson model has the practical significance of identifying critical upper moisture content necessary for the shelf stability of food [21]. However, Oswin holds for a limited range of water activity (a_w = <0.5). The GAB model gave the best fit over the wide range of a_w (0.1- 0.9) evaluated in the present study. The GAB model had the lowest RMSE with values ranging from 0.04 to 0.10 and GAB model had nearly the highest R^2 values at the three temperatures studied (Table 3). The GAB model described the African locust bean pulp flour isotherm better at 40 °C (R^2 = 0.9934 and RMSE = 0.09). The GAB model (also known as the kinetic model based on a multilayer and condensed film) is the most versatile and has been adopted by many researchers to model sorption isotherms of many food materials, due to the physical meaning often attached to its parameters [10]. The fitting precision of the various models used to describe the moisture sorption isotherms of African locust bean pulp flour are presented in Table 3.

Table 3: Fitting precision of the various models used to describe the moisture sorption isotherms of African locust bean pulp flour

Model	RMSE
BET	0.3
Oswin	0.48
Iglesias and Chirife	4.95
Henderson	0.54
Smith	1.67
GAB	0.08

Values are means for the three temperatures investigated. RMSE, root mean square percentage error

GAB parameters

The Table 3 shows the fit parameters and the associated statistics for the GAB model at the 30, 35 and 40 ^{o}C. The GAB model was reported as providing a better evaluation of the amount of water tightly bound by primary adsorption sites (Wm) [10]. The Wm is the measure of water corresponding to the saturation of all sorption sites by a water monolayer (equivalent to Mo value of the BET model) [20]. At any temperature, the value of the Wm was higher than that of Mo for BET model. Similar observation was documented for habaneras cookies [15]. The Wm decreased from 0.3165 g H_2O/100g DM for the African locust bean pulp flour equilibrated at 30^{o}C to 0.2833g H_2O/100g dm and 0.0980g H_2O for LBPF equilibrated at 35 and 40^{o}C, respectively. Such slight decreases were also observed for the Mo of the BET equation (Table 2). This was in agreement with the results reported for food materials such as sugar beet, sweet potato slices and Jerusalem artichoke flour [31]. This implied that that increase in temperature increased the water activity at a constant moisture content and thus, making locust bean pulp flour more susceptible to microbial, nutritional and aesthetic degradation [14,30]. The monolayer moisture content averagely 0.23299 H_2O/100g (DM) corresponds to the optimal moisture content (minimizing spoilage reactions) for storage of locust bean pulp flour.

Effect of temperature on GAB parameters

When the temperature factor was added to the GAB constants (Table 4), the Wm (T) increased from 0.0096 gH_2O/100g DM at 30 ^{o}C to 00.1713 H_2O/100g DM at 35 ^{o}C and then dropped to 0.0995 H_2O/100g DM at 40 ^{o}C. The C (T) and K (T) constants decreased steadily with increase in temperature. Similar results were reported previously by Arogba [14] on the effect of temperature on the moisture isotherm of biscuits containing processed mango kernel flour. Sani et al. [32] also described the similar effect of temperature on GAB parameters for moisture sorption of fufu and tapioca.

Table 4: Effect of temperature on GAB constant

Temperature (^{o}C)	Wm(T)(gH_2O/100g DM	C(T)	K(T)
30	0.0096	1.0201	0.7903
40	0.1713	0.7632	0.6627
50	0.0995	0.6051	0.5757

Values are means of three replications. Abbreviations are as defined in Table 2

Conclusion

The equilibrium moisture contents (EMC) of the African locust bean pulp flour at the three temperatures increased slowly at low water activities (a_w 0.1- 0.3) but increased rapidly at high water activities (a_w 0.5-0.9). The EMC of the flour up to a_w 0.6 decreased with increase in temperature but increased sharply with increase in temperature at high water activities (a_w 0.7-0.9). The Oswin, Henderson and GAB models described adequately the relationship between water activity and the water content of the ALBPF. Oswin model described the ALBPF isotherms better at high than at low temperatures. The Henderson model described the ALBPF isotherm well at 30 °C and 35 °C and less satisfactory at 40°C. The GAB model gave the best fit over the wide range of a_w (0.1- 0.9).

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