



Moisture Sorption Characteristics of Lafun Flour

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Abstract	Article History
<p>Understanding the relationship between relative humidity and equilibrium moisture content (EMC) of any food material assist in maintaining good keeping quality. The adsorption isotherms for lafun supplemented with soy cord and residue were investigated. Six saturated salts were used which are Lithium chloride (RH: 11% a_w, 0.22); potassium (RH: 93%, a_w: 0.38); Magnesium chloride (RH: 33%, a_w:0.56); Potassium chloride (RH: 86%, a_w: 0.40); Potassium acetate (RH: 23%, a_w: 2.81); Potassium carbonate (RH: 43%, a_w: 1.15) providing constant relative humidity environments ranging from 11 – 93%. The experimental data were compared with five widely recommended models in the literature for food adsorption isotherms (GAB, Oswin, Modified Oswin, BET and Henderson). The moisture adsorption isotherm were sigmoidal in shape and was influenced by temperature. Oswin model was best fits for all the samples at different temperatures. The monolayer moisture values for BET model of commercial ‘lafun’ sample are 0.040, 0.036 and 0.034 $kg\ kg^{-1}$, ‘lafun’ enriched with curd 0.036, 0.038 and 0.031 $kg\ kg^{-1}$, control ‘lafun’ 0.057, 0.038 and 0.025 $kg\ kg^{-1}$, and ‘lafun’ enriched with residue 0.030, 0.036, 0.029 $kg\ kg^{-1}$ at 10, 30 and 40°C respectively while GAB model gave monolayer moisture values of 0.0682, 0.063 and 0.053 $kg\ kg^{-1}$ for commercial ‘lafun’ sample, 0.045, 0.042 and 0.039 $kg\ kg^{-1}$ for ‘lafun’ enriched with curd, 0.065, 0.042, 0.039 $kg\ kg^{-1}$ for ‘lafun’ commercial sample 0.081, 0.066 and 0.061 $kg\ kg^{-1}$ for ‘lafun’ enriched with residue at 10 °C, 30°C and 40 °C respectively.</p> <p>Keywords: Sorption, Packaging materials, Equilibrium Moisture Content, Water activity, Models</p>	<p>Received: 01 Apr 2024 Accepted: 21 Apr 2024 Published: 03 Jul 2024</p> <div data-bbox="1203 860 1469 1122" style="text-align: center;"> </div> <p>Scan QR code to view* License: CC BY 4.0*</p> <div data-bbox="1203 1178 1469 1245" style="text-align: center;"> </div> <p>Open Access article.</p>
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Introduction

Controlling the food moisture content during processing and storage is very important since water plays a critical role in food reactions and food quality. Knowledge of sorption isotherm has a great importance in food dehydration (Foo and Hameed, 2010; Hay *et al*, 2022). The connection between the equilibrium moisture content in a biological materials and the relative humidity of ambient air at constant temperature is called a sorption isotherm. It is well known that most hygroscopic biological materials exhibit hysteresis in the adsorption and desorption isotherms. According to Ricardo *et al*. (2011), the characteristics of sorption isotherm in food can be classified into five different types including: Type 1 (Langmuir or similar isotherm), Type 2 (Sigmoidal sorption isotherm), Type 3 (Flory-Huggins isotherm), Type 4 (Adsorption of a swell able hydrophilic solid until a maximum of site hydration is reached) and the last type are Brunauer-

Emmett-Teller (BET). Normally, majority of food products exhibit type 2 and 4 sorption characteristic. In food dehydration, the sorption properties are very pivotal to shelf-life of food materials which are useful for quantitative approach prediction. From the previous research, two or three fitting parameters were used to describe moisture sorption isotherms with the use of mathematic model to predict the moisture content in that particular (Al-Muhtaseb, 2004). Hence, different food products have their specific models that are best for them. So, selected model should be suitable for type of food products.

Cassava (*Manihot esculenta Crantz*) is a planted tuber root crop. It is a major staple food crop is west-Africa, good source of carbohydrate and some other beneficial nutrient which are healthy to the body (Obanijesu and Olajide, 2009). It is one of the leading food and feed plants in the world where several fermented products are obtained including fufu, gari, and

lafun. It has been reported to be a source of food for more than 500 million people in the world (Abu, 2006; Anyaiwe *et al.*, 2018). Fermented cassava flour (lafun), as it called in Nigeria, is an African fermented product from cassava obtained by soaking peeled cassava chunks in water, at ambient temperature (28 - 32° C) for 2s–5days (Adetan, 2003). The cassava chunks are later sun-dried and milled. During the fermentation process, different biochemical changes occur such as degradation of cyanogenic compounds; formation of flavour compounds.

Materials and Methods

2.1 Sources of raw materials

Cassava roots *Manihot, esculenta* (crantz) belonging to the family of *Euphorbiaceae* (Spurge family), were obtained from the Teaching and Research farm of the Federal University of Technology, Akure, Ondo State, Nigeria. Soybeans *Glycine max* (TGX) were purchased from faculty of Agriculture farm produce store, Michael Okpara University of Agriculture, Umudike, Abia State, Nigeria.

2.2. Production of soy curd and residue

Soy bean seed (150 g) were sorted, cleaned, soaked (12 h) in 2 L of tap water containing 0.5 g NaHCO₃ in a cooking pot and

boiled for 25 min. The boiled and dehulled soybean seeds were then wet milled in a hammer mill. Water was added in ratio 1:8 and a muslin cloth was used to extract the milk (pH 6.40) and the residue was kept separate. Thereafter, the pH of the extracted milk was adjusted to 4.6 by adding 1 M citric acid. The soy milk was allowed to stand and the clear whey at the upper part was decanted while the lower part (curd) was collected after six hours. The curd and residue was oven dried (at 60°C for 24 h), milled, packaged in high density polyethylene HDPE and stored in the refrigerator until needed for further use. Figure 1A shows the production chart for the curd and residue.

2.3 Production of lafun

Freshly harvested cassava roots were peeled with knife, washed and cut into chunks, fermented for 4 days (pH 3.67), washed, sifted, milled into pulp and divided into two portions (Figure 1B). One portion was used as control (CL) while the other portion was enriched with either dry soy curd or residue using Pearson scale with, 10% enrichment level and also taking into consideration the water content of the mash at 100%. Sample supplemented with curd was named “Lafun enriched with curd” (LEC) and the other sample “Lafun enriched with residue” (LER). A commercial “Lafun” sample (CS) was obtained from FIIRO Oshodi, Lagos for comparison.

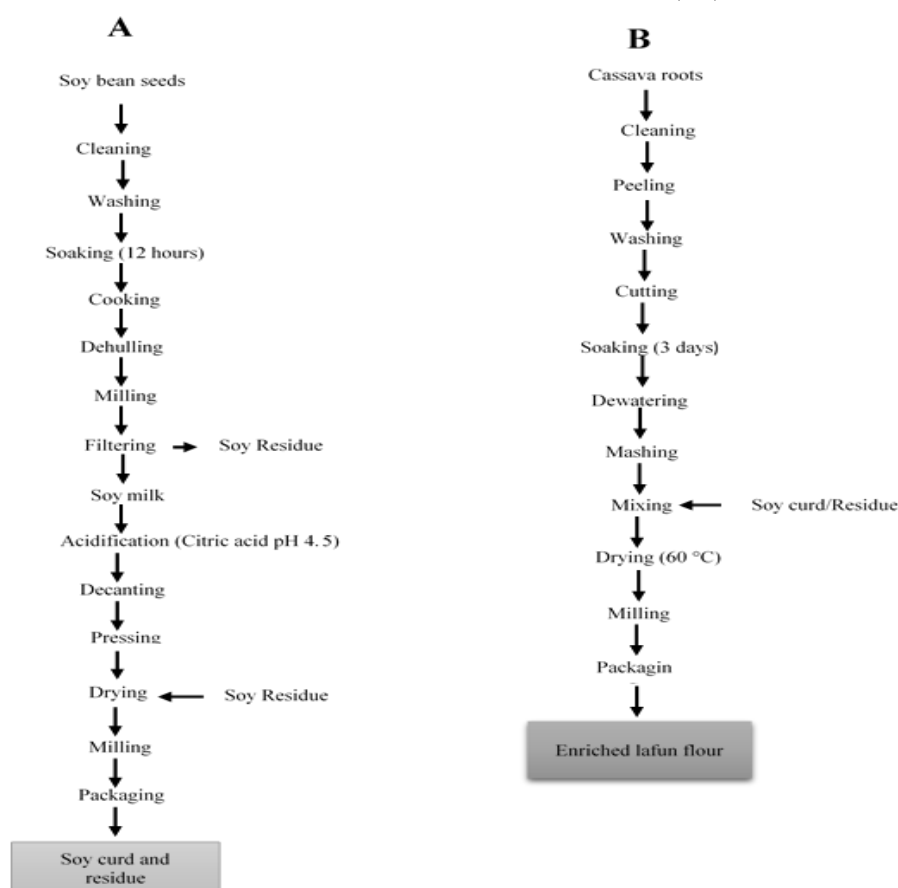


Figure 1: Production of soy supplement (curd and residue) (A) and enriched lafun samples (B)

2.4 Determination of equilibrium moisture content of ‘lafun’ samples

A static gravimetric method was used for the experiment as described (Greenspan, 1977) reported by Al-Ghouti (2020).

About 3 g of lafun sample was placed in a Petri dish inside 6 desiccators containing saturated salt solutions Lithium chloride (RH: 11%, a_w 0.22) potassium (RH: 93%, a_w: 0.38); Magnesium chloride (RH: 33%, a_w: 0.56); Potassium chloride

(RH: 86%, a_w : 0.40); Potassium acetate (RH: 23%, a_w : 2.81); Potassium carbonate (RH: 43%, a_w : 1.15) providing constant relative humidity environments ranging from 11 – 93% (David, 2008). The sample had been pre-dried in a desiccator with P_2O_5 at room temperature for 15 days. At high relative humidity's ($a_w > 0.7$), toluene (1.5 ml) was placed in the desiccators to prevent microbial growth (Halmi et al, 2014; Kumar and Mishra, 2004). The desiccators were kept in temperature incubator at constant temperatures of 10°C, 30°C and 40°C. The samples were weighed at intervals of 3 days using a digital balance (Model Mettler PE1600, Mettler Instruments Corporation, Greifensee, Zurich, Switzerland) until constant weights were obtained after two consecutive recordings, when the samples were assumed to be at equilibrium (± 0.001 g). The bone dry mass was determined by the oven-drying method for 8 – 10 h at 105 – 110°C (AOAC, 1990). The time to reach equilibrium ranged from 15 to 30 days depending on the water activity in each desiccator. The equilibrium moisture contents of 'lafun' sample were calculated from which the moisture sorption isotherms were determined.

2.5 Fitting of isotherm models

The experimental data were fitted to five commonly used models using the linear and nonlinear (NLIN) regression analytical procedures (Xion, 2002). The models were the two-parameter equations such as Hasley, Henderson, Oswin, B.E.T and the three-parameter G.A.B model (Wolf and Cowan, 1971; Chukwu, 2010). The quality of fitness of the models were evaluated by calculating the mean relative per cent deviation (%E), and the standard error of estimate (SEE) and coefficient of determination (r^2) using equations bellow respectively (Xion, 2002; Akanbi *et al.*, 2006):

GAB	$X_{eq} = \frac{M_o a_w c k}{(1 - k a_w)(1 - k a_w + c k a_w)}$
BET	$X_{eq} = \frac{c M_o a_w}{(1 - a_w)(1 + (c - 1) a_w)}$
Oswin	$X_{eq} = c \left[\frac{a_w}{1 - a_w} \right]^n$
Modified Oswin	$X_{eq} = (a + bT) \left(\frac{a_w}{1 - a_w} \right)^c$
Henderson	$X_{eq} = \left(- \frac{\ln(1 - a_w)}{c} \right)^{1/n}$

Where:

X_{eq} is the equilibrium moisture content

a_w is water activity

a , b , c , n are constants

k is the GAB constant

M_o is the monolayer moisture content

$$RSS = \sum_{i=1}^n (M_{cal} - M_{pred})^2$$

$$R^2 = 1 - \frac{\sum_{i=1}^n (M_{cal} - M_{pred})^2}{\sum_{i=1}^n (M_{pred} - M_{cal})^2}$$

$$SEE = \sqrt{\frac{\sum_{i=1}^n (M_{cal} - M_{pred})^2}{df}}$$

Where, M_{cal} = experimental equilibrium moisture content,

M_{pred} = predicted equilibrium moisture content,

n = number of experimental unit

df (degree of freedom) = $n - 1$,

RSS = residual sum of squares

SEE = standard error of estimate.

The experimental data was manually imputed and analysed with Microsoft Excel. Excel solver was used in analysing and determining model parameters. The formulated algorithm is carried out and the predicted curve is overlaid on the experimental data points. The best fit was determined based on closeness of R^2 to unity, the least values of RSS and SEE, the models were evaluated in terms of reliability of fit (Chowdhury *et al.*, 2005; Aviara *et al.*, 2004).

Results and Discussion

3.1 Moisture Sorption Isotherm behavior of 'lafun' samples under storage

Figure 2 showed the adsorption isotherms of control 'lafun' (A), 'lafun' enriched with soy curd (B), commercial 'lafun' sample (C) and 'lafun' enriched with soy residue (D), respectively. They were determined by plotting the equilibrium moisture contents against different water activities. The isotherm followed the sigmoid shape of type II classification by Brunauer *et al.* (1938) reported by Oyelade (2008) for *tacca* food. This isotherms is typical of products with high starch contents as observed by (De-Gruyter, 2019) the water molecules are bound in one layer to the surface of the pores by hydrogen bond or van der Waal forces.

3.2 Effects of Temperature on the moisture sorption isotherm of lafun samples

Figure 2 also showed the moisture sorption isotherms of 'lafun' samples with the same packaging Oluwamukomi (2009) and Oyelade (2008). The shape of the equilibrium isotherm can be divided into three parts depending on the type of fixation. At low RH (materials obtained at different temperatures 10°C, 30°C and 40°C. The moisture content decreased with increase in temperature which agreed with previous findings (Samapundo *et al.*, 2007; Oyelade *et al.*, 2008; Oyelade, 2008). The equilibrium water content in the material is dependent not only on the relative humidity of the ambient air, but also on the temperature of the air hence, the shape of the sorption isotherm are influenced by temperature. The equilibrium moisture content at each water activity represents the mean value of three replications. The water activity shift of food isotherms at constant moisture content with respect to temperature variation has been shown to be directly related to the rates of food deteriorative reactions as described Van den Berg and Bruin, (1981) reported by Chukwu, (2010). The equilibrium moisture content increased as water activity increased at any particular temperature and decreased as temperature increased at constant water activity (Paulo *et al.*, 2010). This indicates that the samples at different temperatures become less hygroscopic as temperature decreases. This agrees with Paulo *et al.* (2010) who observed that increase in temperature causes decrease in equilibrium moisture content of coffee using different processing methods and for saga starch when studying the moisture isotherm at different temperatures Bajpai and Pradeep (2013).

Temperature increase activates water molecules to higher energy levels, allowing them to break away from their sorption sites, therefore decreasing the equilibrium moisture content (Bonner and Kenney, 2013).

As temperature varies, the excitation of molecules, as well as the distance, and thus attraction between molecules varies. This causes the amount of absorbed water to change as temperature varies, at a given relative humidity (Muhammed et al, 2022). Mazza and Lemaguer (2008) suggested that an increase in temperature induces physical or chemical changes in the product, which reduced the

number of active sites for water to bind. The effect of temperature on equilibrium moisture content has an important practical bearing on chemical and microbiological reactions associated with spoilage (Al-Muhtaseb, 2004). This behaviour is most common with hydrated food materials and may simply be attributed to the facts that increase in temperature causes an increase in kinetic energy of water vapour molecules. The van der Waals forces between absorbed water vapour molecules and starch film become weak, thus resulting in less moisture uptake. Therefore, moisture sorption may be regarded as an exothermic process (Bajpai and, 2013). Many isotherms which followed this principle have been reported in the literature (Chowdhury *et al.*, 2005).

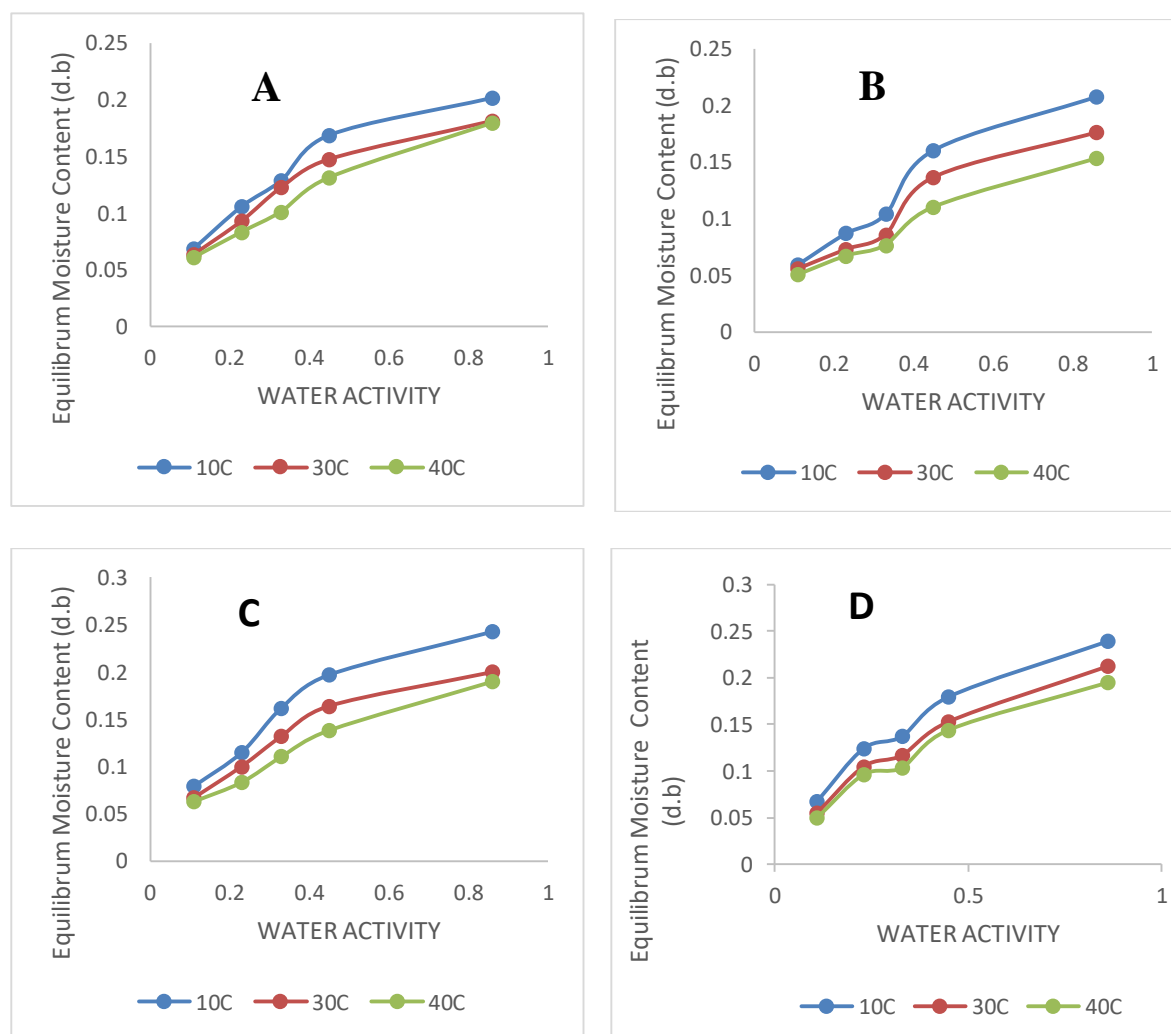


Figure 2: Sorption isotherm behavior of control 'lafun' (A), 'lafun' enriched with soy curd (B), commercial 'lafun' sample (C) and 'lafun' enriched with soy residue (D).

3.3 Fitting of Sorption Isotherm Models to Experimental Data

The calculated equilibrium moisture content was compared and fitted to the experimental sorption data. Based on the closeness of R^2 to unity, the least values of RSS and SEE, the models were evaluated in terms of reliability of fit (Chowdhury *et al.*, 2005, Aviara *et al.*, 2006). The models fitted for 'lafun' samples at 10°C, 30°C and 40°C with the values of the parameters (estimated unknown values) for the models which include R^2 , RSS, SEE and the constants for each model are presented in Tables 1-4. The BET isotherm is

usually valid for water activities (a_w) between 0.1 and 0.5 according to Bell and Labuza (2000). Igbabul *et al* (2013) reported that BET model is valid for water activities (a_w) between 0.1 and 0.5 when studying moisture adsorption isotherms of African arrow root lily (*tacca involucrata*) tuber mash influenced by blanching and natural fermentation. This is considered a disadvantage when compared with the GAB model which predicts equilibrium moisture content up to a_w of 0.90 (Rizvi, 2005). Based on R^2 , RSS and SEE the five models were evaluated and the best fit was determined. Oswin and Henderson models best fits for the three temperatures for

commercial lafun as shown in Table 1. This finding is similar to those reported by Kaymak-Ertekin and Gedik (2004) that Oswin equations are good for products high in starch. This disagreed with Oyelade (2008) that modified Oswin model was found to be most suitable for 'lafun' which could be due to botanical origin in the samples studied. The Oswin model has been reported to provide good descriptions of moisture isotherms throughout the entire range of water activity (Oswin, 1946). Oswin model also gave the best fit for other 'lafun' samples in this study.

3.4 Monolayer moisture content (M_0)

The monolayer moisture values for BET model of commercial 'lafun' sample are: 0.040, 0.036 and 0.034 kgkg^{-1} , for 'lafun' enriched with curd: 0.036, 0.039 and 0.031 kgkg^{-1} , for control 'lafun' 0.057, 0.038 and 0.025 kgkg^{-1} , for 'lafun' enriched with residue 0.030, 0.036, 0.029 kgkg^{-1} at 10, 30 and 40°C respectively while GAB model gave monolayer moisture values of 0.0682, 0.063 and 0.053 kgkg^{-1} for commercial 'lafun' sample, 0.045, 0.042 and 0.039 kgkg^{-1} for 'lafun' enriched with curd, 0.065, 0.042, 0.039 kgkg^{-1} for 'lafun' commercial sample 0.081, 0.066 and 0.061 kgkg^{-1} for 'lafun' enriched with residue at 10°C, 30°C and 40°C. The monolayer moisture content (M_0) is a measure of the moisture content for maximum stability of a food material (Crettenden and Watbon, 2012). This value indicates the maximum amount of water that could be adsorbed in a single layer per gram of dry film and it is an indicator of the number of absorption sites (Othman, 2014). The importance of GAB and BET models has always been used in determining the physiochemical explanations of their parameters (Igbabul *et al.*, 2013). The monolayer moisture content is a parameter used in explaining GAB and BET model (Goula *et al.*, 2007). As the monolayer temperature increased from 10°C to 40°C, the monolayer content of GAB decreased. This agreed with Oluwamukomi

(2009) for soy-melon enriched and control gari using GAB equations. The decreased in monolayer moisture content (M_0) with increased in temperature was an indication that the absorbed molecules gained kinetic energy making the attractive forces to be loosened and this allowed some water molecules to break away from their sorption sites thus decreasing the equilibrium moisture values (Arevalo-Pinedo *et al.*, 2004). The values of monolayer moisture content obtained from this study are in the range of some values reported for cassava products such as tapioca (0.049-0.058, "fufu" (0.043-0.049 by Kuye and Sanni (2002). Oluwamukomi (2009) reported monolayer moisture content of 0.050-0.080 for control "gari" 0.023-0.044 for soy-melon "gari". The values are within the ranged reported by Okewale *et al.* (2013) for adsorption isotherms of starchy adsorbents on uptake of water from ethanol – water systems. The monolayer moisture content were less than 0.1 kgkg^{-1} in all the products which was the maximum value reported for food materials (Oluwamukomi, 2009).

Conclusion

The investigation of sorption isotherm of lafun supplemented with soy curd and residue at temperatures 10, 30 and 40 °C unveiled sigmoid shape type II typical for most food products. The equilibrium moisture contents for supplemented lafun increased with increase in water activity. At low water activity, very small amount of water was absorbed into the active site but at high water activity, much more water was absorbed leading to rapid increase in equilibrium moisture content. Based on R^2 , RSS and SEE the five models were evaluated and the best fit was determined. Oswin and Henderson models best fits for the three temperatures for commercial lafun. 'Lafun' enriched with soy curd, lafun control and lafun with residue showed Oswin model gave the best fit.

Table 1: Estimated values for fitting models and model evaluation indicators of commercial 'lafun' sample at 10°C, 30°C and 40°C

MODELS	N	K	C	$M_0 10^0\text{C}$	SEE	R^2	RSS
OSWIN	0.21416		0.14371		0.000708	0.84855	0.0016
HASLEY	1.9999		0.07522		0.07502	0.6260	0.03506
HENDERSON	2.7633		2.7633		0.05886	0.74350	0.02499
BET			14.539	0.057	0.01998	0.8996	0.00766
GAB		0.1221	14.117	0.065	0.02489	0.8997	0.00766
				30°C			
OSWIN	0.20980		0.12960		0.00585	0.85990	0.00118
HASLEY	1.9999		0.06753		0.06815	0.63155	0.02860
HENDERSON	2.9168		2.9168		0.0536	0.75323	0.02050
BET			6.1414	0.038	0.0190	0.9064	0.0064
GAB		0.1290	18.716	0.042	0.02298	0.9071	0.00642
				40°C			
OSWIN	0.24220		0.11876		0.00045	0.94171	0.00499
HASLEY	1.9999		0.06505		0.05834	0.78545	0.02054
HENDERSON	2.9870		2.9870		0.00355	0.8714	0.0148
BET			8.0374	0.025	0.01486	0.9775	0.00372
GAB		0.16069	12.7406	0.039	0.0183	0.9775	0.00372

a, b, c, n and k are the model constants; RSS = residual sum of squares; SEE= the standard error of estimate and R^2 the co-efficient of fit.

Table 2: Estimated values for fitting models and model evaluation indicators of ‘lafun’ enriched with curd at 10°C, 30°C and 40°C

MODELS	N	K	C	Mo10°C	SEE	R ²	RSS
OSWIN	0.2685		0.1320		0.00087	0.8864	0.00163
HASLEY	1.9999		0.0746		0.24690	0.7239	0.02488
HENDERSON	2.7890		2.7890		0.04679	0.8263	0.01648
BET			8.899	0.036	0.03027	0.9116	0.00633
GAB		0.4571	27.765	0.0452	0.0167	0.94056	0.00373
30°C							
OSWIN	0.26555		0.1126		0.00061	0.88608	0.00143
HASLEY	1.9999		0.0634		0.0529	0.7354	0.01815
HENDERSON	3.0249		3.0249		0.0403	0.8334	-0.0120
BET			10.052	0.039	0.02383	0.9161	0.00431
GAB			17.650	0.0421	0.0146	0.9361	0.00289
40°C							
OSWIN	0.2614		0.0974		0.00036	0.94104	0.00396
HASLEY	1.9999		0.0548		0.0460	0.8094	0.0129
HENDERSON	3.2598		3.2598		0.0353	0.8955	0.0087
BET			3.250	0.031	0.0267	0.9436	0.00483
GAB		0.2581	3.820	0.039	0.0137	0.9744	0.0021

a, b, c, n and k are the model constants; RSS = residual sum of squares; SEE= the standard error of estimate and R² the co-efficient of fit

Table 3: Estimated values for fitting models and model evaluation indicators of ‘lafun’ control at 10°C, 30°C and 40°C

MODELS	N	K	C	Mo10°C	SEE	R ²	RSS
OSWIN	0.22307		0.1696		0.00094	0.8469	0.00258
HASLEY	1.9999		0.0897		0.08706	0.6299	0.0478
HENDERSON	2.5306		2.5306		0.0527	0.74629	0.03377
BET			0.7214	0.040	0.0288	0.9003	0.0099
GAB		0.7968	146.00	0.068	0.02888	0.90032	0.00996
30°C							
OSWIN	0.21729		0.1411		0.00073	0.85171	0.00162
HASLEY	1.9998		0.0741		0.07321	0.6306	0.03551
HENDERSON	2.7838		2.7838		0.05733	0.7475	0.0237
BET			0.4866	0.036	0.02450	0.90301	0.00718
GAB		0.8260	52.440	0.063	0.02450	0.90309	0.00718
40°C							
OSWIN	0.2461		0.1247		0.00053	0.93362	0.00065
HASLEY	1.9999		0.0686		0.0608	0.7735	0.0255
HENDERSON	1.59284		5.3094		0.04711	0.8698	0.0154
BET			0.2394	0.034	0.01896	0.9729	0.00394
GAB		0.0860	12.663	0.0529	0.01896	0.97295	0.00394

a, b, c, n and k are the model constants; RSS = residual sum of squares; SEE= the standard error of estimate and R² the co-efficient of fit

Table 4: Estimated values for fitting models and model evaluation indicators of ‘lafun’ enriched with residue at 10°C, 30°C and 40°C

MODELS	N	K	C	Mo10°C	SEE	R ²	RSS
OSWIN	0.2421		0.1597		0.0009	0.8976	0.00172
HASLEY	1.9999		0.0871		0.0785	0.71139	0.03875
HENDERSON	2.5766		2.5766		0.0607	0.8144	0.0267
BET			7.7281	0.030	0.0346	0.9401	0.0069
GAB		0.1482	9.9923	0.081	0.0237	0.9401	0.0069
30°C							
OSWIN	0.8582		0.6578		0.0001	0.9999	1.0*10 ⁻⁵
HASLEY	1.9999		0.0764		0.0053	0.7408	0.0270
HENDERSON	2.7556		2.7556		0.0499	0.8392	0.01821
BET			4.4950	0.036	0.0183	0.95419	0.00426
GAB		0.1507	8.6529	0.066	0.0270	0.9541	0.00426
40°C							
OSWIN	0.8711		0.6741		0.0001	0.9994	6.9*10 ⁻⁶
HASLEY	0.5999		0.5210		0.0595	0.7248	0.02278
HENDERSON	2.8757		2.8757		0.0454	0.0153	0.0153
BET			9.335	0.029	0.0167	0.9443	0.0035
GAB		0.1672	8.410	0.061	0.0165	0.9445	0.00357

a, b, c, n and k are the model constants; RSS = residual sum of squares; SEE= the standard error of estimate and R² the co-efficient of fit

Competing interests

The authors report no conflicts of interest.

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