Modelling and Absorption Isotherm of Sorghum (*Sorghum bicolor*), Soybean (*Glycine max*) and Orange Fleshed Sweet Potato (*Impomea batatas*) Flours Blends Infant Formula

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Abstract: The study aimed to establish the storage stability of flour blends from fermented and unfermented sorghum, soybean and Orange Fleshed Sweet Potato (OFSP) at 25, 30 and 40. The flours blends [F2: Sorghum: Soybeans: OFSP (56:17:27) %; F3: Sorghum: Soybeans: OFSP (59:31:10) %; UF2: Sorghum: Soybeans: OFSP (56:17:27) %; UF3: Sorghum: Soybeans: OFSP (59:31:10) %; were generated using mixture design of Response Surface Methodology (RSM). The Equilibrium Moisture Content (EMC) of the blends and Control (CT) sample were determined by static gravimetric method. The EMC were calculated and moisture sorption Isotherms were plotted for the dried samples. The monolayer Moisture (M_0) content of the samples was evaluated at each temperature by applying BET (Brunaeur-Emmett-Teller) and GAB (Guggenheim-Anderson-De Boer) equations to the isotherm data and the experimental data were fitted to four commonly used models using linear regression analytical procedure. EMC of flour blends decreased with an increase in temperature at constant water Activity (A_w) and increased with an increase in Relative Humidity (RH) at constant temperature. The sorption isotherm curves of the blends and CT were sigmodal in shape. GAB, BET and Oswin models gave a better fit for sample F2, Oswin and Henderson gave a better fit for sample UF2, Oswin gave a better fit for sample F3. Oswinand GAB gave a better fit for sample UF3 while Henderson and GAB gave a better fit for sample CT at temperatures 25, 30 and 40°C, respectively.

INTRODUCTION

One of the best methods of food preservation is removal of free water (moisture) from food sample, so as to reduce microbial activities which invariably help in increasing food product shelf life. The quality of most foods preserved by drying depends to a great extent upon their physical, chemical and microbiological stability.

This stability is mainly a consequence of the relationship between the EMC of the food material and its correspondence A_w, at a given temperature.

These water sorption isotherms are unique for individual food materials and can be used directly to solve food processing design problems, predict energy requirement and to determine proper storage conditions[1]. Water sorption isotherm equations can be used to predict water sorption properties of foods. There are many empirical and semi-empirical equations describing the sorption characteristics of foods. Sorption Isotherm precise shape is influenced by physical structure, chemical composition and extent of water binding within the food[2-4].

In most developed societies, nutrient-fortified cereals are the first complementary foods introduced to the infant. However, in developing countries like Nigeria, although, a number of convenient fortified proprietary formulas are available, they are often too expensive and out of the reach of most families. The use of home-based or traditional complementary foods that can be readily prepared that are available and affordable is one feeding option. The composition of local complementary foods varies from place to place for instance; traditional complementary food from fermented maize or sorghum (*akamu* or *ogi*) and fermented soybeans are commonly used in Nigeria.

The problem of the traditional complementary foods becoming unacceptable within a short period is usually a source of worry to mothers and caregivers, however, this could be solved by determining the proper storage conditions of food products. Therefore, this study aimed at evaluating the optimum moisture level for storage stability of flour blends of fermented and unfermented sorghum, soybeans and Orange Fleshed Sweet Potato (OFSP) which can be used as substitute for locally produced complementary food, so as to be able to predict its storage life at 25, 30 and 40.

MATERIALS AND METHODS

Samples preparation

Sorghum flour production: Sorghum grains were processed into flour using the method described by Adebayo-Oyetoro *et al*.[5]. Grains were sorted, cleaned manually to remove broken seeds, dust and other extraneous materials. A portion of it was fermented for 72 h and the other was not fermented, both were further processed separately. The samples were oven dried at 60°C for 8 h and milled into flour using laboratory hammer milling machine (Fritsch, D-55743, Idar-oberstein-Germany), sieved (using the 250 μm screen) and packaged for further analysis.

Soybeans flour production: Soybeans were processed into flour using the method described by Osundahunsi[6] with slight modification. The soybeans were sorted to remove dirt and extraneous materials, dehulled, washed and drained. A portion of it was fermented for 72 h and the other was not fermented, both were further processed separately. The samples were oven dried at 60°C for 8 h and milled into flour using laboratory hammer milling machine (Fritsch, D-55743, Idar-oberstein-Germany), sieved (using the 250 μm screen) and packaged for further analysis.

OFSP flour production: OFSP were processed into flour using the method described by Obiakor-Okeke *et al*.[7] with slight modification. The tubers were washed thoroughly with clean water, peeled and sliced into 2 mm using electric slicer and immersed into 0.25% sodium metabisulphite for 5 min to prevent browning reactions and to enhance the colour of the flour. A portion of it was fermented for 72 h and the other was not fermented, both were further processed separately. The samples were oven dried at 60°C for 8 h and milled into flour using laboratory hammer milling machine (Fritsch, D-55743, Idar-oberstein-Germany), sieved (using the 250 μm screen) and packaged for further analysis.

Formulation of experimental flour blends: Sorghum, soybeans and OFSP flours were blend together using mixture design of Response Surface Methodology (Design expert Version 10.0). The following combinations were thereafter obtained, that is F2: Sorghum: Soybeans: OFSP (56:17:27) %; F3: Sorghum: Soybeans: OFSP (59:31:10) %; UF2: Sorghum: Soybeans: OFSP (56:17:27) %; UF3: Sorghum: Soybeans: OFSP (59:31:10) %; for fermented and unfermented samples, respectively. And a commercial sample (CT) was used a control sample.

Chemical analysis

Adsorption isotherm determination: Absorption isotherm of experimental flour blends was determined using a static gravimetric method. About 2.0 g of experimental flour blends (CT; UF2; UF4; F2 and F4) were separately placed in each petri dishes inside 5 different desiccators containing saturated salt solutions (LiCl, NaCl, KCl, $Mg(NO_3)_2.6H_2O$, $MgCl_2$) providing constant relative humidity environments ranging from 11.30-84.34% in desiccators as described by Onayemi and Oluwamukomi[8] and Rockland[9]. The desiccators containing salt solutions and experimental samples were placed inside temperature controlled Gallenkamp DV 400 incubators which were set at 25, 30 and 40°C. The temperatures were monitored to within ±1·0°C The samples were weighed daily using a Mettler PC 2000 electronic balance with an accuracy of 0·001 g.

Equilibrium was considered to have been attained when three identical consecutive measurements were obtained. The dry matter content was produced by oven drying at 105±1·0°C for 72 h. The EMC were calculated on dry basis from which the moisture sorption isotherm was determined. The thermodynamic characteristics of experimental samples were analytically fitted to five commonly used models using the non-linear regression procedure in Statistical Package for Social Science (Version 21·0 for Windows). Models used includes; GAB (Guggenheim-Anderson-De Boer)[10], BET (Brunaeur-Emmett-Teller)[11-14].

RESULTS AND DISCUSSION

Sorption isotherm behaviour: The adsorption isotherm in this present study was determined by plotting the equilibrium moisture content against different water activities, the sorption isotherm curve is similar to that reported by Ramanathan *et al*.[15] for compacted flour, Menkov and Durakova[16] for sesame and walnut flour, Oyelade[4] for lafun, Nurtama and Lin[17] for taro flour, Famurewa *et al*.[18] for pupuru. Figure 1-5 shows curves of the EMC against the range ofwater activities expressed on moisture free basis to each relative humidity at 25, 30 m and 40°C for UF2, UF3, F2, F3 and CT. At increased temperatures water molecules get activated to higher energy levels, causing them to become less stable and break away from the water binding sites of the material, thus decreasing EMC[19]. The sorption isotherm behaviour of the formulated flour blends and control sample have a Sigmoidal shape profile (a type II isotherm according to Brunauer's classification and correspond to multilayer formulation as observed by Kumar[20], Famurewa *et al*.[18] and Osundahunsi[6] that is typical of isotherms of products high in starch content. Previous research documented that the effect of temperature on the sorption isotherm is generally of great importance given that foods are exposed to a range of temperatures during processing and storage[21,22]. Temperature increase leads to water activity (a_w) increase at the same moisture content which in turn causes an increase in the reaction rates leading to quality deterioration. This however, suggests that fluctuations in temperature and relative humidity will greatly have significant effect on storage stability of the samples[18]. The samples adsorbed minimal amount of water at a region of a_w 0.0-0.30 where the moisture is unavailable for reactions (monolayer adsorption). Visible mould growth was observed in some of the samples at 0.85 a_w after two weeks, this agree with the report by Osundahunsi[6] who reported that some samples of native cassava starch beyond 0.75 a_w were discarded.

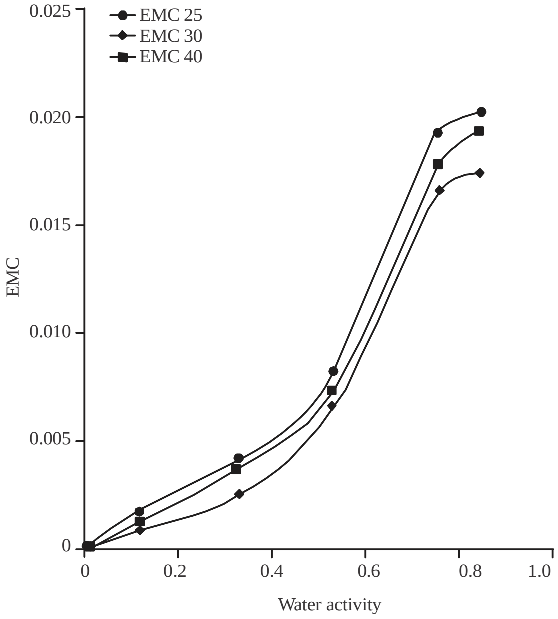

Fig. 1: Sorption isotherm Curve for F2; F2-Fermented [sorghum: soybeans: OFSP (56: 17:27) %]; EMC: Equilibrium Moisture Content

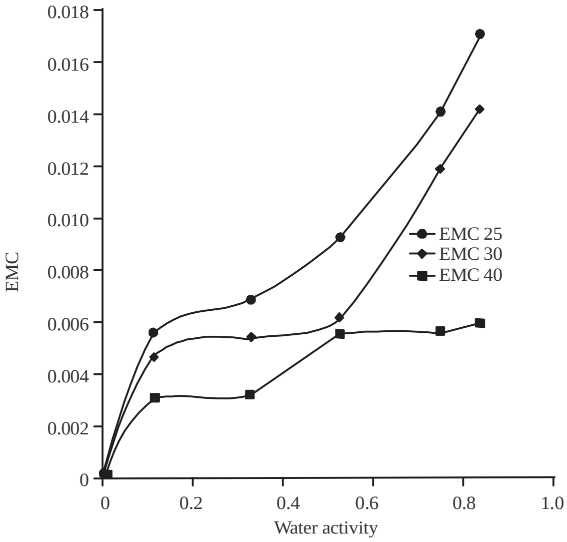

Fig. 2: Sorption Isotherm Curve for UF2; UF2-Unfermented [Sorghum: soybeans: OFSP(56: 17: 27) %]; EMC: Equilibrium Moisture Content

Fitting of experimental data to sorption isotherm models: The result of linear regression analysis fitting calculated using model equation (Table 1) of the experimental data are presented in Table 2-6 for F2, UF2, F3, UF3 and CT, respectively.

Five sorption models, GAB, BET, Oswin, Hasley and Henderson were used to explain the behaviour of the

Table 1: Isotherm models for fitting experimental data

Models	Equations	References
GAB	X_{eq} = MoCkaw/(1-Kaw (1-Kaw+CKaw)	Andrade *et al.*[23]
BET	Xeq = Cmoaw(1-aw) (1+(C-1)aw)	Andrade *et al.*[23]
Oswin	Xeq = C[aw/1-aw]^{n}	Vega-Galvez *et al.*[24]
Halsey	Xeq = (-aw/1-aw)$^{1/n}$	Andrade *et al.*[23]
Henderson	Xeq = (-In (1-aw/c))$^{1/n}$	Andrade *et al.*[23]

Table 2: F2 parameter values obtained for the models

Models	25°C	30°C	40°C
GAB			
K	0.0388	0.03314	0.0159
C	7.5973	7.5358	8.0978
m_o	0.0530	0.0536	0.0570
RSS	1.52x10E^{-5}	1.28x10E^{-5}	8.70×0E-6
SEE	3.18x10E^{-6}	3.20x10E^{-6}	2.17×10E-6
R^2	0.0954	0.8907	0.8507
BET			
C	0.4169	0.2219	0.2133
m_o	0.0302	0.0490	0.0455
RSS	1.96×10E^{-5}	1.40×10E^{-5}	3.68×10E-6
SEE	9.82×10E^{-6}	7.00×10E^{-6}	1.82×10E-6
R^2	0.9041	0.9944	0.8140
OSWIN			
C	0.0094	0.0074	0.0045
N	0.0350	0.3920	0.1949
RSS	1.48×10E^{-6}	4.11×10E^{-6}	1.38×10E-6
SEE	3.72×10E^{-7}	1.02×10E^{-6}	3.47×10E-7
R^2	0.9842	0.9459	0.8233
Hasley			
C	0.0013	0.0272	0.000024
N	0.3929	9.3133	0.1600
RSS	5.56×10E^{-5}	2.93×10E^{-5}	2.35×10E-5
SEE	1.41×10E^{-5}	7.32×10E^{-6}	5.88×10E-6
R^2	0.9683	0.9667	0.6665
Henderson			
C	0.943	10.282	19.073
N	105.62	11.669	11.444
RSS	3.14x10E^{-5}	1.40×10E-5	3.68×10E-5
SEE	27.86x10E^{-6}	4.64×10E^{-6}	4.59×10E^{-6}
R^2	0.9971	0.9644	0.7746

F2 = Fermented [sorghum:soybeans: OFSP (56: 17:27) %]; N, C and K = Model constants; RSS = Residual Sum of Squares; SEE = Standard Error of Estimate; R^2 = Co-efficient of fit

Table 3: UF2Parameter values obtained for the models

Models	25°C	30°C	40°C
GAB			
K	0.0903	0.0444	0.03632
C	7.5980	8.6574	16.2349
m_o	0.0538	0.0841	0.04996
RSS	6.45×10 E^{-4}	2.51×10 E^{-4}	2.05×10E-4
SEE	1.61×10 E^{-4}	6.27×10 E^{-5}	5.13×10E-5
R^2	0.9054	0.8038	0.83494
BET			
C	0.7116	0.9856	0.7324
m_o	0.061	0.056	0.045
RSS	3.65×10E^{-4}	1.15×10E^{-3}	7.92×10E-5
SEE	9.82×10E^{-4}	7.00×10E^{-4}	1.82×10E-5
R^2	0.9145	0.7261	0.9104
Oswin			
C	0.3314	0.0234	0.0222
N	0.1799	0.2466	0.2815
RSS	2.36×10E^{-5}	5.11×10E^{-5}	4.92×10E-5
SEE	5.89×10E^{-6}	1.27×10E^{-5}	1.23×10E-5
R^2	0.9325	0.8482	0.8687
Hasley			
C	0.0048	0.0028	0.0018
N	0.4946	0.3537	0.2261
RSS	1.39×10E^{-3}	5.28×10E^{-4}	4.11×10E-4
SEE	3.48×10E^{-4}	1.32×10E^{-4}	1.03×10E-4
R^2	0.7049	0.8027	0.8048
Henderson			
C	1.2294	1.7310	1.4307
N	27.8171	25.7722	31.1799
RSS	9.65×10E^{-4}	3.96x10E^{-4}	3.13×10E-4
SEE	2.41×10E^{-4}	9.91×10E^{-5}	7.82×10E-5
R^2	0.8373	0.8107	0.8281

UF2 = Unfermented [Sorghum: soybeans: OFSP(56: 17: 27) %]; N, C and K = Model constants; RSS = Residual Sum of Squares; SEE = Standard Error of Estimate; R^2 = Co-efficient of fit

formulated blends overa_w range of 0.1-0.9 at different temperatures (25, 30 and 40°C). The corresponding Residual Sum of Squares (RSS); Standard Error of Estimate (SEE); Co-efficient of fit (R^2) and model constants (K, C, N) are shown in Table 2-6. A model is considered better than another if it has a low SEE, low RSS and a strong R^2 that is highest value (near unity)[25]. However, for this present study high R^2 was prioritized in the choice of the best model. The predicted M_o of GAB and BET models were of particular importance because M_o indicates the amount of water that is strongly adsorbed in specific sites and is considered to be the value at which a food is most stable that is the optimal moisture content that minimizes spoilage reactions especially during storage[23].

In Table 2 (F2) the Henderson model gave the best satisfactory fit to the experimental data at 25°C having a high R^2 of 0.9971 while at 30°C, BET. Model gave a satisfactory fit having high R^2 of 0.9944 while at 40°C and GAB Model gave the best satisfactory fit with high R^2 of 0.8507. The M_o of GAB and BET Models at 25°C were 0.0530 and 0.0302, 30°C (0.0536 and 0.0490) and 40°C (0.0570 and 0.0455), respectively.

In Table 3 (UF2) the Oswin model gave the best satisfactory fit to the experimental data at 25°C having a high R^2 of 0.93248 while at 30°C, Oswin model gave a satisfactory fit having high R^2 of 0.8482 while at 40°C, BET model gave the best satisfactory fit with high R^2 of 0.9104. The M_o of GAB and BET Models at 25°C were 0.0538 and 0.061, 30°C (0.0841 and 0.056) and 40°C (0.049 and 0.045), respectively.

It was observed in Table 4 (F3) that Oswin model gave the best satisfactory fit to the experimental data at 25°C having a high R^2 of 0.8478 while at 30°C, Oswin model gave a satisfactory fit having high R^2 of 0.8130 while at 40°C, Oswin Model gave the best satisfactory fit

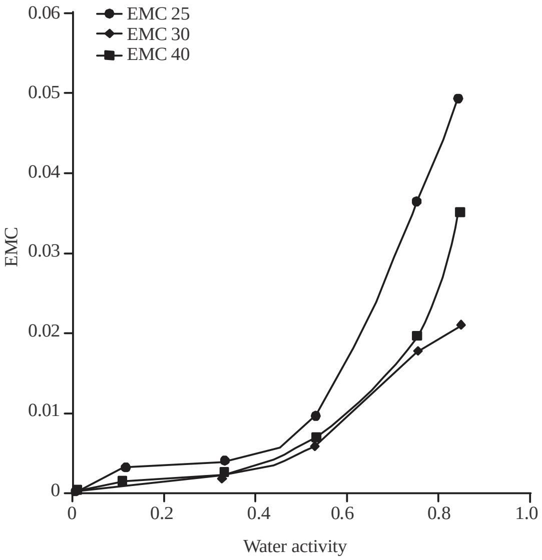

Fig. 3: Sorption Isotherm Curve for F3; F3-Fermented [Sorghum: soybeans: OFSP (59:31:10 %]; EMC: Equilibrium Moisture Content

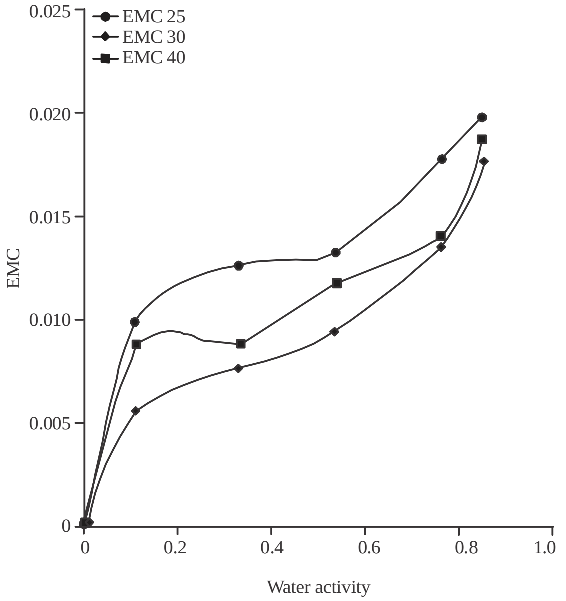

Fig. 4: Sorption Isotherm Curve for UF3; UF3-Unfermented [Sorghum: Soybeans: OFSP (59: 31: 10%)]; EMC: Equilibrium Moisture Content

with high R^2 of 0.8855. The M_o of GAB and BET Models at 25°C were 0.021 and 0.010, 30°C (0.0179 and 0.024) and 40°C (0.0580 and 0.016), respectively.

In Table 5 (UF3), Oswin Model gave the best satisfactory fit to the experimental data at 25°C having a high R^2 of 0.992 while at 30°C, Oswin Model gave a satisfactory fit having high R^2 of 0.9650 while at 40°C

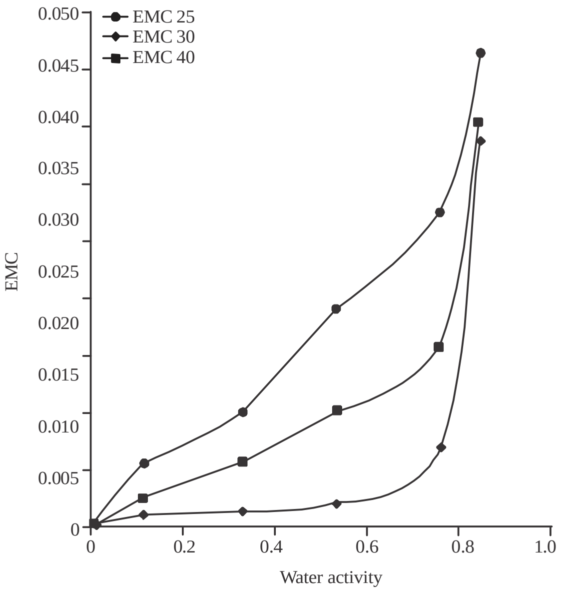

Fig. 5: Sorption Isotherm Curve for CT; CT [Commercial sample-Cerelac]; EMC: Equilibrium Moisture Content

Table 4: F3 parameter values obtained for the models

Models	25°C	30°C	40°C
GAB			
K	0.0069	0.0232	0.0060
C	122.705	42.99	49.566
m_o	0.021	0.0179	0.0580
RSS	$3.20\times10E^{-5}$	$1.26\times10E^{-5}$	2.83×10E-6
SEE	$1.06\times10E^{-5}$	$4.23\times10E^{-6}$	8.63×10E-6
R^2	0.5506	0.7749	0.8271
BET			
C	0.4760	0.3854	0.8759
m_o	0.010	0.024	0.016
RSS	$8.67\times10E^{-5}$	$2.67\times10E^{-5}$	1.51×10E-6
SEE	$2.89\times10E^{-6}$	$8.91\times10E^{-6}$	3.83×10E-6
R^2	0.4537	0.6876	0.8256
Oswin			
C	0.0088	0.0053	0.0073
N	0.225	0.914	0.3285
RSS	$1.84\times10E^{-5}$	$9.80\times10E^{-5}$	1.33×10E-5
SEE	$3.69\times10E^{-6}$	$1.96\times10E^{-5}$	2.26×10E-6
R^2	0.8478	0.8130	0.8855
Hasley			
C	0.0029	0.00066	0.0011
N	1.0371	0.0156	0.4056
RSS	$7.52\times10E^{-5}$	$8.22\times10E^{-5}$	4.28×10E-5
SEE	$1.88\times10E^{-5}$	$2.05\times10E^{-5}$	$1.07\times10E^{-5}$
R^2	0.8402	0.7228	0.8579
Henderson			
C	1.099	4.963	7.616
N	10.99	16.93	16.46
RSS	$6.87\times10E^{-5}$	$1.24\times10E^{-5}$	3.74×10E-5
SEE	$1.37\times10E^{-6}$	$2.56\times10E^{-6}$	$7.48\times10E^{-6}$
R^2	0.7103	0.7228	0.8068

F3 = Fermented [Sorghum: soybeans: OFSP(59:31:10 %]; N, C and K = model constants; RSS = Residual Sum of Squares; SEE= Standard Error of Estimate; R^2= Co-efficient of fit

Table 5: UF3 parameter values obtained for the models

Models	25°C	30°C	40°C
GAB			
K	0.099	0.025	0.0131
C	59.268	48.751	85.11
m_o	0.052	0.021	0.050
RSS	$5.71\times10E^{-5}$	$3.725\times10E^{-5}$	1.19×10E-5
SEE	$1.90\times10E^{-5}$	$1.24\times10E^{-5}$	3.96×10E-6
R^2	0.6897	0.7017	0.8511
BET			
C	0.219	0.431	0.3495
m_o	0.012	0.0151	0.048
RSS	$8.67\times10E^{-5}$	$2.67x10E^{-5}$	1.51×10E-6
SEE	$2.89\times10E^{-6}$	$8.91x10E^{-6}$	3.83×10E-6
R^2	0.6890	0.6563	0.8437
Oswin			
C	0.014	0.0118	0.0099
N	0.1937	0.232	0.3386
RSS	$1.91\times10E^{-6}$	$6.78\times10E^{-6}$	1.84×10E-6
SEE	$3.83\times10E^{-7}$	$1.35\times10E^{-6}$	$3.68\times10E^{-7}$
R^2	0.9921	0.9650	0.9902
Hasley			
C	0.00017	0.0044	0.00089
N	0.004	1.1339	0.2313
RSS	$2.22\times10E^{-4}$	$1.24\times10E^{-4}$	5.59×10E-5
SEE	$4.40\times10E^{-6}$	$2.48\times10E^{-5}$	1.09×10E-5
R^2	0.6743	0.777	0.8942
Henderson			
C	6.406	7.610	7.539
N	12.306	11.704	12.408
RSS	$1.5\times10E^{-4}$	$9.5\times10E^{-5}$	$3.91\times10E^{-5}$
SEE	$3.01\times10E^{-6}$	$1.9\times10E^{-5}$	$7.42\times10E^{-6}$
R^2	0.7591	0.8056	0.9181

UF3 = Unfermented [Sorghum: soybeans: OFSP (59: 31: 10%)]; N, C and K = Model constants; RSS = Residual Sum of Squares; SEE= Standard Error of Estimate; R^2 = Co-efficient of fit

and Oswin Model gave the best satisfactory fit with high R^2 of 0.9902. The M_o of GAB and BET Models at 25°C were 0.052 and 0.012, 30°C (0.021 and 0.0151) and 40°C (0.050 and 0.048), respectively.

In Table 6 (CT), Oswin Model gave the best satisfactory fit to the experimental data at 25°C having a high R^2 of 0.9914 while at 30 °C, BET Model gave a satisfactory fit having high R^2 of 0.9859 while at 40°C, and Oswin Model gave the best satisfactory fit with high R^2 of 0.9865. The M_o of GAB and BET Models at 25°C were 0.029 and 0.027, 30°C (0.0188 and 0.016) and 40°C (0.078 and 0.011), respectively.

More than one model has been reported to describe sorption characteristics of foods[26]. Akanbi *et al.*[27] reported Oswin and GAB as the best models that described the sorption isotherm of dehydrated tomato slices at 25, 30 and 40°C while Vega-Galvez *et al.*[24] reported Smith and Henderson models as the best among the eight models tried for modeling of the adsorption isotherm of *Chilean papaya* at 5°C. However, in the present study, more than one model (GAB, BET, Oswin and Henderson) were presented the best fit for the description of the moisture adsorption isotherm of the blends and the control samples.

Table 6: CT(*Cerelac*) parameter values obtained for the models

Models	25°C	30°C	40°C
GAB			
K	0.0042	0.876	0.0093
C	54.98	121.55	31.248
m_o	0.029	0.0188	0.078
RSS	$6.84\times10E^{-6}$	$2.51\times10E^{-6}$	3.77×10E-7
SEE	$2.28\times10E^{-6}$	$8.36\times10E^{-7}$	$1.88\times10E^{-7}$
R^2	0.9793	0.9768	0.9378
BET			
C	0.1178	0.6398	0.688
m_o	0.027	0.016	0.011
RSS	$8.83\times10E^{-6}$	$1.08\times10E^{-5}$	1.94×10E-5
SEE	$2.94\times10E^{-6}$	$3.61\times10E^{-6}$	9.71×10E-6
R^2	0.9796	0.9859	0.9849
Oswin			
C	0.0179	0.0080	0.000384
N	0.549	0.921	0.2732
RSS	$1.25\times10E^{-6}$	$4.87\times10E^{-6}$	1.40×10E-6
SEE	$2.51\times10E^{-7}$	$9.75\times10E^{-6}$	2.93×10E-7
R^2	0.9914	0.9585	0.9865
Hasley			
C	0.0035	0.0028	0.00003
N	0.425	0.444	0.0006
RSS	$9.21\times10E^{-5}$	$4.06\times10E^{-5}$	2.5×10E-4
SEE	$2.30\times10E^{-5}$	$1.01\times10E^{-5}$	6.4×10E-5
R^2	0.9717	0.9548	0.8140
Henderson			
C	0.9296	3.5237	0.277
N	44.121	15.632	25.455
RSS	$3.38\times10E^{-5}$	$1.08\times10E^{-4}$	$3.71\times10E^{-4}$
SEE	$6.77\times10E^{-6}$	$2.17\times10E^{-5}$	$9.28\times10E^{-5}$
R^2	0.9832	0.8997	0.6838

CT [Commercial sample-*Cerelac*]; N, C and K = model constants; RSS = Residual Sum of Squares; SEE= Standard Error of Estimate; R^2= Co-efficient of fit

CONCLUSION

The result of this study established that adsorption isotherms provided valuable information about the EMC and it presented a clear idea on the storage stability of these flours. The experimental results showed that the adsorption isotherms of the control sample and the formulated flour blends at the three temperatures were characterized by a sigmoid shape (curve typical of the type II classification shape). The GAB, BET and Oswin Models gave a better fit for sample F2 while Oswin and Henderson gave a better fit for sample UF2 meanwhile, Oswin gave a better fit for sample F3. However, Oswin, GAB gave a better fit for sample UF3 while Henderson and GAB gave a better fit for CT at temperatures 25, 30 and 40°C, respectively.

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