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ARTICLE

Studies on Moisture Adsorption Isotherm of *Ighu* from Different Cassava Varieties and Accelerated Shelf Life Storage in Different Packaging Materials

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ABSTRACT

Moisture sorption isotherm characteristics and shelf life of *Ighu*, a traditional food product derived from three cassava varieties processed using two indigenous methods in Isuochi (Umunneochi LGA, Abia State) and Udi (Enugu State), Nigeria was studied. The equilibrium moisture content (EMC) was determined using the static gravimetric method across water activity (aw) levels of 0.08 to 0.1 at different temperatures. Experimental data were fitted to four standard sorption models: Brunauer–Emmett–Teller (BET), Guggenheim–Anderson–de Boer (GAB), Oswin, and Halsey. The water vapour transmission rate (WVTR) and permeability coefficients of three packaging materials—low-density polyethylene (LDPE), high-density polyethylene (HDPE), and laminated nylon were also assessed at 30°C and relative humidity (RH) levels from 20.16% to 90.70%, using saturated salt solutions. Results showed a direct correlation between EMC and a_w, with sorption curves displaying Type II sigmoid behaviour. The BET model exhibited the best fit for the adsorption data, while laminated nylon presented the best barrier properties to moisture migration, with the lowest WVTR values (0.048–2.250 g H_2O/day/m²) compared to HDPE (0.240–2.391 g H_2O/day/m²) and LDPE (1.201–4.011 g H_2O/day/m²). *Ighu* stored in laminated nylon at 20–33% RH maintained a projected shelf life of up to 10 years compared to those from LDPE and HDPE. These findings underscore the significance of packaging material and environmental conditions in enhancing the storage stability of *Ighu*, supporting its long-term preservation and commercial viability.

Keywords: *Ighu*; Accelerated Shelf Life; Improved Cassava; Adsorption Isotherm; Equilibrium Moisture Content

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1. Introduction

Temperature, moisture, and surrounding relative humidity are known to impact the stability and quality of food products due to their influence on water activity (aw) [1,2]. Moisture sorption isotherms, which represent the equilibrium relationship between water activity and moisture content at constant temperature, are crucial tools in food preservation, packaging design, and quality assessment These isotherms vary with food type, processing technique, and temperature [2], and can be described by a variety of mathematical models—some mechanistic, others empirical or semi-empirical [3–5].

Since no universal model captures the sorption behavior of all food products, selecting an appropriate model depends on the specific characteristics of the food matrix [6]. For hygroscopic foods, moisture sorption data also reveal critical structural parameters such as surface area, porosity, and crystallinity [7], which are important for understanding deterioration mechanisms and optimizing storage conditions [8–11].

Ighu is one of the various edible products made from cassava tubers [12]. Unlike other products from cassava, *Ighu* is processed by boiling unpeeled cassava tubers for about 30–40 minutes depending on the quantity and rate of heat supply, cooling, peeling and shredding with a metallic shredder (*nko*) having many openings on it [13]. The thinly sliced shreds are sometimes spread in a rectangular-shaped wooden basket (called *nmimi* by Isuochi people of Abia State) for several hours to further reduce the cyanogenic glycoside in the tuber by washing and drying in the wooden baskets on an elevated platform referred to as "*nlugbu"*. It is also called *jigbo, mpataka, abacha, eberebe jiapu, asharasha, jiapu mmiri, nsisa, etc.* in different Igbo dialects [14]. This study aims to explore how these indigenous methods, along with relative humidity and packaging type, affect the moisture sorption behaviour and shelf life of *Ighu* made from improved cassava varieties.

2. Materials and Methods

2.1. Material

The stems of some improved cassava varieties obtained from National Root Crop Research Institute (NRCRI) Abia State including Tropical *Manihot esculenta* (TME 4779), Tropical *Manihot* specie (TMS 98/87164) and National Root (NR 8082) were cultivated and harvested at eight (8) months maturity, while all reagents used are of analytical grade.

2.2. Methods

2.2.1. Processing of *Ighu*

Processing of cassava tubers into *Ighu* was done as stated by Nwagbara and Iwe with slight moderation (**Figure 1**) [12]. Similar processing methods are used by indigenous peoples of Isuochi in Abia State and Udi in Enugu State Nigeria but with different shapes and openings in the metallic material used for shredding of cassava tuber called *nko* (**Figures 2** and **3**) resulting in *Ighu* with average diameters of 2 mm and 4 mm, respectively for Isuochi and Udi *Ighu* strands (**Figure 4**).

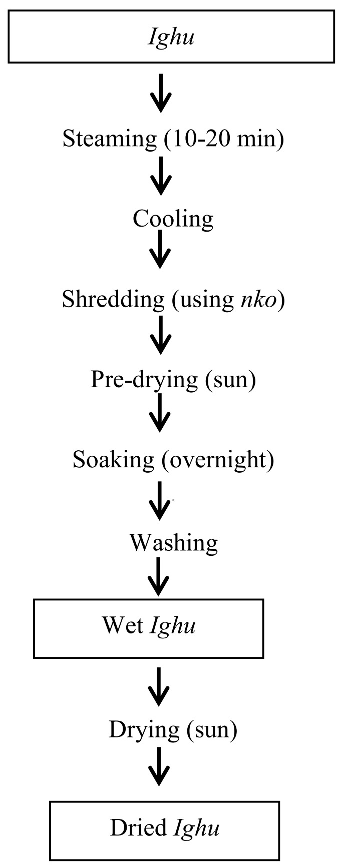

Figure 1. Flow Chart for *Ighu* Processing [12].

Figure 2. *Nko* from Udi Enugu State.

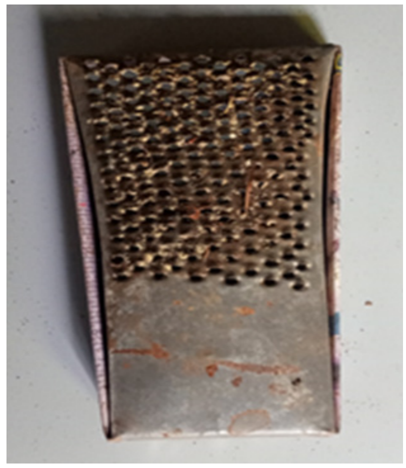

Figure 3. *Nko* from Isuochi Abia State.

Ighu from cassava variety NR8082 Isuochi method

Ighu from cassava variety TMS 98/87614 Isuochi method

Ighu from cassava variety TME 4779 Isuochi method

Ighu from cassava variety NR8082 Udi method

Ighu from cassava variety TMS 98/87614 Udi method

Ighu from cassava variety TME 4779 Udi method

Figure 4. Dried *Ighu* Samples.

2.2.2. Experimental Design for Moisture Sorption Isotherm Studies

The experiment was conducted based on general factorial design (GFD) where each factor can have a different number of levels. Factors considered were different varieties of cassava (3-levels), indigenous/local methods of processing cassava roots into *Ighu* (2-levels) and varying sorption temperature (3-levels). A total of eighteen (18) experimental runs tested at eight (8) different water activity levels were generated, while the analysis was conducted in duplicates. Experimental variables for processing of *Ighu* and moisture sorption modelling are presented in **Tables 1** and **2**.

Table 1. Process Variable for the Experimental Design.

Independent variables	K=3	Variable levels		
Cassava variety	X_1	i A	ii B	iii C
Ighu processing method	X_2	Isuochi	Udi	-
Sorption temperature	X_3	20	30	40

Where A, B and C represent cassava varieties TME 4779, TMS 98/87164 and NR 8082.

Table 2. Actual Variables of the Experimental Design.

Experimental run/factors	Cassava varieties	Processing method	Temperature
1	TMS 98/87614	ISUOCHI method	40 °C
2	TME 4779	ISUOCHI method	20 °C
3	NR 8082	UDI method	30 °C
4	TME 4779	ISUOCHI method	30 °C
5	TMS 98/87614	ISUOCHI method	20 °C
6	TMS 98/87614	ISUOCHI method	30 °C
7	NR 8082	ISUOCHI method	40 °C
8	TME 4779	UDI method	40 °C
9	TMS 98/87614	UDI method	40 °C
10	NR 8082	UDI method	20 °C
11	NR 8082	ISUOCHI method	30 °C
12	TMS 98/87614	UDI method	30 °C
13	TME 4779	UDI method	30 °C
14	TMS 98/87614	UDI method	20 °C
15	NR 8082	ISUOCHI method	20 °C
16	NR 8082	UDI method	40 °C
17	TME 4779	ISUOCHI method	40 °C
18	TME 4779	UDI method	20 °C

2.2.3. Moisture Sorption Isotherm Experiment

Moisture interaction with the samples was assessed under controlled humidity using a static gravimetric technique. To simulate various humidity conditions, sulfuric acid solutions (15–65%) corresponding to water activities ranging from 0.10 to 0.90 were used. Experiments were conducted at three distinct temperatures, 20°C, 30°C, and 40°C, while the *Ighu* samples (0.5 g each) were placed in tightly covered containers above the solutions and in a thermostatically controlled incubator. Sample weights were monitored every 12 hours until readings stabilized (less than 0.5% difference). The final moisture content was calculated on a dry-weight basis (Equation 1) after oven-drying samples at 105°C for 4 hours and cooling in a desiccator. The total time for removal and putting back in the airtight containers was about 1–2 minutes to reduce atmospheric adsorption occurring as recommended by the cooperative project, costing 90, as reported by Ojike *et al*. [15].

$$\text{EMC} = \frac{MW_1+100(W_3-W_2)}{W_1+(W_3-W_2)} \quad (1)$$

EMC = Equilibrium moisture content M = initial moisture content of the sample, W_1 = Weight of sample used during sorption, W_2 =Initial weight of sample plus crown cork, W_3= Final weight of sample plus crown cork at equilibrium

2.2.4. Modelling of Sorption Data

Experimental EMC and a_w values were fitted to four established isotherm models of BET, GAB, Halsey, and Oswin. These models were selected due to their proven application to carbohydrate-rich food matrices. The GAB as well as Oswin and Halsey models have been used to describe sorption isotherms of agro-foods within the water activity range of 0.00–0.95 as noted by Iguedjtal et al. [16], while BET has been used for water activity levels below 0.5 (a_w <0.5). Model parameters were derived using nonlinear regression analysis (SPSS v23.0) [17]. Goodness of fit between the experimental and predicted EMCs was assessed using coefficient of determination (R²) and root mean square error (RMSE) using

$$\textbf{BET} \quad \frac{M}{M_O} = \frac{Ca_w}{(1-a_w)[1+(C-1)a_w]} \quad (2)$$

$$\textbf{Or} \quad \frac{\boldsymbol{a_w}}{(\boldsymbol{1-a_w})\boldsymbol{M}} = \frac{1}{M_oC} + \frac{C-1}{(M_oC} a_w \quad (3)$$

$$\textbf{GAB} \quad \frac{CKa_w}{(1-Ka_w)(1-Ka_w+CKa_w)} \quad (4)$$

HALSEY $M = \left[\frac{exp\ AT+B}{-ln(a_w)}\right]^C$ (5)

OSWIN = $M = A\left[\frac{a_w}{1-a_w}\right]^B$ (6)

Where:
a_w = water activity
M_o = monolayer moisture content
A, B, C = model constants

2.2.5. Determination of Adsorption Surface Area (So)

Monolayer moisture content (Mo) from BET equation was used to estimate the adsorption surface area (So) with equation 7. The derived surface area offers insight into the food's moisture-binding potential.

$$So = \frac{A_o X N_o X M_o}{M_s} = 3530Mo \quad (7)$$

Constants include: Avogadro's number (N_o = 6.023×10^{23} molecules/mole),
Ms = molar mass of water (18 g/mol),
A = apparent surface area of one water molecule (1.05×$10^{-19}$$m^2$),
M_o = monolayer moisture content (g H_2O/100g solid),
S_o is the apparent area of sorption.

2.2.6. Determination of Water Vapour Permeability Coefficient of the Packaging Material

This was determined for packaging materials used in this work including LDPE, HDPE and laminated polyethylene/nylon at various storage conditions using the protocol of ASTM [18]. Salts of known saturation were used to maintain different ranges of relative humidity of 20.16 to 90.70. About 5 samples of the packaging materials were prepared with the addition of ten (10) grams of desiccant or silica gels into three of the five samples; the other two (2) packages without silica gel were used as controls (**Figure 5**) and were sealed using a band-sealer. The thickness and surface area of the materials were measured using a micrometre screw gauge and meter rule, respectively. The experiment was carried out in a controlled system which maintained temperature of 30 °C and varying range of relative humidity 20.16 to 90.70%. The materials were reweighed at day two intervals until a constant weight was achieved. The weight gains by the packaging materials during the experiment were subtracted from the weight gain of the control samples, and were plotted in a graph. The slope (weight gain vs time in days) of the graph was used to determine the water vapour transmission rate (WVTR) and permeability coefficient (P) of the packaging material using

$$WVTR = \frac{W}{tA} \quad (8)$$

$$P = \frac{W}{tA}\frac{X}{\Delta P} \text{ or } P = \frac{WVTR}{\Delta P} \quad (9)$$

where Q/t = slope, A = total surface area of both sides of package, X = thickness, Δp = partial pressure change, Po = saturated vapor pressure of pure water (30 °C) and RH = storage relative humidity.

Figure 5. Accelerated Shelf Life Testing Set-Up According to ASTM.

Note:

$$\Delta P = P_0 - P_i \quad (10)$$

According to ASTM E 96 Pi = 0 because vapor pressure inside the packaging is in equilibrium with the desiccant, thus:

$$\Delta P = \frac{P_s}{100}(RH) \quad (11)$$

2.2.7. Accelerated Shelf Life Prediction of *Ighu*

The prediction and simulation of the shelf life of packaged *Ighu* will follow the method stated by Kulchan *et al.* [19], as given in

$$t = \frac{GL}{AP\Delta P} \quad (12)$$

Where:
$G = d\,(M_C - M_0)$
d = mass of dry product (g)
M_C = critical moisture content (%)
M_O = initial moisture content (%)
Therefore;

$$t\ (\text{days}) = \frac{d(M_O - M_C)}{AP\Delta P} L \quad (13)$$

Where:
t = shelf life (days)
d = weight of dry product (g)
M_C = critical moisture content (%)
M_O = initial moisture content (%)
L = thickness of the packaging materials (mm)
A = Area (m^2)
P = Permeability coefficient ($gmmd^{-1}m^{-2}mmHg^{-1}$)
ΔP = vapour pressure difference (mmHg)

3. Results and Discussion

3.1. Effect of Processing Methods of *Ighu* on Water Activity and Equilibrium Moisture Content (EMC) at Different Temperatures

Results of the effect of water activity on equilibrium moisture content (EMC) of *Ighu* processed using two indigenous methods at different temperatures are presented in **Figures 6** and **11**.

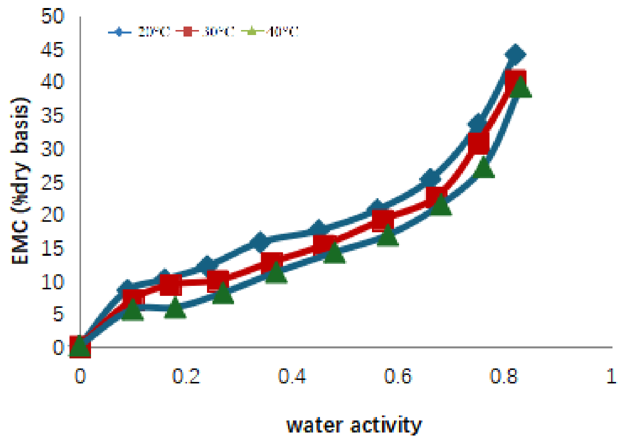

Figure 6. Effect of Water Activity on Equilibrium Moisture Content of *Ighu* from TME 4779 (Isuochi Method).

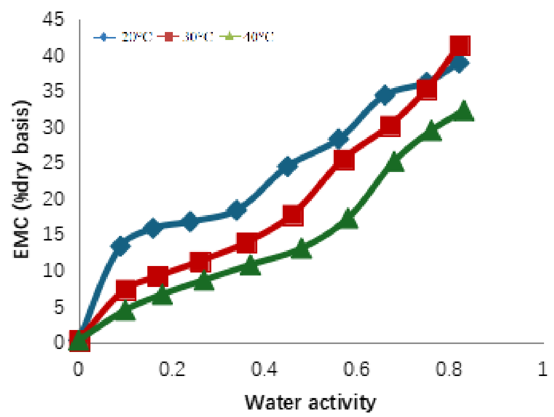

Figure 7. Effect of Water Activity on Equilibrum Moisture Content of *Ighu* from TME 4779 (Udi Method).

The results clearly showed adsorption isotherm behaviour of the *Ighu* samples as affected by changing levels of water activity and storage temperatures. It was observed that an increasing EMC with increasing water activity levels for the samples irrespective of the processing method adopted. The higher equilibrium moisture content recorded for *Ighu* samples processed using the Udi method (**Figures 7, 9** and **11)** could be attributed to the larger shred diameter (average of 4 mm) of *Ighu* strands from this method, which resulted from the bigger shredding aperture of the metallic device (*nko*) used for shredding. The larger strands of *Ighu* samples resulted in increased absorption of moisture at the storage temperatures observed (20, 30 and 40 °C). It could also be observed (**Figures 6** and **11**) that at water activity level above 0.5, there was increase in water adsorption for every small rise in a_W. This could be attributed to low water activities as physical sorption occurs on strongly active binding sites of substrate, present on the surface film. In the intermediate range of water activity (a_w), adsorption of moisture occurred at less active sites, which is an indication that the region could be a zone more susceptible to spoilage. Reports from Ariahu et al. supported this result for behavior of different hygroscopic foods [7]. The displayed moisture adsorption isotherm curves are typically a sigmoid shape and Type II isotherms [15,19]. This type of isotherm curve (Type II) usually leads to the formation of multiple layers of adsorbate molecules at the internal surface of food solid, a characteristic specific to most organic tissues and especially in hygroscopic food products [20]. However, Shivhare et al. reported a contrasting kind of moisture adsorption for *Awara* which was attributed to high protein content of the product [21].

Furthermore, **Figures 6, 8** and **10** (*Ighu* processed using Isuochi method) showed an overlapping isotherm at temperatures of 20 °C and 30 °C while a clear line was observed at higher temperatures (40 °C). The increasing EMC of the cassava shreds with increasing a_W at selected temperatures irrespective of the processing method could also be a result of increase in water vapour pressure present in food with that of the surrounding which corresponds with findings of Shivhare et al. in adsorption study using mushroom [21], Ojike et al. for *Gongronema latifolium* leaf grits and Shivhare et al. for *awara* (tofu-like product from soybean [15,21]. This trend has been reported to be common to all food materials and it is an indication that *Ighu* samples would adsorb more water at higher relative humidity/water activity. However, if relative humidity of the storage environment is kept constant, *Ighu* may absorb more moisture at lower temperatures than at higher temperatures. Whereas at constant moisture content, increasing temperature would lead to lowering of isotherm curves, which will increase aw, thereby making Ighu shreds prone to microbial spoilage by microbes.

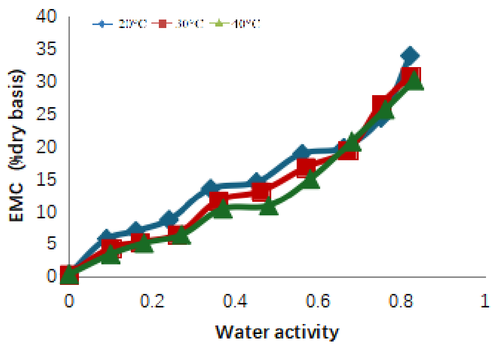

Figure 8. Effect of Water Activity on Equilibrum Moisture Content of *Ighu* from TMS 98/87614 (Isuochi Method).

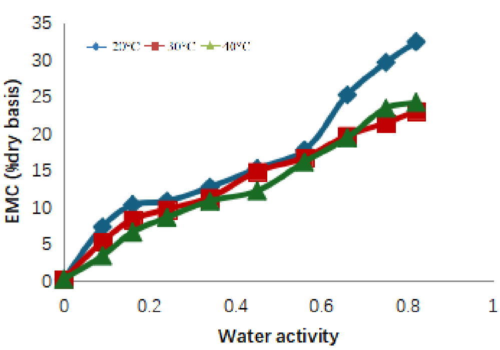

Figure 9. Effect of Water Activity on Equilibrum Moisture Content of *Ighu* from TMS 98/87614 (Udi Method).

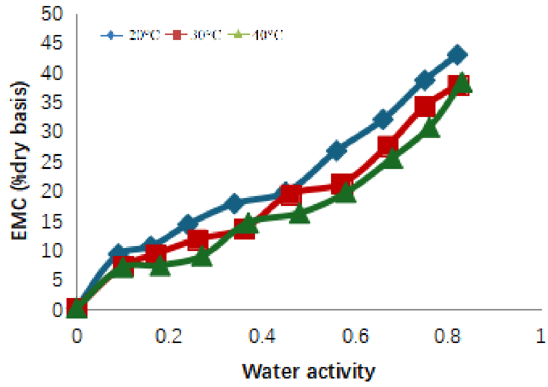

Figure 10. Effect of Water Activity on Equilibrum Moisture Content of *Ighu* from NR 8082 (Isuochi Method).

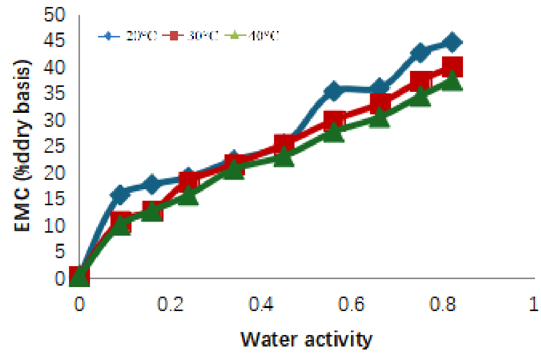

Figure 11. Effect of Water Activity on Equilibrum Moisture Content of *Ighu* from NR 8082 (Udi Method).

3.2. Moisture Sorption Models and Derivatives

The results of the different moisture sorption equations used to fit generated data are presented in **Table 3**.

3.2.1. Monolayer Moisture Content

Monolayer moisture content (Mo) of *Ighu* samples was determined using the BET and GAB models. A significantly lower BET monolayer moisture content [8.64 to 10.35 g H_2O/100 g (Isuochi method) and 7.60 to 13.12 g H_2O/100 g (Udi method) for TME4779; 6.91 to 9.14 gH_2O/100 g (Isuochi method) and 8.60 to 13.97 g H_2O/100 g (Udi method) for TMS 98/87614 and 9.36 to 11.80 g H_2O/100 g (Isuochi method) and 13.89 to 15.28 g H_2O/100 g (Udi method) for NR 8082] were obtained for the *Ighu* samples compared to GAB monolayer moisture content [8.07 to 10.75 g H_2O/100 g (Isuochi method) and 7.60 to 13.12 g H_2O/100 g (Udi method) for TME 4779; 8.11 to 10.57 g H_2O/100 g (Isuochi method) and 8.60 to

13.97 g H_2O/100 g (Udi method) for TMS 98/87614 and 10.09 to 14.78 gH_2O/100 g (Isuochi method) and 13.89 to 15.28 g H_2O/100 g (Udi method) for NR 8082. The variation in monolayer moisture content of the models could be attributed to the fact that GAB model is a specialized or improved form of BET model. The Monolayer moisture content revealed the highest amount of water that is strongly adsorbed to specific sites of dry substance per gram and an indication of a favourable value at which a food is more stable [22,23]. The M_o is described as the water level in a food at which biochemical, enzymatic, and microbial reactions will occur negligibly due to strong binding of water to the surface [24]. It is therefore a crucial parameter for selecting suitable storage conditions for hygroscopic food products. There was a marked inverse variation between monolayer values of the samples with the temperature from 20 to 30 °C, but increased at 40 °C irrespective of varietal effect or processing method used. This might be due to lower number of active sites for water binding as a result of the physical and chemical changes as affected by temperature [15]. Also, high temperature could activate the water molecules of the food due to an increase in energy levels, causing less stability and breaking away from the water binding sites. Thus, the monolayer moisture content of the food is reduced, and it is supported by the reports of Ariahu et al., who evaluated adsorption isotherm of tropical water crayfish, including other authors [7]. However, increasing monolayer moisture content and temperature might also be due to more opening of new binding sites or some of its components becoming increasingly hydrophilic as the temperature increases. This can allow greater water vapor molecules to bind, resulting in increase in M_o [15].

Table 3. Moisture Adsorption Parameters for *Ighu* Processed Different Cassava Varieties.

	***Ighu* from Isuochi method**			***Ighu* from Udi method**		
Models/temp	**20 °C**	**30 °C**	**40 °C**	**20 °C**	**30 °C**	**40 °C**
***Ighu* from TME 4779**						
GAB Model						
Mo (g H_20/100 g solid)	10.75	8.88	8.07	17.37	12.15	9.10
C	27.18	23.44	10.82	26.56	9.61	9.00
K	0.85	0.95	0.95	0.72	0.89	0.87
R^2	0.9718	0.9758	0.8743	0.9511	0.8452	0.8529
RMSE	1.548	1.569	1.599	1.548	1.569	1.599
BET Model						
Mo (g H_20/100 g solid)	10.35	8.62	8.64	13.12	10.12	7.60
So (m^2/g solid)	363.67	302.94	303.94	461.09	355.66	269.10
C	31.24	28.81	9.64	113.65	14.67	10.98
R^2	0.9897	0.9956	0.9609	0.9894	0.9962	0.9940
RMSE	0.643	0.674	0.722	0.642	0.673	0.703
Oswin Model						
A	3.02	2.88	2.68	3.35	3.18	2.90
B	0.42	0.45	0.58	0.28	0.45	0.47
R^2	0.9799	0.9763	0.9786	0.9486	0.9758	0.9694
RMSE	7.09	6.18	6.18	7.49	7.49	7.10
Halsey Model						
A	2.72	2.56	2.35	3.02	2.66	2.31
B	−0.64	−0.69	−0.78	−0.46	−0.74	−0.80
R^2	0.9945	0.9950	0.9902	0.9669	0.9836	0.9760
RMSE	9.22	8.89	8.51	9.87	9.21	8.45
***Ighu* from TMS 98/89617**						
GAB Model						
Mo (g H_20/100 g solid)	10.59	9.48	8.11	9.83	13.11	13.30
C	8.429	5.502	4.920	30.724	10.400	4.691

Table 3. *Cont.*

	Ighu from Isuochi method			*Ighu* from Udi method		
Models/temp	**20 °C**	**30 °C**	**40 °C**	**20 °C**	**30 °C**	**40 °C**
K	0.9658	0.8707	0.9029	0.8600	0.6281	0.6482
R^2	0.8093	0.5421	0.7728	0.9454	0.9765	0.7969
RMSE	1.549	1.570	1.600	1.545	1.550	1.550
BET Model						
Mo (g H_20/100 g solid)	9.14	8.59	6.19	8.60	13.97	8.60
So (m^2/g solid)	321.22	301.89	242.85	302.34	490.96	302.24
C	11.05	5.52	6.83	63.93	14.97	8.60
R^2	0.9456	0.9838	0.908	0.9953	0.9910	0.9283
RMSE	0.645	0.678	0.707	0.644	0.645	0.647
Oswin Model						
A	2.761	2.617	2.515	3.481	3.358	3.268
B	0.459	0.561	0.600	0.282	0.311	0.305
R^2	0.9813	0.9758	0.991	0.9516	0.9510	0.9688
RMSE	6.64	6.57	6.57	7.73	7.53	7.41
Halsey Model						
A	2.437	2.289	2.102	2.598	2.393	2.211
B	−0.687	−0.830	−0.880	−0.597	−0.567	−0.725
R^2	0.9655	0.9648	0.9728	0.9813	0.9273	0.9157
RMSE	8.48	8.16	8.00	8.82	8.25	8.07
***Ighu* from NR 8082**						
GAB Model						
Mo (g H_20/100 g solid)	14.78	12.12	10.09	19.23	23.29	20.47
C	18.178	13.423	11.888	0.7449	0.5978	0.6029
K	0.8539	0.8465	0.9809	31.977	13.446	13.983
R^2	0.9392	0.9025	0.7715	0.9481	0.9799	0.9878
RMSE	1.548	1.569	1.600	1.548	1.548	1.548
BET Model						
Mo (g H_20/100 g solid)	11.80	10.91	9.36	13.98	15.28	13.89
So (m^2/g solid)	414.70	383.42	328.94	491.32	537.00	488.15
C	24.795	11.733	11.295	−127.22	16.96	19.28
R^2	0.9819	0.965	0.9181	0.9974	0.9848	0.9876
RMSE	0.643	0.673	0.700	0.641	0.642	0.642
Oswin Model						
A	3.147	2.967	2.849	3.481	3.358	3.268
B	0.426	0.462	0.483	0.282	0.311	0.305
R^2	0.9905	0.9918	0.9726	0.9516	0.9510	0.9688
RMSE	7.37	7.08	6.99	7.73	7.53	7.41
Halsey Model						
A	2.846	2.647	2.513	3.154	2.971	2.881
B	−0.640	−0.686	−0.717	−0.459	−0.542	−0.529
R^2	0.9805	0.9819	0.9769	0.9683	0.9362	0.9476
RMSE	9.57	9.12	8.88	10.25	9.86	9.66

3.2.2. Apparent Surface Area of Sorption

The BET model was used to estimate the apparent/specific surface area (S_o) of monolayer moisture according to equation 7. The range of 302.94 to 363.74 m^2/g solid (Isuochi method) and 269.10 to 461.09 m^2/g solid (Udi method) were reported for apparent surface area of moisture of *Ighu* samples from TME 4779, 242.85 to 321.22 m^2/g solid (Isuochi method) and 302.24 to 490.96 m^2/g solid (Udi method) for TMS 98/87614, 328.94 to 414.70 m^2/g solid (Isuochi method) and 488.15 to 537.00 m^2/g solid (Udi method) for NR 8082. Higher apparent area of sorption was recorded at the lower storage temperatures and reduced with increasing temperature. This was as a result of the quantity of sorbed moisture at that level. The S_o values reported in this study were higher than the range of 100–250 m^2/g solid reported by Tunç and Duman (2007) and 179.2545 to 249.1489 m^2/g solid reported by Ojike *et al*. for *Gongronema latifolium* leaves dried using oven and sun [15]. The large disparity between the reported values and the values obtained in this research could be attributed to the nature of the products and higher starch content in cassava. Apparent surface area of sorption is used to estimate the water-binding properties of foods [15]. According to Yogendrarajah *et al*. [24], water adsorption could be influenced by surface area, porosity, composition, and the quantity of binding sites. The amount of water adsorbed by a food material is a result of the affinity between the surface and the water molecules, water vapor concentration, temperature, and the absolute amount of surface area revealed. A larger surface area means greater adsorption capacity, which gives rise to faster deterioration of food [24]. This statement is in line with the findings of the current research indicating the difference in S_o of the samples with respect to processing method.

3.2.3. The GAB Isotherm Parameters

The GAB constants (C and K) varied with temperature for adsorption isotherms of *Ighu* from different processing methods. The variation in C-values (**Table 3**) with temperature was greater than that of K-value. The C-values decreased with increasing temperature while K values for adsorption isotherm did not show any definite pattern across the temperature, and higher values were obtained for *Ighu* samples from Isuochi. Lower value of K indicates a much less structured state of the sorbate in the layer following the monolayer, which is the sorbates's pure liquid state. Timmermann *et al*. reported that the value of K is practical, which is near but always less than the unit (1) [25]. When the value of K is equal to 1, it is an indication that the multilayer has properties of bulk water, and the moisture sorption behavior could be modeled using the BET equation [23]. In this present research, it could be observed that the values of K were less than one (1) which indicated that multilayer of the samples has the properties between those of monolayer and the bulk water, which is in agreement with the GAB model assumption. It also revealed that GAB isotherm model could be applied on the *Ighu* samples irrespective of processing methods used. Several researchers reported K-values below 1 in their research on wheat, rice and corn flours; and *Gongronema latifolium* leaf grits, respectively [15,26,27].

The C value on the other hand showed more temperature dependence than K, as the values of C were observed to decrease with increasing temperature for both samples. The inverse relationship between C-values and temperature is an indication that GAB constant C is mostly enthalpy related. Ojike et al. reported that the GAB constant C corresponds to the magnitude of differences between the chemical potentials of monolayer water molecules and multilayer water molecules [15]. The C-value also explains the adsorbent (food product)-adsorbate (water) interactions. As reported by Blahovec and Vanniotis [28], when C value is greater than 2, the GAB model would yield a sigmoid shape curve with point of inflection. However, if the value is less than 2 but greater than 0, the isotherm would correspond to type III only (isotherm without point of inflection). The C values obtained in this present study were greater than 2 irrespective of temperatures and processing methods used. The obtained values supported the predictions mentioned earlier that isotherm curves obtained were sigmoid and of type II. Ojike et al. also reported C values (25.7151 to 762.689), which are within the values reported in this research [15].

3.2.4. BET Isotherm Parameter

The BET model has been reported to fit experimental sorption data in the range of water activities below 0.5. According to Agurre and Owoicho et al., the main limitation of BET theory appears to be the assumption that all absorbed molecules in layers other than the 1st have liquid-like evaporation-condensation properties [27,29]. The BET constant C ranged from 9.64 to 31.24 (Isuochi method) and 10.98 to 113.65 (Udi method) for cassava variety TME 4779; 6.83 to 11.05 (Isuochi method) and 8.60 to 63.93 (Udi method) for TMS 98/87614; 11.30 to 24.80 (Isuochi method) and 19.28 to −127.22 (Udi method) for NR 8082.

3.2.5. Oswin Isotherm Model Derivatives

The Oswin derivatives (A and B) were determined by linear regression of Equation 6. The calculated fitting parameters of the Oswin model of this study are in good agreement with several authors [26,30,31]. The Oswin A-values of *Ighu* obtained for different cassava varieties showed the range of 2.619 to 3.022 (Isuochi method) and 2.897 to 3.349 (Udi method) for TME 4779; 2.515 to 2.761 (Isuochi method) and 2.751 to 3.033 (Udi method) for TMS 98/87614; while 2.849 to 3.147 (Isuochi method) and 3.268 to 3.481 (Udi method) were reported for NR 8082. On the other hand, derivative B ranged from 0.418 to 0.579 (Isuochi method) and 20.278 to 0.472 (Udi method) for TMS 4779; 0.459 to 0.600 (Isuochi method) and 0.304 to 0.377 (Udi method) for TMS 98/87614, and 0.426 to 0.483 (Isuochi method) and 0.282 to 0.305 (Udi method) for NR 8082.

The Oswin derivatives A and B showed a direct opposite relationship with temperature. The results showed that values of parameter A decreased with increasing temperature (from 20°C to 40°C), while the values of B increased with decreasing temperature regime and vice versa. This showed that these parameters are more temperature-dependent than dependent on changes in processing method. Ojike et al. reported similar Oswin B parameters of 0.480 to 0.491 and 0.383 to 0.442 for spray-dried sweetened yoghurt powder and Gongronema leaf grits, respectively, which are within the value range recorded in this work [15,32]. The Oswin model was first applied to type II isotherms by Oswin and was identified to be a good fit for sorption of various food products and might also be fit for non-proteinous materials and best for starchy foods. The Oswin model usually has a high coefficient of regression and has the advantage of simplicity of use in describing moisture sorption isotherms. It was reported to be a good fit for sorption in various food products especially for non-proteinous materials and best for starchy foods [33].

3.2.6. Halsey Isotherm Model Derivatives

Chirife and Iglesias modified the Halsey equation (28) which was used to determine derivatives A and B [34]. The success of the modified equation relies on linearization of the former to accommodate these parameters. Earlier research showed that the Halsey model has been successfully applied to foods of different compositions. The result of Halsey's model parameter A for *Ighu* obtained in this work showed the range of 2.342 to 2.724 (Isuochi method) and 2.311 to 3.021 (Udi method) for TME 4779; 2.102 to 2.437 (Isuochi method) and 2.211 to 2.598 (Udi method) for TMS 98/87614; while 2.513 to 2.846 (Isuochi method) and 2.881 to 3.154 (Udi method) was reported for NR 8082. Crapsite and Rostein applied Halsey model to describe sorption behaviour of starchy food [35]; Linko *et al.* used it for proteineous food while Lomauro *et al.* used it in modelling sorption characteristics of nuts and oil seeds [36,37]. They reported that the model showed good fit in explaining these data, which corresponds to the high coefficient of determination obtained in the present study.

3.2.7. Goodness of Fit of Chosen Models

The usefulness of sorption models depends on the overall objective of the user. It is important to select a model that is simple (i.e., with fewer parameters) with ease of use in engineering manipulations. The BET, GAB, Oswin and Halsey models were among the most widely used models for sorption experiment of food products due to their simplicity, versatility and ability to explain data of various food materials, and were used to describe experimental data through weighted non-linear regression analysis. The coefficient of determination (R^2) and root mean square error (RMSE) are among the commonly used parameters in literature to evaluate the goodness of fit of sorption models [38–40].

R^2 indicates how well the variability has been explained by the given model while the RMSE is a measure of how precisely the parameters have been estimated and also measures magnitude of varying quantities. For all the models tested, their coefficients of determination were closer to 1, which conferred goodness of fit to the models. However, only BET and GAB models returned RMSE less than 2.0. In view of the aforementioned, the Brunauer-Emmet-Teller model was best in describing moisture sorption behaviour of *Ighu* for both processing methods in terms of coefficient of determination and RSME, followed closely by the GAB model's RSME, which did not return better R2 compared to Oswin and Halsey models. This performance is in agreement with recently published research involving sorption isotherms of food materials [15,26,27]. There was no particular trend with fitted models with change in temperature or varietal differences but solely depended on the variation of EMC and water activity levels. Providing a universal mathematical model, either theoretical or empirical, for correct prediction of sorption isotherms in the whole range of water activity for different types of foods is difficult [41]. Taking into account that food materials with similar chemical composition but different physical characteristics may give different sorption isotherms, it is necessary to obtain experimental data and then determine specific values for the adequate model [42].

3.2.8. Water Vapour Transmission Rate (WVTR) and Permeability Coefficient (p) of Packaging Materials at Different Relative Humidity

The water-vapour transmission rate (WVTR) and coefficient of permeability (P) of the three different packaging materials used in this research [low and high densities polyethylene (LDPE and HDPE)], and laminated nylon were tested for their adequacy to provide the necessary protection to *Ighu* during storage and transportation as presented in **Table 4**.

The WVTR for the different packaging materials LDPE, HDPE and laminated nylon ranged from 1.201 to 4.011 g H^2O/day/ m^2, 0.240 to 2.391 g H_2O/day/ m^2 and 0.048 to 2.250 g H_2O/day/ m^2 respectively while permeability coefficient of the packaging materials ranged from 0.037 to 0.0278 g mm/m^2 day mmHg for LDPE, 0.00561 to 0.01242 g mm/m^2 day mmHg for HDPE and 0.000599 to 0.006237 g mm/m^2 day mmHg for laminated nylon.

Table 4. Effect of Different Relative Humidity on WVTR and Permeability Coefficients of Different Packaging Materials.

Salt	RH	Δp [(30°C) mmHg]	Q/t (g H_2O/ day)			WVTR (g H_2O/ day/ m^2)			P (g mm/m^2 day mmHg)		
			LDPE	HDPE	Laminated nylon	LDPE	HDPE	Laminated nylon	LDPE	HDPE	Laminated nylon
CH_3COOK	20.16	6.415	0.01884	0.0046	0.0014	1.201	0.240	0.048	0.028082619	0.007482463	0.002618862
$MgCl_2$	32.40	10.310	0.0298	0.0076	0.0026	1.592	0.400	0.090	0.023161979	0.007759457	0.003055286
K_2CO_3	43.20	13.746	0.025	0.0110	0.0244	2.122	0.579	0.840	0.023155827	0.008424269	0.02138804
$NaNO_2$	63.50	20.206	0.0446	0.0201	0.0299	2.840	1.060	1.030	0.021082847	0.010491933	0.017841235
NaCl	75.50	24.024	0.0491	0.0327	0.0467	3.131	1.722	1.610	0.019549201	0.014335664	0.023455711
KCl	83.34	26.519	0.0502	0.0399	0.0597	3.202	2.100	2.060	0.018111543	0.015837701	0.027188054
KNO_3	90.70	28.861	0.0630	0.0454	0.0653	4.011	2.391	2.250	0.020846471	0.016569072	0.027285957
Surface area (m^2)			0.557	0.557	0.459						
Thickness (mm)			0.15	0.20	0.35						

Key: RH = relative humidity, Δp = difference in saturated vapour pressure of pure water and RH, Q/t= slope of isotherm curve, WVTR= water-vapour transmission rate, P= permeability coefficient, LDPE and HDPE = low and high densities polyethylene

Both WVTR and permeance of the packaging materials decreased with increasing relative humidity at constant temperature of 30 °C and saturated vapour pressure of pure water of 31.82 mmHg, which is an indication that the ability of packaging materials to maintain their barrier to moisture migration was significantly impacted by changing relative humidity levels. The results showed that LDPE had the highest WVTR and permeability coefficient over the different RH studied followed by HDPE while laminated nylon recorded least values. The different WVTR recorded for the different packaging materials in this research established further that dissimilar materials or components layered in a packaging material contribute to its moisture uptake and WVTR. The concept of permeability is normally associated with the quantitative evaluation of the barrier property of a packaging material [43]. According to Macedo et al. [44], packaging film permeability can be described as (i) transmission rate that describes permeation per area basis, (ii) permeance that describes permeation as area per pressure difference basis, and (iii) permeability which describes permeation as area per pressure difference per thickness basis. Previous researchers have also reported higher WVTR of LDPE compared to other packaging materials [45,46]. Moyls and Yaptenco *et al*. showed that changing storage temperature at constant RH could affect WVTR of some packaging materials [47,48]. Moyls reported values of 3.71 (32 °C), 3.00 (44 °C), and 5.22 (61 °C) for Polyethylene [47], while Yaptenco *et al*. reported 1.47 (30 °C) and 2.17 (35 °C) for Polyethylene, 0.45 (30 °C) and 1.29 (35°C) for Polypropylene [48].

3.2.9. Shelf-Life Estimation for *Ighu* as Affected by Packaging Materials and Relative Humidity

The shelf life of *Ighu* was calculated using Equation 13. The values obtained for initial, critical and storage moisture contents as well as WVTR and permeability results were used in estimating shelf life of *Ighu* as affected by varying relative humidity and constant storage temperature of 30°C. The estimated shelf life is presented in **Table 5** below.

Table 5. Predicted Shelf Life (Days) of *Ighu* in Different Packaging Materials at 30°C and Different Relative Humidity.

Sample	RH	ISUOCHI			UDI		
		LDPE	HDPE	Laminated Nylon	LDPE	HDPE	Laminated Nylon
TME 4779							
	20.16	168	840	5097	150	750	4548
	32.40	204	810	4369	182	723	3898
	43.20	204	746	624	182	666	557
	63.50	224	599	748	200	535	668
	75.50	241	438	569	215	391	508
	83.34	260	397	491	232	354	438
	90.70	226	379	489	202	338	436
TMS 98/87164							
	20.16	138	692	4200	151	757	4594
	32.40	168	668	3600	183	730	3937
	43.20	168	615	514	183	672	562
	63.50	184	494	617	202	540	674
	75.50	199	361	469	217	395	513
	83.34	214	327	406	235	358	442
	90.70	186	313	403	204	342	441

Table 5. *Cont.*

Sample	RH	ISUOCHI			UDI		
		LDPE	HDPE	Laminated Nylon	LDPE	HDPE	Laminated Nylon
NR 8082							
	20.16	148	742	4502	152	762	4621
	32.40	180	715	3859	185	734	3961
	43.20	180	659	551	185	676	566
	63.50	198	529	661	203	543	678
	75.50	213	387	503	219	398	516
	83.34	230	351	434	236	360	445
	90.70	200	335	432	205	344	444

Key: RH = Relative humidity, LDPE = Low density polyethylene and HDPE =High density polyethylene

The result showed that the shelf life of a product is directly related to the permeance or permeability coefficient (P) of the packaging material. A lower material permeance to water, oxygen or other storage environmental factors, the higher it can keep and protect what is packaged in it. The result also revealed the effect of changing storage relative humidity on packaged *Ighu*. It was observed that shelf life of *Ighu* increased with increasing RH in LDPE material up to 83.34%, then decreased remarkably. However, in HDPE and laminated nylon material, an increase in shelf life was observed as RH of the environment increased up to 75.50% RH.

Generally, *Ighu* processed using Isuochi indigenous method recorded higher shelf life compared to those processed using Udi method. This could be attributed to lower initial moisture and critical moisture contents of the former, as well as thickness variations among the products. Packaging *Ighu* in LDPE material at 30°C over the various relative humidity tested resulted in lower storability (<1year), i.e., 168 to 260 days (Isuochi method) and 150 to 232 days (Udi method) for *Ighu* from TME 4779, 136 to 214 days and 151 to 352 days (Udi method) for *Ighu* from TMS 98/87614 while 148 to 230 days and 152 to 236 days (Udi method) for NR 8082. Higher shelf life was recorded for *Ighu* stored in HDPE material above two years but less than four years. Shelf life in HDPE material for *Ighu* from various cassava varieties was in the range of 379 to 840 days (Isuochi method) and 338 to 750 (Udi method) for *Ighu* from TME 4779, 313 to 692 days (Isuochi method) to 342 to 757 days (Udi method) for *Ighu* from TMS 98/87614) while 335 to 742 days (Isuochi method) and 344 to 762 days (Udi method) were recorded for *Ighu* from NR 8082.

The result of shelf life prediction showed that shelf life of *Ighu* recorded highest values in laminated nylon, especially at lower relative humidity of 20.16 and 32.40%, which recorded shelf life values above ten years. This is an indication that laminated nylon was almost impermeable to moisture, which resulted in higher storability of the products in this condition. Further increase of RH up to 43% drastically reduced shelf life by over 80%. Results from this research were supported by previous researches. Yaptenco *et al*. reported decreased shelf life of whole dried sandfish with change in relative humidity while Patindol and Norio reported low shelf life of blue butterfly pea power stored in nylon/PE material [46,48].

4. Conclusions

Moisture adsorption isotherms of *Ighu* from different cassava varieties, studied over various storage temperatures (20 °C, 30 °C and 40 °C) and varying water activity levels showed that dried *Ighu* samples adsorbed more moisture at lower temperatures and exhibited sigmoid shape type II isotherm curves which is peculiar with most food products and EMC directly increased with water activity at constant temperature. Processing methods and varietal differences had significant impact on EMC of the products studied. Higher moisture adsorption was recorded for *Ighu* processed using Udi method compared to samples from Iusochi method. There was no sorption model that satisfactorily described the sorption behaviour of *Ighu* from different cassava varieties over the studied temperature range and water activity. This underlined the difference between varieties of cassava available. However, in general terms, the BET model which accounted for EMC of water activities below 0.50 was best in describing the sorption behaviour of *Ighu* samples with the highest coefficient of determination and lowest RMSE values compared to GAB, Oswin and Halsey models in descending order of reliability. The results of WVTR and permeability coefficient of the three packaging materials (LDPE, HDPE and laminated nylon) used to simulate shelf life of *Ighu* at different relative humidity showed that laminated nylon had the lowest WVTR and permeability at lower relative humidity which increased with increasing RH levels compared to HDPE and LDPE at the same relative humidity. At these low RH values (20.16 and 32.40) *Ighu* will last up to 10 years in laminated nylon which reduced exponentially when relative humidity was increased to 43% and above. It is therefore recommended that cassava tubers should be converted to *Ighu* as the product will have higher shelf stability up to 10 years. Recommendation is therefore made that cassava roots should be converted to *Ighu* since it lasts longer on the shelf (>10 years) especially when stored in laminated nylon under low relative humidity

Author Contributions

Conceptualization, A.L-C., and M.O.I. methodology A.L-C and T.U.N.; software A.L-C; validation M.O.I and T.U.N; formal Analysis, A.L-C.; investigation, C.O.A-L.; resources A.L-C; data curation A.L-C; writing—original draft preparation, A.L. and C.O.A-L; writing-review and editing A.L-C and M.O.I; , visualization C.O.A-L; supervision M.O.I and T.U.N, project administration A.L-C; funding acquisition M.O.I and A.L-C All authors have read and agreed to the published version of the manuscript.

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Not applicable.

Data Availability Statement

The datasets generated during and/or analyzed during the current study are available from the corresponding author (AL-C) upon reasonable request.

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Conflicts of Interest

The authors declare no conflict of interest. The funders had no role in the design of the study; in the collection, analyses, or interpretation of data; in the writing of the manuscript; or in the decision to publish the results.

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