

Effect of Heat-Moisture Treatment on Equilibrium Moisture Content Models for Cassava Starches

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Abstract

The level of water contained in food products is known to affect several unit operations in food processing including drying, storage, and thermodynamics phenomena which are related to the sorption attributes of food. Heat-moisture treatment as an aspect of the thermodynamic properties of the food affects sorption phenomena and invariably the associated empirical models for the sorption behaviour.

In this study, effect of heat-moisture treatment on sorption moisture isotherms for cassava starch at 27, 32, and 37°C, respectively was determined for selected cassava genotypes (TMS 97/4763 and TMS 98/0510) including its impact on equilibrium moisture content (EMC) empirical models. The moisture isotherms were sigmoidal indicating effect of temperature and show the influence of the heat pre-treatment. Six widely recommended three parameters sorption models were fitted to the EMC data from the gravimetric method. Desorption isotherms appears well fitted than adsorption isotherms. Analysis shows that due to the heat-moisture treatment, the Modified Oswin Equation (MOE) and Modified Halsey Equation (MHE) models are the preferred empirical equations for the modelling of the emc data for the cassava starches.

Keywords: Cassava starch, equilibrium-moisture-content, heat-moisture treatment, sorption, isotherms.

1. Introduction

Water contained in food will normally affect physical properties, behaviour during processing, susceptibility to microbial attack, sensory attributes, biological and chemical stability (Fennema, 1996, Yu et al., 2008). This is an indication that water is the central component in food systems and it is known to exist in the 'free' or 'bound' states depending on how it interacts with other food components such as proteins, carbohydrates and salts (Duggan et al., 2008). The form in which the water exists may also affect the rate at which it may be removed from the food product through dehydration. Therefore, drying is an important process in starch preparation and preservation, particularly when the material to be dried contains high water content (Sundaram & Durance, 2008). Drying is a critical process since it may involve high energy costs depending on the water content and its form within the food, and these additional costs may make the whole process uneconomical as the operating condition may significantly affect quality of final product (Liebanes et al., 2008).

Water can interact with crystalline solids by adsorption of moisture on particle surfaces, by crystal hydrate formation, by deliquescence, and through capillary condensation. In contrast, amorphous materials have a high capacity for water vapour sorption because the amount of water taken up is greater than what could be accounted for only by surface adsorption. Invariably, the bulk properties of the solid can be significantly affected as previously observed by Airaksinen et al., (2005). It is, therefore, important to know the sorption characteristics of starchy food products upon heat-moisture treatment because of the sensitivity of the solid granules to changes in heat and moisture content. The sorption characteristics of the food materials may be modelled using empirical equations which can be applied in other unit processing operations, such as drying and storage, and can be used to determine thermodynamics properties of food products. The aim of this study was to determine the effect of heat-moisture treatment on the sorption characteristics of cassava starches, and determine the effect that this will produce on the empirical models for the equilibrium moisture content of the selected cassava starches.

2. Materials and methods

2.1 Material preparation

Following a previous sorption study on 5 cassava genotypes by Oyelade and Khokhar (2011), heat-moisture modified samples from 2 genotypes (TMS 97/4763 and TMS 98/0510) were used as samples by adjusting the

initial moisture contents in the native starches to 20, 35 and 45%, respectively, and consequently heat treated at either 40 or 55°C for 16h. Samples for moisture sorption study were selected as those samples of the starches that have been treated at moisture content of 35% following their suitability for use as functional food ingredients (Oyelade, 2009). The native starch of each cultivar was used as a control.

Thin layer drying of each of the modified starch (100g) was carried out in an oven at 105°C. Weight loss was monitored until the weight of the sample corresponded to the moisture levels recorded in Oyelade and Khokhar (2011) previous study, and which are 11.5 and 15.5 %, d.b for TMS 97/4763 and TMS 98/0510, respectively, in order to be able to objectively determine the effect of heat-moisture pre-treatment on the starch samples. A range of constant relative humidity environment (a_w between 0.15 and 0.85) was provided for the sorption experiment using sulphuric acid solution to ensure uniformity of moisture contents and micro-climate regions with the conditions earlier reported in the sorption study of the native starches by Oyelade and Khokhar (2011).

2.3 Determination of adsorption isotherms

Detailed static gravimetric method recommended by the COST 90 project (Wolf et al., 1985), was used in this study as previously described by Bell and Labuza (2000) and reported by Oyelade & Khokhar (2011) in an aspect of this study involving the unmodified cassava starch.

2.4 Determination of desorption isotherms

A step-wise transfer of the equilibrated samples in the adsorption phase at each higher a_w to the immediate lower a_w point was made. This involved the cyclic transfer of equilibrated samples after the adsorption regime, within the range of a_w of 0.85-0.25 to the range of a_w points of 0.75-0.15 (Oyelade, 2008, Oyelade et al., 2008a; 2008b, Oyelade and Khokhar, 2011), and equilibration was considered reached when at least three consecutive weight measurements gave identical readings for the desorption regime.

2.5 Isotherm equations and modelling

Six previously experimented sorption isotherm equations of Guggenheim, Anderson and de Boer (GAB), modified equations by GAB (MGAB), Oswin (MOE), Henderson (MHDE), Chung Pfsot (MCE), and Halsey (MHE) for the native cassava starches by Oyelade and Khokhar (2011) and presented in Table 1 were used to fit the experimental sorption isotherm data in this study using a SAS 9.1 statistical package.

Table 1: Sorption isotherm models for cassava starch

Model	$M = f(a_w, T)$
GAB	$M = \frac{abca_w}{(1-ca_w)(1-ca_w+bc a_w)}$
MGAB	$M = \frac{a(\frac{c}{T})ba_w}{(1-ba_w)\left[1-(ba_w) + (\frac{c}{T})ba_w\right]}$
MOE	$M = (a + bT)\left(\frac{a_w}{1-a_w}\right)^c$
MHDE	$M = \left[\frac{\ln(1-a_w)}{-a(T+b)}\right]^{1/c}$
MCE	$M = -\frac{1}{c} \ln\left[\frac{(T+b)\ln(a_w)}{-a}\right]$
MHE	$M = \left(\frac{\ln(-\ln a_w)}{a+bT}\right)^c$

GAB= Guggenheim, Anderson and de Boer equation, MGAB = modified Guggenheim, Anderson and de Boer equation, MOE = modified Oswin equation, MHDE = modified Henderson equation, MCE = modified Chung-Pfost equation, MHE = modified Halsey equation

Where, M= equilibrium moisture content (% , dry basis);
 a, b, c = unknown values to be estimated;
 T = temperature, °C

The suitability of the models in Table 1 was determined by comparing three (3) evaluation indicators for each of the models. These indicators which are presented in Equations 1 to 3 are the residual sums of squares (RSS), the sum of the square error (SEE) , and the co-efficient of determination (R^2) as an index of variability between the experimental and predicted data (Chowdhury et al., 2005, Akanbi, et al., 2006, Cordeiro, et al., 2006, Oyelade, 2008; Oyelade et al., 2008a, Oyelade et al., 2008b):

$$RSS = \sum_{i=1}^n (M_{calculated} - M_{predicted})^2 \quad 1$$

$$SEE = \sqrt{\frac{\sum_{i=1}^n (M_{calculated} - M_{predicted})^2}{d.f}} \quad 2$$

$$R^2 = \left(1 - \frac{RSS}{TSS}\right) \quad 3$$

Residual plots for each of the models, wherein extent of random distribution of the residuals around zero indicating degree of reliability of the predicted data were also generated to further clarify level of inconsistencies between the predicted and experimental sorption data (Kuye & Ariri, 2005., Chowdhury et al., 2005, Oyelade & Khokhar, 2011).

3. Results and discussion

3.1 Effect of HMT on isotherm plots

Hygroscopic equilibration for the cassava starch samples was reached within 10 days for adsorption and 7 days for desorption at each temperature level. The values of EMC including the standard deviation obtained for each sample are presented in Table 2. The three replicate EMC values are close for all the samples and indicating a high reliability for the experimental methodology as previously observed by Cordeiro et al. (2006).

The data in Table 2 show a deviation between the data for the raw starch and the heat-moisture treated samples, indicating that there is an influence of heat treatment on the sorption characteristics of cassava starch resulting in the hygroscopic attributes being higher for the raw starch than for the modified starch. This trend is consistent with the data of Gevaudan et al., (1989) obtained for gelatinized cassava mash (96% gelatinisation, 48 h fermentation) compared to the native cassava starch, and Bizot et al., (1985) for gelatinised potato starch (5% freeze-dried gel) compared to the native potato starch. This is a further indication that water sorption capacity of starchy materials is affected by the method of preparation (Gevaudan et al., 1989), and so may be a factor affecting the pattern of the extent of starch hydrolysis when starch was heat-treated in the present study.

The isotherm plots of Figures 1 and 2 showed the effect of hydrothermal processing on the sorption characteristics of the starch samples for TMS 97/4763 and TMS 98/0510, respectively. Comparison of sorption plots of the native and modified starches showed that each hydro-thermally treated starch had lower EMC than the native starch for each a_w points at all the temperatures where sorption characteristics was investigated (27, 32 and 37°C).

The curves shows that each desorption isotherm overlays the corresponding adsorption isotherm which is consistent with the reports of other authors for potato and rice (Roman et al., 2004), kudzi starch and sweet potato starch (Boki & Ohno, 1991), pea starch film (Zhang & Han, 2008), pistachio nuts (Tavakolipour & Kalbasi-Ashtari, 2008), yam flour (Oyelade et al., 2008), potato slices dried and texturised by controlled sudden decomposition (Iguedjtal et al., 2008) and whole yellow dent corn (Samapundo et al., 2007). All hysteresis plots showed a tendency for a closed loop between the upper and lower bounds of a_w ($0 \leq a_w \leq 1$). The data and curves also show that the EMC decreased with increasing temperature, at a constant a_w , indicating that the cassava starch samples became less hygroscopic at these points. This may be due to reduction in total number of active sites for water binding because of physical and/or chemical changes in the samples induced by temperature (Mazza & LeMaguer, 1980, Cordeiro et al., 2006). However, with increasing a_w the slope of the isotherms increased due to higher sorption of water molecules by starch granules (Raj et al., 2002). Similar trends have been obtained for other food products including cassava mash (Gevaudan et al., 1989), potato starch (Bizot et al., 1985), gari (Chuzel & Zakhia, 1991), yam flour (Oyelade et al., 2008), lafun (Oyelade, 2008), orange leaves (Mohammed et al., 2005), walnut kernels (Togrul & Arslan, 2007), and cowpea flour (Oyelade et al., 2001).

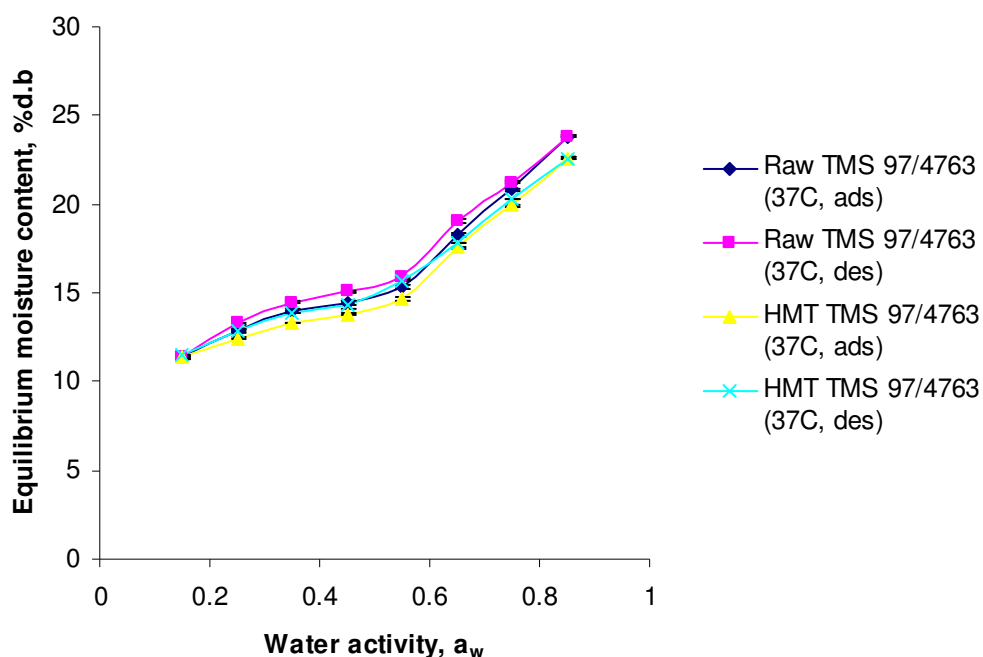


Figure 1: Effect of HMT on equilibrium moisture content and hysteresis plot for cassava starch (TMS 97/4763)

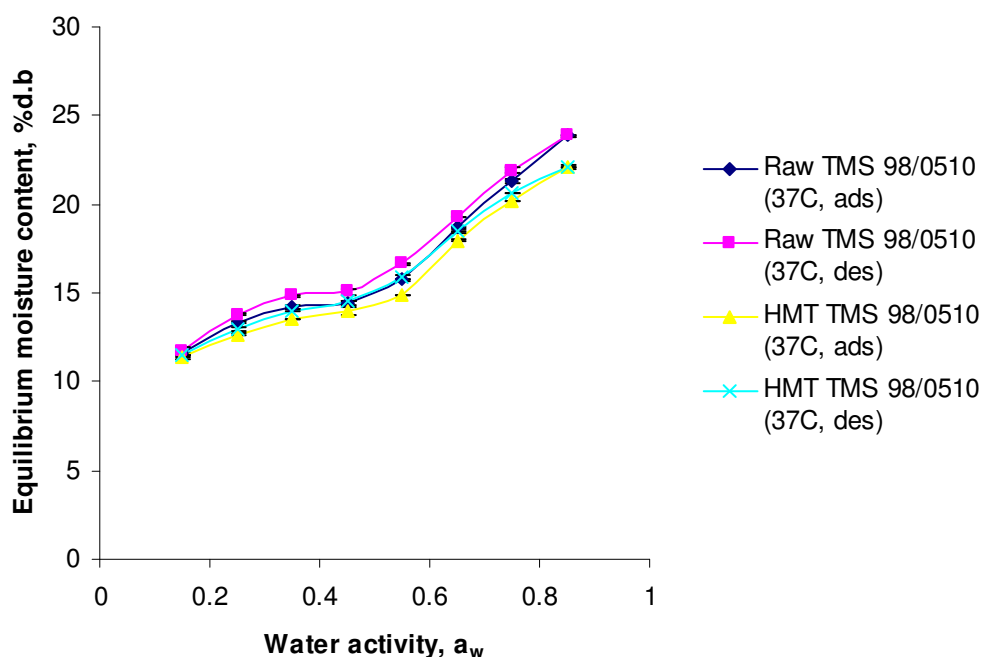


Figure 2: Effect of HMT on equilibrium moisture content and hysteresis plot for cassava starch (TMS 98/0510)

3.2. Effect of HMT on reliability of the predicted equilibrium moisture contents for starch samples

The six predictive isotherm equations listed in Table 1 were fitted to the mean of each experimental sorption data presented in Tables 2 (for adsorption cycle) and 3 (for desorption cycle) using the SAS 9.1 package. The

estimated co-efficients (a, b, c) and calculated values for the evaluation parameters (RSS, SEE, R^2) of each model are shown in Tables 4 and 5 for adsorption and desorption cycles, respectively.

The graphical representations of Figs 3 to 10 show varying effects of genotype, heat-moisture treatment, and sorption cycles for the starch samples. Figs 3 and 5 shows that heat-treatment has effect on the desorption cycle of cassava starch because of varying isotherms patterns while Figs 4 and 6 statistically confirms this effect as indicated in the random distributions for the desorption cycle using TMS 97/4673 starch sample. Similar trends were obtained for the adsorption cycle with TMS 98/0510 starch sample with Figs 7 and 9 indicating varying isotherms configuration, and Figs 8 and 10 showing random distribution plots for standardised residuals.

Results show that the GAB, MGAB, MCE & MHDE models are not good models for fitting the emc in this study. MCE & MHDE could not converge despite various trials. Residuals plots for the GAB models are not randomised whilst for the MGAB some of the residuals satisfy the randomised criteria but the emc predicted for some of the samples showed an inverse trend as the a_w increased. Analysis shows clearly that the MOE & MHE models reproduces the experimental sorption isotherms with high accuracy at all temperatures. The distributions of the residuals with the two models are not biased, confirming the high accuracy of the predictability of the isotherms.

Based on these results, and the comparison of the various indices of evaluation (R^2 , RSS and SEE), it is suggested that the MOE and MHE models are suitable for fitting of the emc data because they produced the highest R^2 value (0.9991) and the least RSS (6.8969) and SEE values (0.5361). The residual plots for MOE displayed more randomised patterns than for MHE which makes the MOE model rather better than the MHE model. This result underlines the needs to apply various indices in assessing the fitting adequacy of models (Cordeiro et al., 2006, Oyelade et al., 2008).

Table 2: Effect of HMT on equilibrium moisture content values at 27, 32 & 37°C for cassava starch samples (Adsorption)

a_w	Equilibrium moisture content, M (% d.b)					
	27°C		32°C		37°C	
	Native	HMT	Native	HMT	Native	HMT
TMS 97/4763						
0.15	13.11±0.06	11.78±0.07	11.78±0.07	11.34±0.06	11.34±0.08	11.34±0.08
0.25	14.88±0.05	14.67±0.04	13.76±0.04	13.77±0.02	12.89±0.04	12.44±0.04
0.35	16.86±0.14	15.98±0.08	15.29±0.11	14.66±0.08	13.99±0.12	13.34±0.02
0.45	17.53±0.06	16.67±0.06	15.99±0.08	15.52±0.03	14.44±0.11	13.77±0.06
0.55	18.21±0.01	18.15±0.05	17.31±0.04	17.09±0.05	15.33±0.13	14.64±0.08
0.65	20.44±0.05	21.33±0.03	20.00±0.13	18.89±0.03	18.27±0.07	17.54±0.06
0.75	23.66±0.09	24.13±0.05	21.73±0.15	21.06±0.01	20.84±0.04	19.92±0.03
0.85	26.21±0.08	27.50±0.05	24.30±0.08	23.91±0.01	23.84±0.09	22.61±0.04
TMS 98/0510						
0.15	13.56±0.07	12.45±0.06	12.01±0.01	11.79±0.05	11.57±0.05	11.34±0.05
0.25	15.40±0.06	14.67±0.08	14.22±0.03	14.28±0.08	13.33±0.04	12.67±0.05
0.35	16.88±0.08	16.44±0.07	14.89±0.13	14.81±0.02	14.22±0.14	13.56±0.03
0.45	17.46±0.05	16.84±0.07	16.00±0.11	15.25±0.02	14.45±0.13	14.01±0.21
0.55	19.39±0.10	17.92±0.02	18.08±0.07	17.65±0.08	15.75±0.04	14.87±0.02
0.65	21.37±0.11	20.18±0.03	19.91±0.13	19.75±0.06	18.67±0.03	17.97±0.08
0.75	25.17±0.13	23.71±0.01	22.80±0.13	22.70±0.07	21.31±0.14	20.20±0.03
0.85	29.06±0.21	27.41±0.01	26.56±0.07	26.54±0.09	23.86±0.09	22.12±0.11

*Readings are average of 3 replicates

Table 3: Effect of HMT on equilibrium moisture content values at 27, 32 & 37°C for cassava starch samples (Desorption)

a _w	Equilibrium moisture content, M (% , d.b)					
	27°C		32°C		37°C	
	Native	HMT	Native	HMT	Native	HMT
TMS 97/4763						
0.15	13.45±0.09	11.69±0.03	12.88±0.06	11.58±0.03	11.41±0.08	11.46±0.07
0.25	15.29±0.06	14.97±0.02	13.98±0.04	13.96±0.07	13.29±0.06	12.86±0.06
0.35	17.07±0.04	16.35±0.06	15.29±0.03	14.84±0.04	14.48±0.03	13.87±0.02
0.45	18.08±0.07	17.04±0.06	15.99±0.06	16.01±0.03	15.08±0.03	14.32±0.18
0.55	18.79±0.02	19.18±0.08	17.41±0.02	17.69±0.11	15.87±0.04	15.68±0.04
0.65	21.16±0.03	22.01±0.03	20.25±0.04	19.26±0.05	19.06±0.06	17.82±0.03
0.75	24.20±0.05	24.50±0.01	22.42±0.03	21.42±0.03	21.22±0.07	20.28±0.13
0.85	26.21±0.06	27.50±0.01	24.30±0.01	23.91±0.01	23.84±0.03	22.61±0.07
TMS 98/0510						
0.15	14.31±0.06	12.63±0.02	12.70±0.03	11.86±0.03	11.78±0.12	11.49±0.02
0.25	15.68±0.01	14.76±0.05	14.43±0.04	14.56±0.05	13.81±0.02	12.96±0.08
0.35	16.94±0.03	17.03±0.07	15.08±0.07	14.95±0.04	14.84±0.04	14.01±0.07
0.45	17.84±0.02	17.31±0.03	16.85±0.01	16.08±0.03	15.07±0.24	14.58±0.02
0.55	19.66±0.08	18.23±0.01	18.32±0.01	18.24±0.06	16.68±0.05	15.93±0.11
0.65	21.78±0.03	21.12±0.08	20.21±0.02	20.14±0.02	19.26±0.03	18.46±0.03
0.75	24.57±0.17	23.89±0.03	23.22±0.02	23.19±0.09	21.93±0.21	20.64±0.04
0.85	29.06±0.04	27.41±0.05	26.56±0.04	26.54±0.05	23.86±0.10	22.12±0.03

*Readings are average of 3 replicates

Table 4: Effect of HMT on estimated equation constants for fitting models to adsorption equilibrium moisture content data for cassava starches and evaluation indicators for models

Equation	Equation constants			RSS	SEE	R ²
	a	b	c			
MOE*	9.6408	0.2342	0.2112	7.0976	0.543814	0.9991
MGAB*	-11.7653	13.3808	35.2121	2182.5	9.536116	0.7213
GAB*	11.7985	179.1	0.6186	30.3285	1.124139	0.9961
MHE*	9.6408	0.2342	4.7346	7.0976	0.543814	0.9991
MOE**	6.7728	0.3092	0.2245	8.5084	0.595413	0.9989
MGAB**	-11.1725	13.2656	35.0559	2021.3	9.177191	0.7315
GAB**	11.2745	171.3	0.6408	49.9289	1.442349	0.9934
MHE**	6.7728	0.3092	4.4540	8.5084	0.595413	0.9989
MOE***	7.7563	0.3079	0.2292	8.3551	0.590025	0.9990
MGAB***	-11.8015	13.2041	35.0621	2312.3	9.815591	0.7259
GAB***	Parameters Not Estimated					
MHE***	7.7563	0.3079	4.3631	8.3551	0.590025	0.9990
GAB****	11.0535	2845.5	0.6629	46.3585	1.389822	0.9941
MOE****	7.8338	0.2846	0.2269	13.2512	0.743057	0.9983
MGAB****	-11.3805	13.1806	35.0938	2187.7	9.547469	0.7193
MHE****	7.8338	0.2846	4.4074	13.2512	0.743057	0.9983

* TMS 97/4763 Native Starch

**Modified TMS 97/4763 Starch (HMT)

*** TMS 98/0510 Native Starch

****Modified TMS 98/0510 Starch (HMT)

Table 5: Effect of HMT on estimated equation constants for fitting models to desorption equilibrium moisture content data for cassava starches and evaluation indicators for models

Equation	Equation constants			RSS	SEE	R ²
	a	b	c			
MOE*	9.7061	0.2337	0.2158	31.0716	1.137827	0.9961
MGAB*	-11.8631	13.3852	35.2049	2175.2	9.520154	0.7260
GAB*	Parameters Not Estimated					
MHE*	9.7061	0.2337	4.6332	31.0716	1.137827	0.9961
MOE**	7.1696	0.3090	0.2176	6.8969	0.536070	0.9991
MGAB**	-11.5178	13.3675	35.0985	2100.7	9.355702	0.7316
GAB**	12.2367	75.3541	0.6006	48.8412	1.426552	0.9938
MHE**	7.1696	0.3090	4.5959	6.8969	0.536070	0.9991
MOE***	8.7401	0.2907	0.2190	7.7566	0.568499	0.9991
MGAB***	-12.1381	13.1880	35.0937	2385.1	9.968910	0.7278
GAB***	11.9240	2019.9	0.6487	41.6096	1.316713	0.9953
MHE***	8.7401	0.2907	4.5657	7.7566	0.568499	0.9991
MOE****	8.4520	0.2778	0.2198	9.8331	0.640088	0.9988
MGAB****	-11.7293	13.2679	35.1389	2282.0	9.751068	0.7184
GAB****	11.9467	131.6	0.6262	43.1043	1.340154	0.9947
MHE****	8.4520	0.2778	4.5503	9.8331	0.640088	0.9988

* TMS 97/4763 Native Starch
 **Modified TMS 97/4763 Starch (HMT)
 *** TMS 98/0510 Native Starch
 ****Modified TMS 98/0510 Starch (HMT)

3.3: Evaluation of effect of HMT on predicted moisture isotherms for cassava starch samples

Analysis of the SEE and R² error indicators on the generated predictive moisture isotherms using equations 1 to 3, and presented in Tables 4 and 5 showed varying levels of suitability for each model to predict the isotherm curves. The range of SEE values ranged between 0.54 to 1.14, 9.18 to 9.97, 0.54 to 1.14 and 1.12 to 1.43 for the MOE, MGAB, MHE and GAB models, respectively while R² values ranged from 0.7184 to 0.9991 for the sorption cycles.

Results of residual plots indicated that MHDE and MCE are not suitable compared to what obtained for the MOE model.

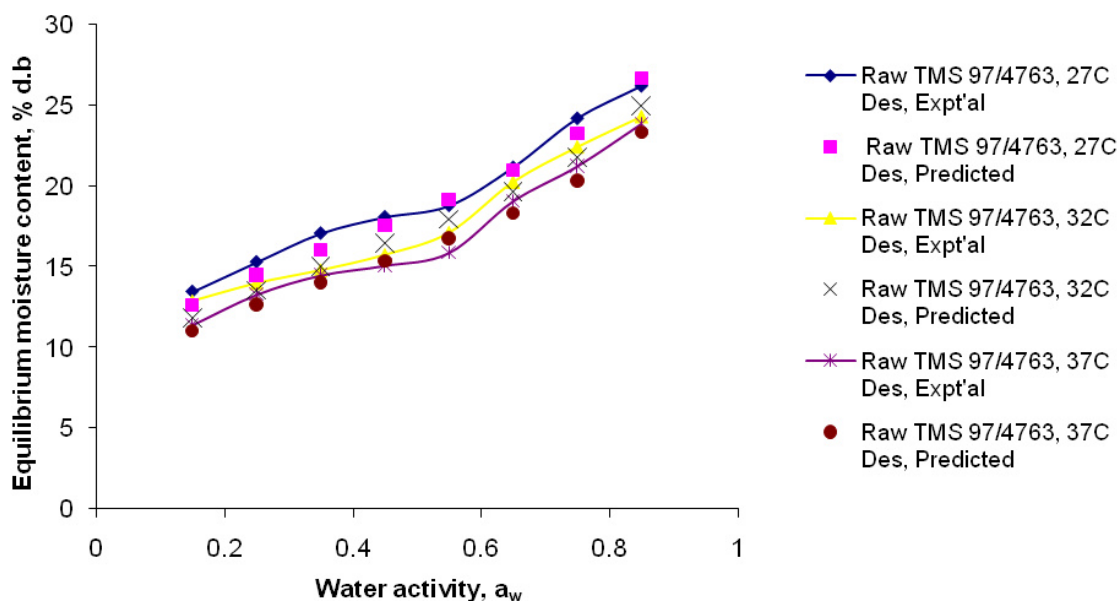


Figure 3: Comparison of experimental and MOE predictive desorption isotherms (Raw/Native TMS 97/4763)

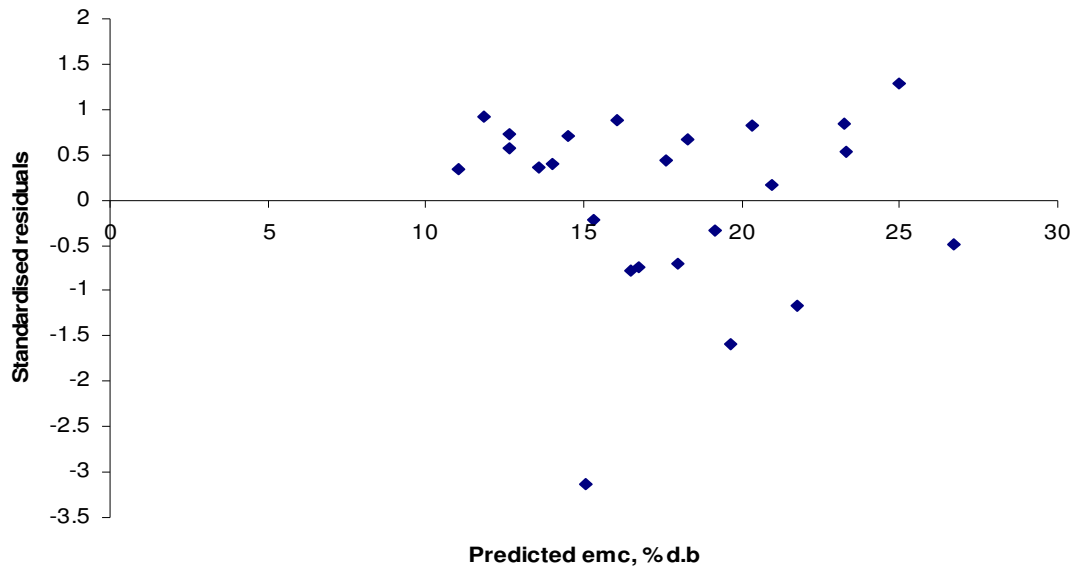


Figure 4: Desorption isotherms predictive model residual plot for Raw/Native TMS 97/4763 (MOE)

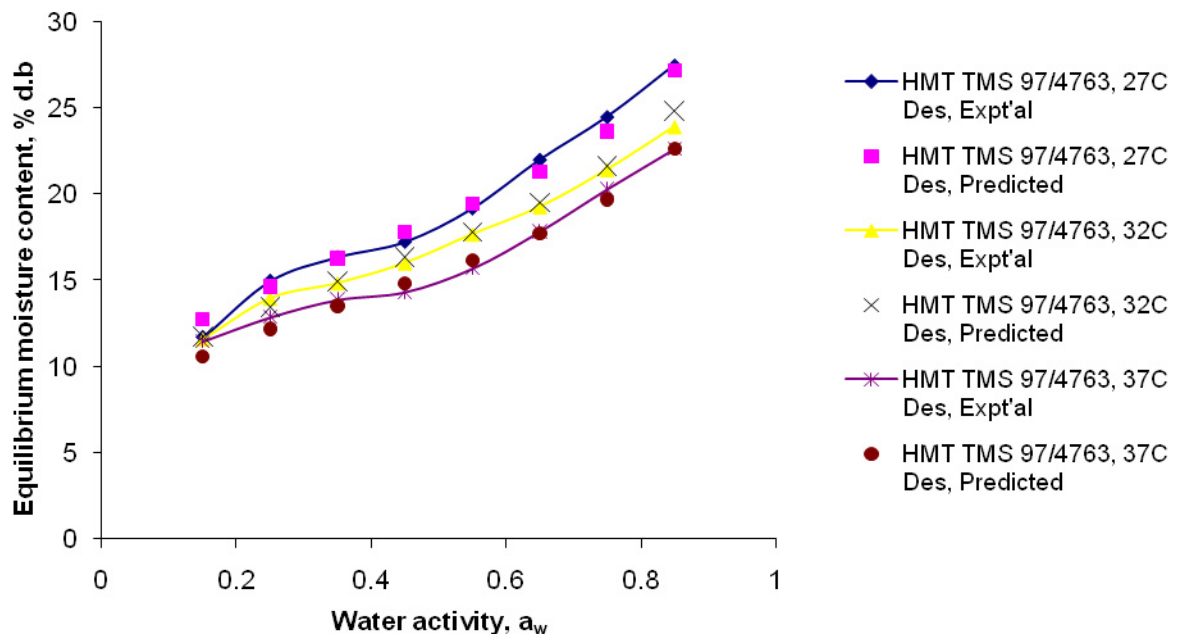


Figure 5: Comparison of experimental and MOE predictive desorption isotherms (modified TMS 97/4763)

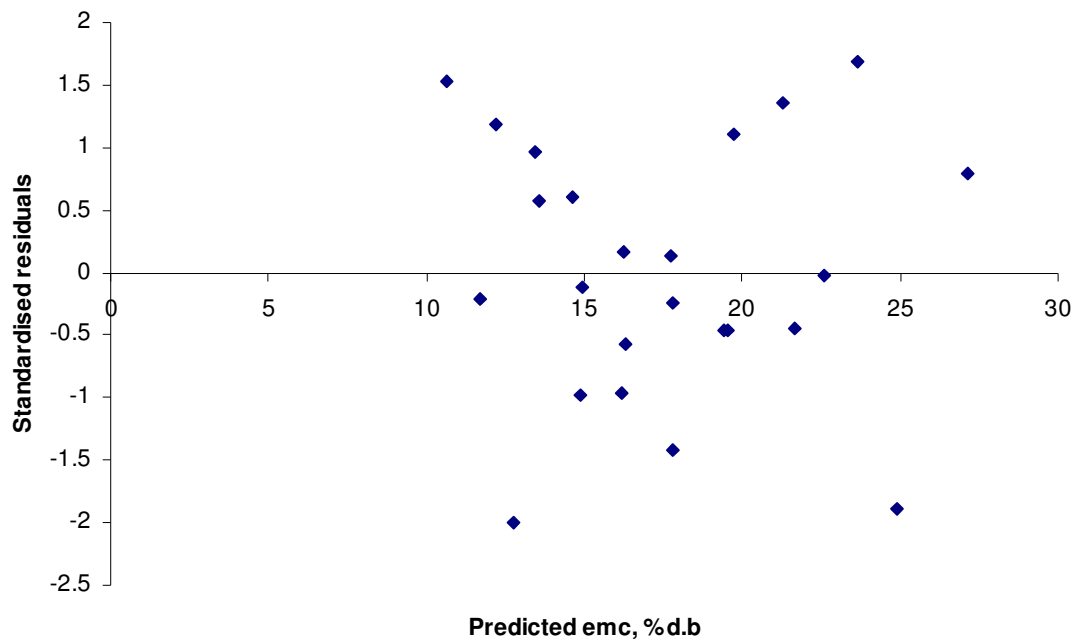


Figure 6: Desorption isotherms predictive model residual plot for modified TMS 97/4763 (MOE)

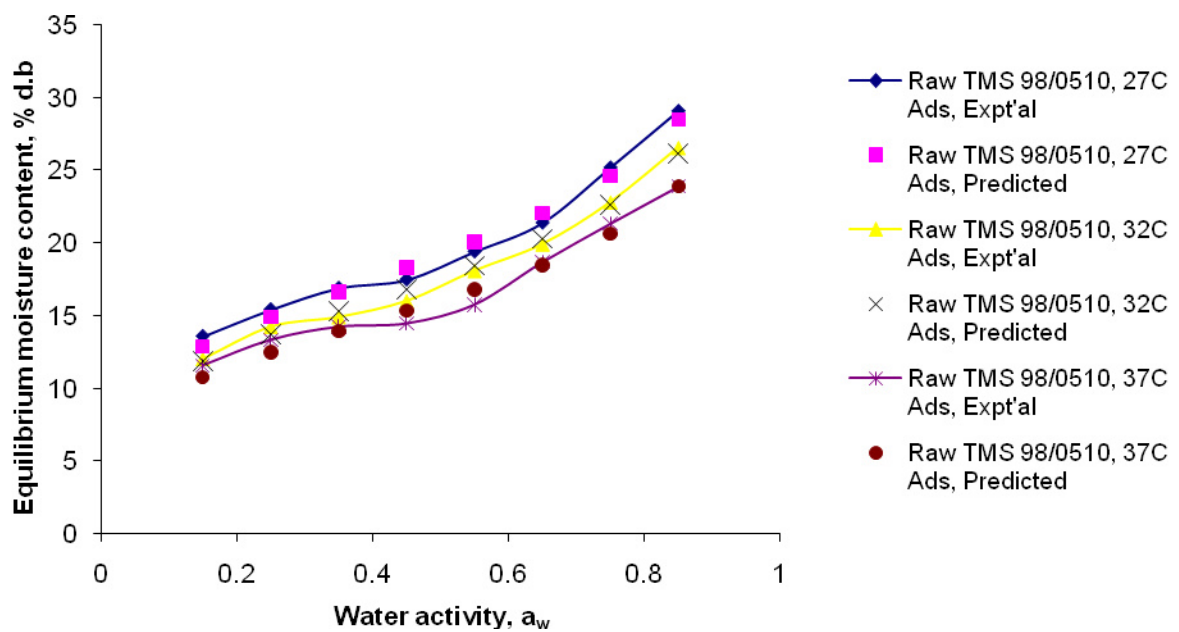


Figure 7: Comparison of experimental and MOE predictive adsorption isotherms (Raw/Native TMS 98/0510)

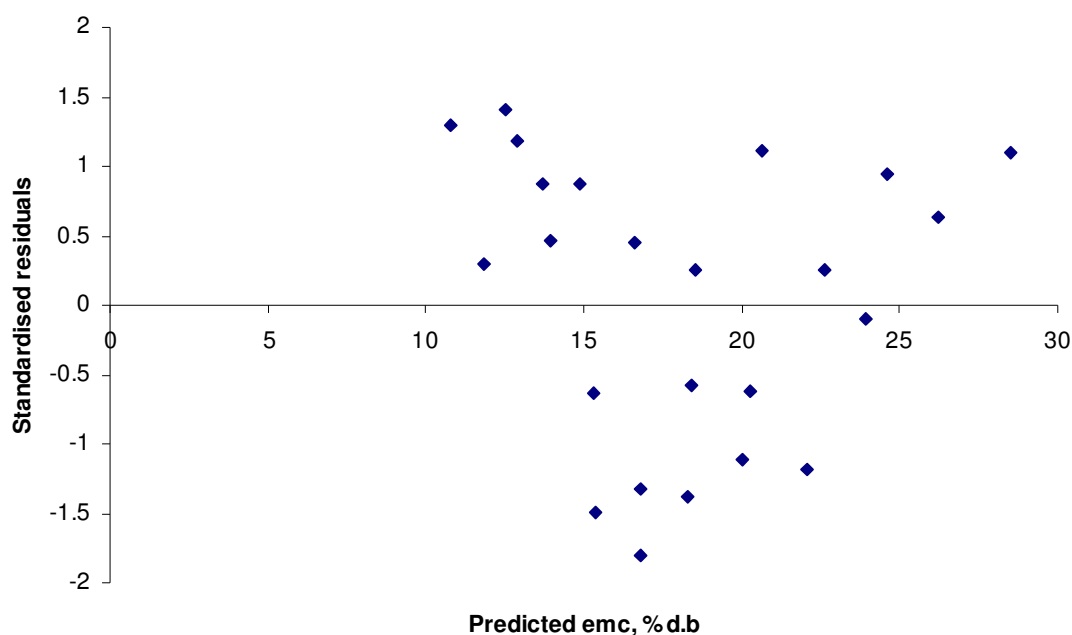


Figure 8: Adsorption isotherms predictive model residual plot for Raw/Native TMS 98/0510 (MOE)

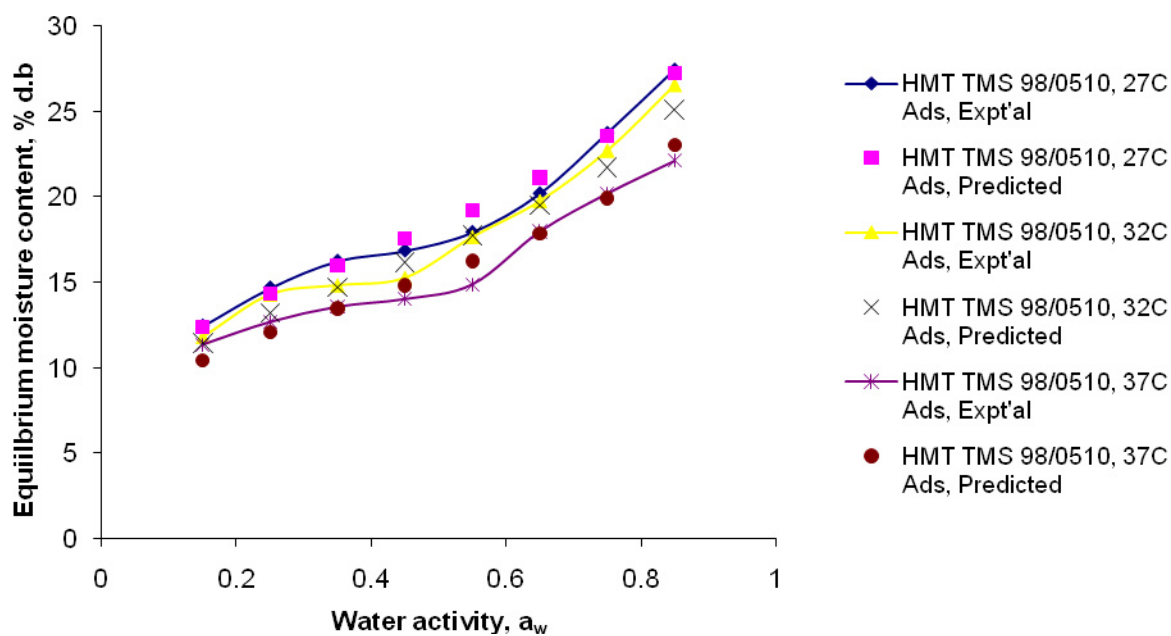


Figure 9: Comparison of experimental and MOE predictive adsorption isotherms for modified TMS 98/0510 starch.

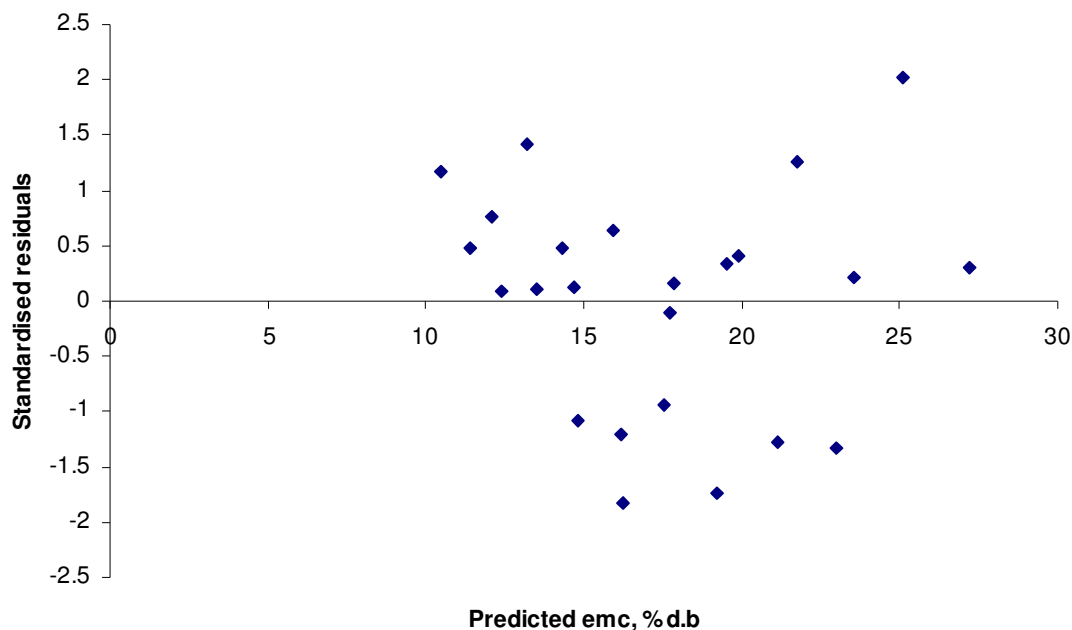


Figure 10: Adsorption isotherms predictive model residual plot for modified TMS 98/0510 starch (MOE)

4. Conclusions

On the basis of results obtained, the following conclusions can be drawn:

- The moisture sorption (adsorption and desorption) isotherms are sigmoidal in shape and are influenced by temperature; the sorption isotherms show the occurrence of moisture sorption hysteresis as obtained for the native starch samples.
- The GAB and MGAB models are not suitable because they could not predict all the isotherms accurately due to effect of heat treatment while the MCE and MHDE models did not converge for any investigated isotherms despite several trials as also obtained for the native starch samples.
- The MOE isotherm is best in describing the hygroscopic properties of the heat-moisture modified cassava starch samples at 27, 32 & 37°C, respectively among six isotherms considered (GAB, MGAB, MOE, MHDE, MCE & MHE) within the range of water activity between 0.15 and 0.85. The MHE model could also predict moisture isotherms of the starch samples, having the same levels of reliability with the MOE model except for the standardised residual plots. The residual plots for the MOE model are more randomised than those obtained in any other investigated models. However, both MOE and MHE models are satisfactorily suitable for fitting EMC data for the native starch samples.

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