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EFFECT OF TEMPERATURE AND HYSTERESIS ON MONOLAYER MOISTURE CONTENT OF MORINGA SEED GRIT DURING STORAGE

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Abstract

The adsorption and desorption isotherm data of stored moringa (*Moringa oleifera*) seed grit within the water activity and temperature ranges of 0.09–0.92 and 20–40°C respectively were used to determine its monolayer moisture content (M_o) using the Brunauer-Emmett-Teller (BET), Guggenheim-Anderson-DeBoer (GAB) and Caurie equations. Results showed that M_o decreased from 3.03 to 2.064 %db (BET); 4.38 to 2.792 %db (GAB); 4.24 to 2.63 %db (Caurie) and 3.007 to 2.011 %db (BET); 4.346 to 2.621 %db (GAB); 4.215 to 2.545 % (Caurie) in adsorption and desorption processes respectively as temperature increases from 20–40°C at specified relative humidity. The GAB model gave the highest M_o values at various temperatures followed by Caurie equation while the BET model gave the lowest values. The M_o were lower in desorption process than the corresponding adsorption values for the three models, thereby indicating the presence of moisture sorption hysteresis. However, one way analysis of variance (ANOVA) showed that there were no significant differences between adsorption and desorption M_o at ($p \leq 0.05$), thereby indicating that moisture hysteresis has a negligible effect on the monolayer moisture content of moringa seed grit.

Keywords: Moringa grit, temperature, hysteresis, monolayer moisture content, BET, GAB, Caurie equation.

1. Introduction

Moringa (*Moringa oleifera*), also known as drumstick or horseradish (Aviara *et al.*, 2015), is a multi-purpose tree crop that is widely cultivated in Nigeria. The plant produces several seeds with oil content ranging from 27 to 38% in the kernels (Aremu and Akintola, 2014). The seed oil is mostly used as raw material in the cosmetic industry and as lubricating oil for machineries (Adejumo and Abayomi, 2012). The large amount of oil in the seed raises concerns about the grit's consistency when stored for an extended period of time. During storage and processing of agricultural materials, it is essential to have information about the effect of environmental conditions on the behavior of the materials. The knowledge of monolayer moisture content is highly important while determining the physical, chemical and structural stability of these materials such as oxidative rancidity, browning and enzymatic reactions (Bell and Labuza, 2000). Monolayer moisture content (M_o) is of utmost importance because it indicates the exact amount of moisture that is strongly adsorbed to specific pores at the surface of the material. It is generally considered as the least moisture content to which food material can be dried to guarantee storage stability. It has been reported that deterioration reactions occurring below this moisture content differ from those that occur at higher values (Aviara *et al.*, 2016). At moisture contents below the monolayer, autoxidation is likely to occur and this results in oxidative rancidity which has direct effect on the



flavour and odour. This observation negates the generally accepted view that the stability of a material increases as moisture content decreases. For most dry foods, the rate of quality loss due to chemical reactions is negligible at the monolayer value (Sudathip *et al.*, 2009). Several researchers have investigated the monolayer moisture content of various agricultural materials such as Aviara and Ajibola (2002) for melon seed and cassava, Ajibola *et al.*, (2003) for cowpea, Aviara *et al.*, (2004) for soya bean, Oyelade *et al.*, (2008) for maize flour, Moreira *et al.*, (2010) for chestnut and wheat flours, Koc *et al.* (2010) for yoghurt powder, Rodriguez-Bernal *et al.*, (2015) for borojo fruit and gum arabic powder, Aviara *et al.*, (2016) for selected crops and Mohammad *et al.*, (2021) for centella powder. However, there is no information about monolayer moisture content of moringa grit and its behaviour under varying temperature and water activity.

2. Methodology

Matured moringa pods were harvested from OYSCATECH farm in Igboora, Oyo State. The kernels were shelled and the seeds were washed to eliminate the impurities. The clean seeds were then dried in GENLAB oven at 60°C until equilibrium moisture content (7.56±0.05% dry basis) was attained. An electric laboratory burr mill (Gourmia, GCG185) was used to grind the dried seeds in similar methods to Alakali and Satimehin (2007). The grit was then dried for seven days in desiccators over concentrated sulphuric acid (H₂SO₄) to get rid of any remaining moisture and kept in airtight containers until they were analyzed (Ogunsina *et al.*, 2014). Equilibrium moisture content was determined for both adsorption and desorption at five temperature levels (20, 25, 30, 35, 40°C) and nine levels of water activities using concentrated tetraoxosulphate (IV) acid (H₂SO₄) as shown in Table 1 to provide constant water activities at the temperature levels.

Glass desiccators (16cm diameter by 16cm height) containing 350ml of concentrated H₂SO₄ to provide constant relative humidity. The desiccators were placed in a thermostatic water bath at the desired temperature and allowed to reach equilibration. 10g of moringa seed grit was placed in petri dish and was used for each experimentation (both desorption and adsorption). The samples were weighed at intervals of four hours until uniform weight is attained thereby getting the equilibrium moisture content. The experiment was replicated three times and the average was recorded for further analysis.

Table 1: Percentage concentrated H₂SO₄ and their corresponding water activity values at different temperature.

Percent H ₂ SO ₄	Temperature (°C)				
	20	25	30	35	40
15	0.9237	0.9241	0.9245	0.9253	0.9261
25	0.8218	0.8218	0.8252	0.8285	0.8317
35	0.6607	0.6651	0.6693	0.6773	0.6846
40	0.5599	0.5656	0.5711	0.5816	0.5914
45	0.4524	0.4589	0.4653	0.4775	0.4891
50	0.3442	0.3509	0.3574	0.3702	0.3827
55	0.2440	0.2505	0.2563	0.2685	0.2807
60	0.1573	0.1625	0.1677	0.1781	0.1887
65	0.0895	0.0933	0.0972	0.1052	0.1135

Bell and Labuza (2000) as reported by Aviara (2020).



Monolayer moisture content

The monolayer moisture content (M_0) of moringa seed grit was evaluated at 20°C, 25°C, 30°C, 35°C, 40°C temperature for the adsorption and desorption arms of their isotherms by applying the two-parameter BET (Brunauer-Emmett-Teller) and Caurie's equations and three-parameter GAB (Guggenheim-Anderson-De Boer) equation to the isotherm data.

Brunauer-Emmett-Teller (BET) Equation

The BET isotherm equation (Brunauer *et al.*, 1938) is a widely used moisture sorption isotherm model and it gives a good fit to data over the region $a_w < 0.45$ (Chirife and Iglesias, 1978). The BET equation in terms of EMC as a function of a_w is stated in Equation 1.

$$M = \frac{M_0 C a_w}{(1-a_w)[1+(C-1)a_w]} \quad (1)$$

where: M is equilibrium moisture content (% db), M_0 is monolayer moisture content (% db), a_w is water activity in the range 0.01-0.45 and C is energy constant related to net heat of sorption. The algebraic manipulation of Equation 1 yields the linear form as shown in Equation 2.

$$\frac{a_w}{(1-a_w)M} = \frac{1}{M_0 C} + \frac{a_w(C-1)}{M_0 C} \quad (2)$$

A plot of $\frac{a_w}{(1-a_w)M}$ against a_w at each temperature within the water activity range of 0.01-0.45 gives the slope as $\frac{(C-1)}{M_0 C}$ and intercept as $\frac{1}{M_0 C}$. From the slopes of the plots from Equation 2 at different temperatures, the BET monolayer moisture content, M_0 and energy constant, C, of the products in adsorption and desorption processes, were obtained. The values of BET adsorption and desorption M_0 were plotted against temperature to obtain the sorption isotherms.

Guggenheim-Anderson-De Boer (GAB) Equation

The three-parameter GAB equation was derived independently by Guggenheim, Anderson and de Boer as stated by Van den Berg and Bruin (1981) and Bizot (1983). In terms of EMC as a function of a_w , the GAB model is as expressed in Equation 3.

$$M = \frac{M_0 C k a_w}{(a - k a_w)[1 - k a_w + C k a_w]} \quad (3)$$

where: M is equilibrium moisture content (% db), M_0 is monolayer moisture content (% db), C and k are sorption constants of the GAB model and a_w is the water activity of the entire range.

Equation 3 was transformed into a polynomial of the second order as shown in Equation 4 and used to determine the constants and monolayer moisture content.

$$\frac{a_w}{M} = \alpha a_w^2 + \beta a_w + \gamma \quad (4)$$



where:

$$\alpha = \frac{K}{M_o} \left(\frac{1}{C} - 1 \right)$$

$$\beta = \frac{1}{M_o} \left(1 - \frac{2}{C} \right)$$

$$\gamma = \frac{1}{M_o C k}$$

Equation 4 was solved by plotting a_w/M versus a_w at each temperature and fitting the plot with a second order Polynomial using Microsoft Excel (2007) in order to determine the values of the coefficient of the quadratic term, α , the linear term coefficient, β , and the constant, γ . The values of GAB adsorption and desorption M_o obtained were then plotted against temperature.

Caurie's Equation

Caurie's monolayer moisture content was computed for the entire range of water activity using Equation 5.

$$\ln \left(\frac{1}{M} \right) = -\ln(M_o C) + \left(\frac{2C}{M_o} \right) \ln \left(\frac{1-a_w}{a_w} \right) \quad (5)$$

where: M is equilibrium moisture content (% db), M_o is monolayer moisture content (% db), a_w is the water activity of the entire range and C is Caurie's constant. Rearranging Equation 5 results in Equation 6.

$$\ln \left(\frac{1-a_w}{a_w} \right) = \ln \left(\frac{1}{M} \right) \left(\frac{M_o}{2C} \right) + \ln(M_o C) \left(\frac{M_o}{2C} \right) \quad (6)$$

A graph of $\ln \left(\frac{1-a_w}{a_w} \right)$ was plotted against $\ln \left(\frac{1}{M} \right)$ to obtain a straight line with slope $\left(\frac{M_o}{2C} \right)$ and intercept $\ln(M_o C) \left(\frac{M_o}{2C} \right)$. The monolayer moisture content, M_o , was then obtained through regression analysis.

3. Results and Discussion

Monolayer moisture contents of moringa seed grit determined using BET, GAB and Caurie models at different temperature levels are presented in Table 2. 3.03 to 2.064 %db (BET); 4.38 to 2.792 %db (GAB); 4.24 to 2.63 %db (Caurie) and 3.007 to 2.011 %db (BET); 4.346 to 2.621 %db (GAB); 4.215 to 2.545 % (Caurie) in adsorption and desorption processes respectively as temperature increases from 20–40°C.



Table 2: Monolayer moisture content of moringa seed grit determined using BET, GAB and Caurie models at different temperatures.

T (°C)	Adsorption			Desorption		
	BET M _o	GAB M _o	Caurie M _o	BET M _o	GAB M _o	Caurie M _o
20	3.030	4.380	4.240	3.007	4.346	4.215
25	2.823	4.145	4.086	2.776	4.120	4.054
30	2.441	3.432	3.606	2.405	3.393	3.424
35	2.200	2.977	2.851	2.168	2.899	2.833
40	2.064	2.792	2.630	2.011	2.621	2.545

It has been reported that the decrease in the monolayer moisture content with increase in temperature is due to reduction in the number of active sites as a result of chemical and physical changes induced by temperature (McMinn and Magee, 2003). Similar reports were given by Oyelade *et al.* (2008) for maize flour, Moreira *et al.*, (2010) for chestnut and wheat flours, Koc *et al.* (2010) for yoghurt powder, Rodriguez-Bernal *et al.*, (2015) for borojo fruit and gum Arabic powder and Mohammad *et al.* (2021) for centella powder. GAB and Caurie monolayer values were close and higher than the corresponding BET values. Timmermann *et al.*, (2001) also reported a similar observation. This occurrence may be as a result of BET model being associated more with moisture sorption at the immediate layer, thereby making it more suitable to predict the monolayer moisture content of dry materials, while GAB and Caurie models are with moisture sorption at the multilayer region, making them more suitable for predicting the monolayer moisture content of intermediate to high moisture foods (Maleki-Majd *et al.*, 2014).

Variations in monolayer moisture content of moringa seed grit with temperatures during adsorption and desorption are presented in Figures 1a and 1b respectively. It was observed that adsorption monolayer moisture values were higher than the corresponding desorption values at the same temperatures and relative humidity. The ANOVA results showing the effects of hysteresis on the monolayer moisture content during adsorption and desorption for BET, GAB and Caurie models are shown in Tables 3a, 3b and 3c respectively. One way analysis of variance showed that there were no significant differences between adsorption and desorption monolayer moisture content at $p \leq 0.05$. This means that hysteresis had not occurred before monolayer moisture content was attained. The monolayer moisture content (M_o) calculated using the three models varied linearly with temperature for both adsorption and desorption processes. Suitable regression models that could be reliably used to predict the M_o of moringa seed grits at different temperature levels are presented in Table 4.

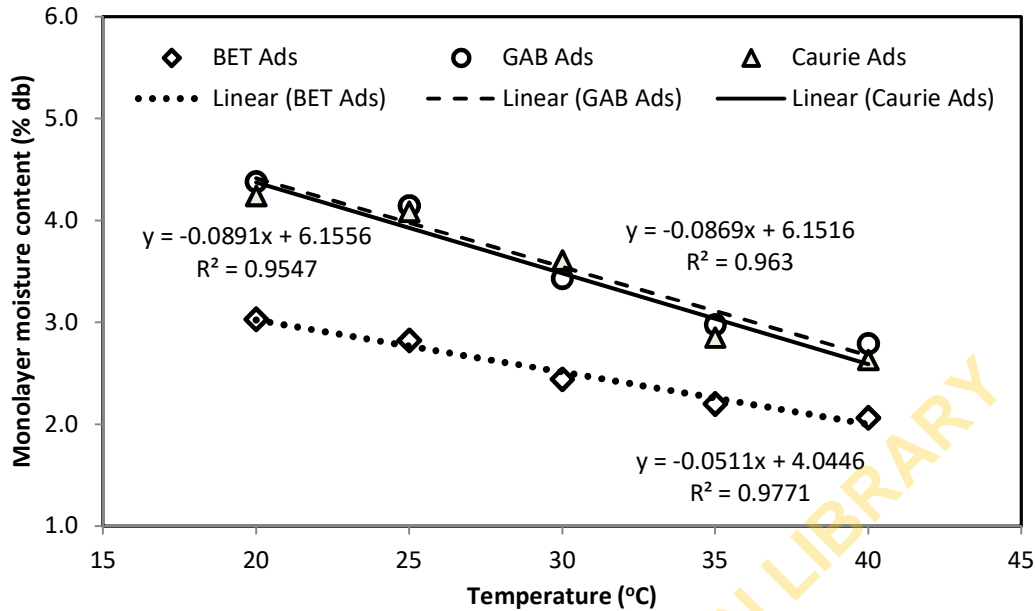


Figure 1a. Variations of adsorption M_0 of moringa seed grit with temperatures.

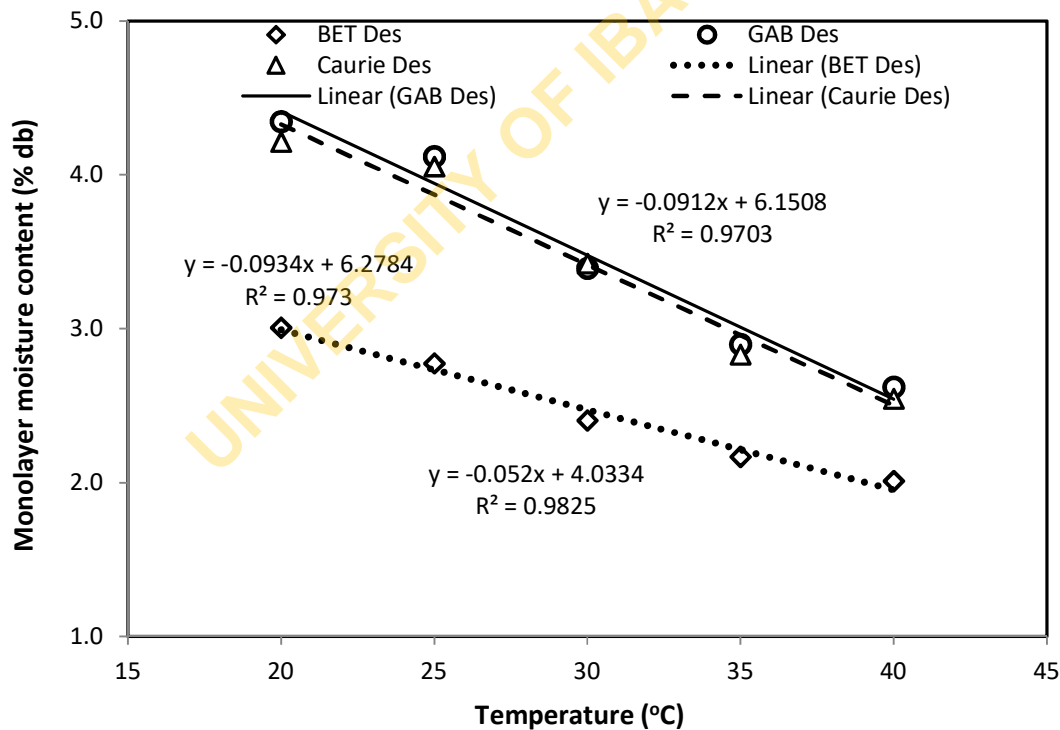


Figure 1b: Variations of desorption M_0 of moringa seed grit with temperatures.



Table 3a: One-way Analysis of Variance for BET Monolayer Moisture Content

Source	SS	df	MS	F	Prob > F
Between groups	.014516067	1	.014516067	0.03	0.8717
Within groups	4.17385402	8	.521731753		
Total	4.18837009	9	.465374455		

Bartlett's test for equal variances: chi-square (1) = 0.0003; Prob>chi² = 0.985

Table 3b: One-way Analysis of Variance for GAB Monolayer Moisture Content

Source	SS	df	MS	F	Prob > F
Between groups	0.003648099	1	0.003648099	0.02	0.8870
Within groups	1.35619048	8	0.16952381		
Total	1.35983858	9	0.151093176		

Bartlett's test for equal variances: chi-square (1) = 0.0008; Prob>chi² = 0.978

Table 3c: One-way Analysis of Variance for Caurie's Monolayer Moisture Content

Source	SS	df	MS	F	Prob > F
Between groups	0.012040908	1	0.012040908	0.02	0.8834
Within groups	4.20210591	8	0.525263238		
Total	4.21414682	9	0.468238535		

Bartlett's test for equal variances: chi-square (1) = 0.0161; Prob>chi² = 0.899

Table 4: Relationship between BET, GAB and Caurie models monolayer moisture content and temperature

Models	Adsorption	Desorption
BET	$M_o = -0.0511T + 4.0446$ $R^2 = 0.9771$	$M_o = -0.052T + 4.0334$ $R^2 = 0.9825$
GAB	$M_o = -0.0869T + 6.1516$ $R^2 = 0.963$	$M_o = -0.0934T + 6.2784$ $R^2 = 0.973$
Caurie	$M_o = -0.0891T + 6.1556$ $R^2 = 0.9547$	$M_o = -0.0912T + 6.1508$ $R^2 = 0.9703$



Conclusions

The adsorption and desorption monolayer moisture content of moringa seed grit was determined at various temperatures using BET, GAB and Caurie moisture sorption isotherm equations. It was deduced that monolayer moisture content decreased with increase in temperature in both adsorption and desorption for the three models which shows that temperature affects monolayer moisture content significantly. GAB and Caurie monolayer moisture contents were greater than those of the corresponding BET monolayer moisture content for both adsorption and desorption. Temperature had significant effect on the monolayer moisture content while moisture sorption hysteresis did not significantly affect the monolayer moisture content of moringa seed grit.

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