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RESEARCH ARTICLE

BEHAVIOR OF AN AGROMATERIAL BASED ON NEEM SEED EXTRUSION RAFFINATE IN RELATION TO WATER - MODELING OF THE ADSORPTION ISOTHERM

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Abstract

The valorization of neem seed extrusion raffinate, a co-product of the oilseed agro-industry that remains largely untapped offers promising prospects for the development of bio-based agromaterials. The water adsorption isotherms of the agromaterial were modeled using six commonly used semi-empirical and empirical mathematical models (GAB, BET, Henderson, Oswin, Peleg). The resulting curve exhibits the characteristic profile of a Type III isotherm according to the Brunauer-Emmett-Teller (BET) classification. This type of isotherm, which reflects weak water-agromaterial interaction at low water activities and adsorption that intensifies as water activity increases, is characteristic of multimolecular (multilayer) adsorption and is consistent with the predominantly fibrous composition of the agromaterial. A comparison of the statistical fit criteria (correlation coefficient, and Mean Squared Deviations) shows that Peleg's four-parameter model most accurately describes the material's adsorption behavior. The three-parameter GAB model ranks second, yet offers the advantage of providing physically interpretable quantities, notably the water content of the monolayer, which is useful for estimating optimal storage conditions. These results confirm the hygroscopic nature of the neem raffinate-based agromaterial, even though adsorption is low (approximately 6.61% at 75% relative humidity) compared to most bio-based materials, and highlight the relevance of the Peleg model for predicting its water behavior under varying relative humidity (RH) conditions. Itprovides a useful database for defining the storage, transport, and application conditions for this new bio-based material, with a view to valorizing agro-industrial byproducts in Africa.

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Introduction:-

The research for bio-based materials derived from the recovery of agro-industrial byproducts is part of a global effort to reduce dependence on fossil fuels and ensure the sustainable management of agro-industrial waste. From this perspective, residual oilseed meal obtained after oil extraction by mechanical pressing or twin-screw extrusion represents a particularly promising raw material for the development of agromaterials, due to its high content of lignocellulosic fibers and protein fractions (Geneau, 2006), as has been demonstrated for sunflower meal processed by twin-screw extrusion and injection molding (Geneau, 2006). Among these byproducts, the raffinate derived from the extrusion of neem (*Azadirachta indica*) seeds remains largely underutilized, even though it offers significant potential for the development of biomaterials, particularly due to its high content of lignocellulosic fibers and residual protein compounds (Diedhiou et al., 2026).

The development of such agromaterials, which are viewed as sustainable alternatives to conventional materials, requires a rigorous characterization of their behavior with respect to water. Indeed, an agromaterial made from a plant-based matrix remains, by nature, hygroscopic: it constantly exchanges moisture with its environment, a behavior that directly affects its dimensional stability, mechanical properties, and durability in service. The characterization of this behavior with respect to water typically involves establishing adsorption isotherms to directly determine the agromaterial's performance properties dimensional stability, mechanical strength, durability, and susceptibility to mold and is therefore a critical parameter for any application.

The study of water adsorption isotherms is the standard method for characterizing this water-agromaterial interaction. It allows us to describe, at a constant temperature, the relationship between the agromaterial's equilibrium water content and the water activity (or relative humidity) of the surrounding environment. Mathematical modeling of these isotherms, using semi-empirical models widely established in the literature (GAB, BET, Henderson, Peleg, Oswin, among others), also provides valuable information on the adsorption mechanisms involved (monolayer, multilayer, capillary condensation) and on thermodynamic parameters useful for predicting the agromaterial's behavior under actual storage or use conditions.

This study fits within this context and aims to characterize the behavior of an agromaterial made from a neem seed extrusion raffinate in the presence of water, through the experimental determination of its adsorption isotherm and the modeling of these data using standard mathematical models, in order to identify the dominant adsorption mechanisms and evaluate its suitability for preservation.

Material and Methods:-

Plant material :-

The agromaterial used in this study was produced by thermopressing a raffinate obtained through aqueous fractionation of neem seeds in a Clextral BC 21 twin-screw extruder; its composition is shown in Table 1. This raffinate is produced by pressing the oil followed by aqueous extraction of azadirachtin using a twin-screw extruder. This agromaterial is thermopressed using a MAPA 50-type hydraulic press with heated plates at a temperature of 200°C under a pressure of 200 kg/cm² for four (4) minutes, using a square mold (5 cm × 5 cm). The resulting agromaterial sheets (Figure 1) are then stabilized in a climate chamber at 60% relative humidity (RH) and 25°C for two weeks before characterization.

Table 1: Composition of the neem seed extrusion raffinate used for shaping the agromaterial by thermopressing

Dry matter (%)		96.59
Proteins (% DM)		3.45
Lipids (% DM)		5.85
Parietal fibers	Cellulose (% DM)	49.41
	Hemicellulose (% DM)	24.67
	Lignin (% DM)	4.87
	Total (% DM)	78.95
Minerals (% DM)		3.70
Other constituents (% DM)		8.05

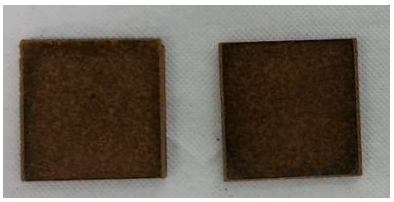


Figure 1: Thermopressed agromaterial in sheet form

Water adsorption of the agromaterial:-

This analysis is performed manually or automatically using the Dynamic Vapor Sorption device (SMS, Great Britain), in accordance with French standard NF X 15-014. It measures the mass of atmospheric water adsorbed by the samples to be analyzed until its reach equilibrium. The samples were previously equilibrated in a climate chamber (60% relative humidity at 25°C) for two weeks. To prevent the growth of microorganisms, a small amount of crystallized thymol is placed in the chambers when relative humidity reaches 60% or higher. Once the samples have finally reached equilibrium, its are weighed again. The water adsorption is then calculated based on the water activity at the 25°C isotherm. The mass and dry matter content of each sample are known. Water uptake is assessed by conditioning the samples for three weeks in a climate chamber containing a saturated potassium chloride saline solution, which gives it a relative humidity of 85% at 25°C in the free space of the chamber (French Standard NF X 15-014). To prevent the proliferation of microorganisms, a small amount of crystallized thymol is also placed in the chamber. When the samples are finally balanced (after approximately three weeks), its are weighed again. The mass of adsorbed water is then calculated based on the water activity (aw) at the 25°C isotherm: aw = 0.60 for a relative humidity of 60% and aw = 0.85 for a relative humidity of 85%. Water uptake is expressed. The number of replicates analyzed is three (3) and the result is obtained by averaging the three (3) trials.

Selection of mathematical models for modeling adsorption isotherms:-

The mathematical models proposed for fitting and modeling the water adsorption isotherms of the agromaterial (Table 2) are well described in the literature. These can be classified based on the number of parameters used or on whether its are constructed based on theoretical or empirical assumptions regarding adsorption phenomena. Two theoretical models were selected: the GAB model, recommended for water adsorption isotherms on biopolymers (McKenna, 1984; De Oliveira et al., 2017; Abdullah et al., 2020; Ocieczek et al., 2022; Tavares et al., 2023) and the BET model, for determining the water adsorption monolayer in water activity ranges below 0.4. The four other models (Peleg, Oswin, Hasley, and Henderson) selected are based on empirical assumptions. With the exception of the BET model, the selection of the five other models corresponds to the water activity range compatible with that of the experimental adsorption isotherms obtained for the agromaterial between 0 and 90% relative humidity.

Table 2: Equations of mathematical adsorption models (Aguirre-Loredo et al., 2016; Kakou et al., 2015; Peleg, 1993; Ribeyre, 2015)

Models	Equations	Range
BET(Brunauer, Emmett and Teller) (1938)	$X_{eq} = \frac{X_0\, C a_w}{[(1-a_w)(1+(C-1)a_w)]}$	0.05<aw<0.4
GAB (Guggenheim, Anderson and De Boer)(1966)	$X_{eq} = \frac{X_0\, K\, C\, a_w}{(1-K\,a_w)(1-K\,a_w+K\,C\,a_w)}$	aw<0.9
Peleg (1993)	$X_{eq} = A\, a_w^B + C\, a_w^D$	aw<0.9
Oswin (1946)	$X_{eq} = A\left(\frac{a_w}{1-a_w}\right)^B$	0.05< aw<0.95
Kakou et al., 2015	$X_{eq} = X_0\left[\frac{-A}{R\,T\,\ln(a_w)}\right]^{1/B}$	0.05<aw<0.80
Anderson (1952)	$X_{eq} = \left[-\frac{\ln(1-a_w)}{A}\right]^{1/B}$	0.05< aw<0.90

Fitting Models to experimental adsorption isotherm data:-

Modeling adsorption isotherms requires statistical methods of regression and correlation analysis. Regression analysis is performed using Excel software, employing the Solver's nonlinear GRG algorithm. The parameters X_0, A, B, C, D, and K of the various models are determined by fitting them to the experimental adsorption curves, minimizing the sum of the Mean Squared Deviations (MSD) (Kakou et al., 2015).

$$\text{MSD} = \frac{1}{n}\sum_{i=1}^{n}\left(X_{eqi,exp} - X_{eqi,pre}\right)^2$$

Xeqi,exp : ith experimental equilibrium moisture content (in % DM);
Xeqi,pre : ith predicted equilibrium moisture content (in % DM);
n : Number of experimental data points.

In order to select the most appropriate model for describing the relationship between equilibrium moisture content, water activity, and temperature, the correlation coefficient (r), related to the coefficient of determination (R^2) is considered the primary criterion for selecting the best equation to describe the adsorption curves. In addition to the correlation coefficient, calculating the values of the Relative Mean Error (RME), the Standard Error of Moisture Content (SEM), and the Residual Error (ei) that is, the difference between a predicted equilibrium value helps justify the choice of model (Table 3).

Table 3: Expression of the statistical parameters used to determine the most reliable model (Aghfir et al., 2005; Benhamou et al., 2010; Mghazli et al., 2016)

Statistical parameters	Equations
Correlation coefficient (r)	$r = \sqrt{\frac{\sum_{i=1}^{n}\left(X_{eqi,pred} - \overline{X_{eqi,exp}}\right)^2}{\sum_{i=1}^{n}\left(X_{eqi,exp} - \overline{X_{eqi,exp}}\right)^2}}$
Mean Relative Error (MRE)	$\text{MRE}\ (\%) = \frac{100}{n}\sum_{i=1}^{n}\left\lvert\frac{\sum_{1}^{n}\left(X_{eqi,exp} - X_{eqi,pre}\right)}{\sum_{1}^{n}\left(X_{eqi,exp}\right)}\right\rvert$
Standard Error of Moisture Content (SEM) or Mean Square Deviations	$\text{SEM} = \sqrt{\frac{\sum_{i=1}^{n}\left(X_{eqi,exp} - X_{eqi,pre}\right)^2}{df}}$
Residual Error (e_i)	$e_i = X_{eqi,pre} - X_{eqi,exp}$

Given the nonlinear regression approximation, a value of $r \geq 0.98$ is considered to indicate a good fit. With regard to the Mean Relative Error (MRE), values below 5% indicate a good fit, values between 5% and 10% indicate a reasonable fit, and values above 10% indicate a poor fit (Aguirre-Loredo et al., 2016). The best model is the one with the highest r value and the lowest values for the Mean Squared Deviations (MSD), the standard error of moisture content, and the residual error (ei) (Kakou et al., 2015). The residual error (ei) indicates whether the differences between the values predicted by the model and the experimental values are minimal. The constants for each model, which allow for comparison among the models, are listed in Table 4.

Table 4: Constants of the adsorption isotherm models for the thermocompressed agromaterial

	BET	GAB	Peleg	Oswin	Hasley	Henderson
X_0	1.714	2.409	-	-	46.936	-
A	-	-	7.046	3.747	28.328	0.161
B	-	-	12.969	0.518	1.600	1.092
C	18061643	7.202	0.881	-	-	-
D	-	-	8.528	-	-	-
K	-	0.889	-	-	-	-

Results and Discussion:-

Behavior of the agromaterial in relation to water:-

The results of the mass analysis of adsorbed water at equilibrium as a function of relative humidity (RH) for the agromaterial obtained by thermopressing the extrusion raffinate of neem seeds made it possible to plot its water adsorption isotherm (Figure 2). Note that the agromaterial's water adsorption remains low at relative humidity levels ranging from 10% to 85%. This agromaterial can be stored at relative humidity levels up to 90% without a significant risk of degradation by microorganisms (water content below 12%). This behavior is attributed to the fibers, which are the major constituents.

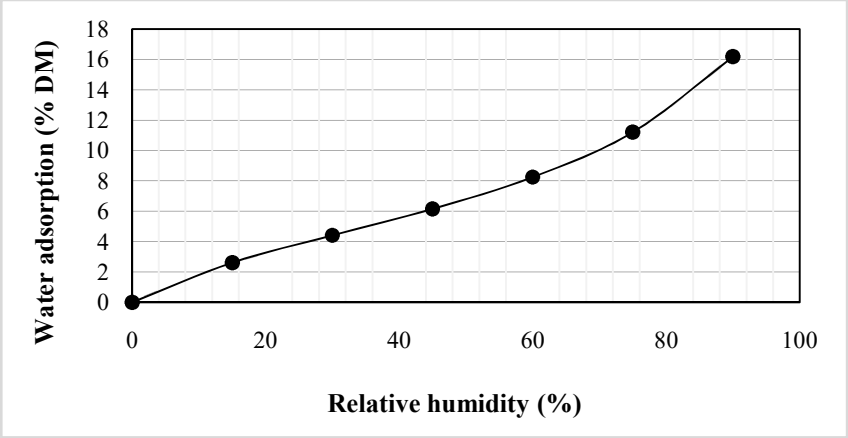


Figure 2: Water adsorption isotherm of the agromaterial

Modeling the adsorption isotherm:-

The results for the mass of adsorbed water as a function of relative humidity (or water activity, aw = RH/100), obtained by minimizing the Mean Squared Deviations (MSD) between the experimental values and those predicted by the models, are listed in Table 5. The results from each model are compared with the experimental results, as shown in the graphs (Figure 3).

Table 5: Experimental values and values predicted by each model as a function of relative humidity

Relative humidity (%)	Experimental values (% DM)	Predicted values (% dry matter) from the models for the thermopressedagromaterial					
		BET	GAB	Peleg	Oswin	Hasley	Henderson
0	0	0	0	0	0	0	0
15	1.251	2.502	1.462	1.325	1.524	1.945	1.005
30	2.491	4.342	2.378	2.441	2.415	2.585	2.065
45	3.578	6.449	3.325	3.502	3.377	3.341	3.313
60	4.566	-	4.606	4.66	4.624	4.416	4.899
75	6.612	-	6.765	6.585	6.623	6.322	7.157
90	11.699	-	11.664	11.702	11.709	11.843	11.39

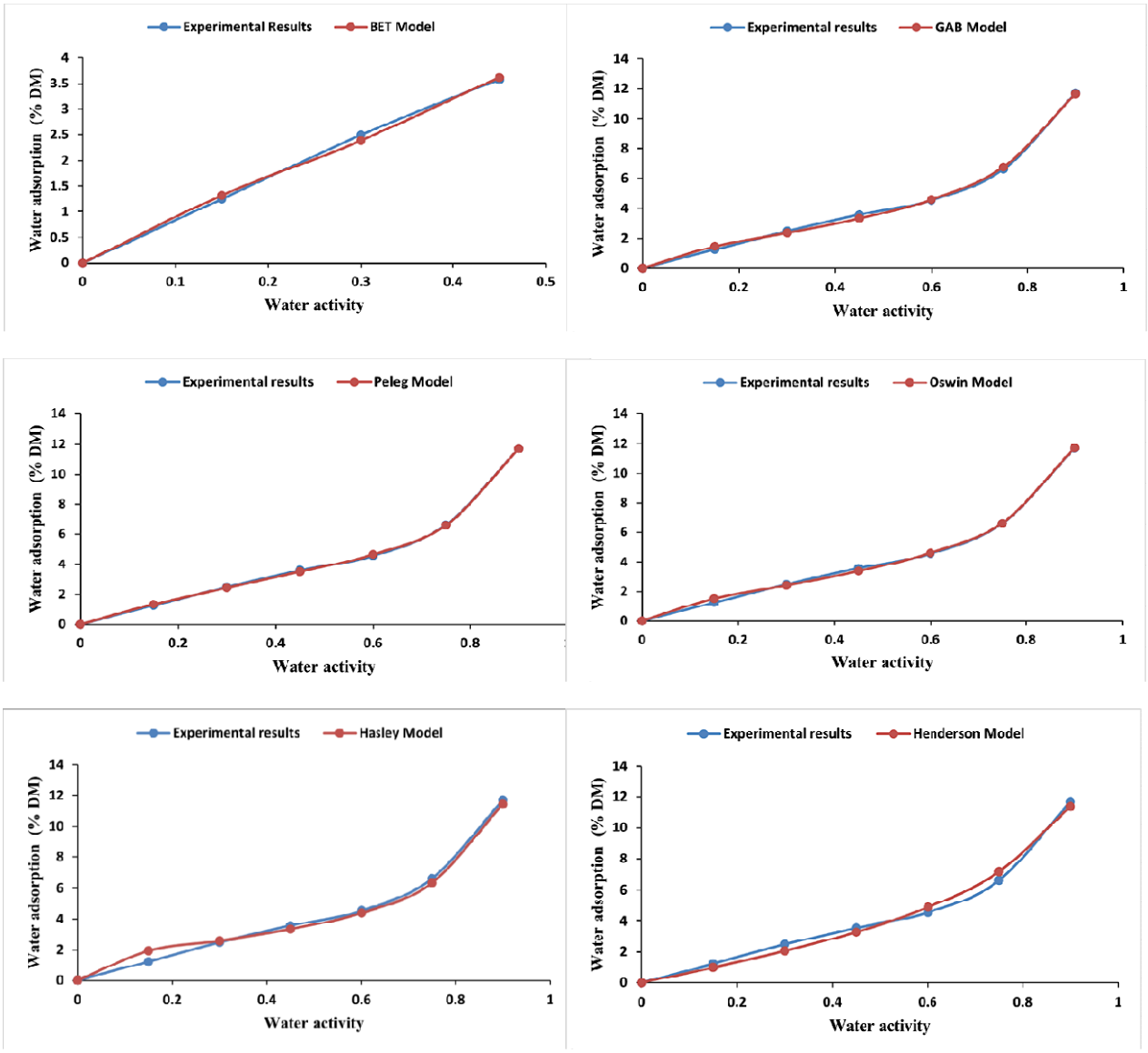


Figure 3: Experimental data on the equilibrium moisture content of thermocompressedagromaterial compared to isotherms predicted by models

The curve from Peleg's model matches closelythe experimental curve, and the curve from GAB's model is virtually superimposable on the experimental curve. The curve from Oswin's model is also almost superimposable on the experimental curve.

Model selection:-

The statistical parameters of each model (Table 6) were used to compare and select the best models. The correlation coefficient r is the primary criterion for model fit.

Table 6: Statistical results for the thermopressedagromaterial

	BET	GAB	Peleg	Oswin	Hasley	Henderson
R	0.947	0.999	1	0.999	0.996	0.996
MRE (%)	18.968	4.572	1.802	4.585	10.690	8.902
SEM	0.568	0.403	0.163	0.282	0.804	0.950

Statistical analysis of the six models shows that the correlation coefficients of these models for the isotherm are all high (>0.98), with the exception of the BET model (0.947) for relative humidities between 0% and 45%. This

indicates that the BET model does not accurately reflect the experimental results. This is further evidenced by high values for the Standard Error of Moisture Content (SEM) (0.568) and the Relative Mean Error (RME) (18.968%).
Thus, in the case of the agromaterial based on neem seed extrusion raffinate, the RME of the Hasley model is greater than 10% (10.69%), indicating a poor fit of the agromaterial's isotherm by this model. The other four models (GAB, Peleg, Oswin, and Henderson) provide a reasonable fit. However, a comparison of the correlation coefficients r, the standard errors of moisture content (SEM), and the MRE shows that Peleg's empirical model has the highest correlation coefficient (equal to 1) and the lowest SEM (0.163), as well as the lowest MRE (1.802%).
Based on these comparisons, Peleg's four-parameter model appears to be the most suitable model for describing the adsorption isotherms of the agromaterial obtained by thermopressing neem seed raffinate. Since the residual error values for the seven experimental data points (Table 7) are small, their distribution appears relatively random. This result validates both the estimation method and the selected model.

Table 7: Residual errors (e_i) of the Peleg model for the various experimental data points

Relative Humidity (%)	0	15	30	45	60	75	90
Agromaterial	0	-0.075	-0.051	0.076	0.093	0.027	-0.003

However, the three-parameter GAB theoretical model also provides a good fit, as its correlation coefficient is greater than 0.98 (r = 0.999) and its Mean Relative Error is less than 5% (4.572%). The water content of the monolayer is given by the constant of the GAB model, which provides a better fit than the BET model (X_0 = 2.409). Among the models studied, Peleg's model best fits the experimental results of the agromaterial's isotherm, with a predicted curve that perfectly overlaps with the experimental one. The GAB model comes in second placein the water adsorption for the agromaterial. The Oswin model, however, provides a good fit.

Analysis and interpretation of water adsorption isotherms using the Peleg and GAB models:-
Peleg's empirical model, which provides a better understanding of the rate of water adsorption by the adsorbent (Peleg, 1993), and GAB's theoretical model, considered suitable for representing the adsorption isotherm of biopolymers (Geneau, 2006), are therefore used in the interpretation of the results.

Shape of the isotherm:-
The values of the parameter C in the GAB equation, as shown in Table 4, are used to characterize the adsorption isotherms. When $C \leq 10$, the isotherm is Type III, whereas when $C \geq 10$, the isotherm is Type II (Kakou et al., 2015). In this study, the value of the C parameter is less than 10 (7.202). Thus, this result indicates that the curve representing the water adsorption isotherm of the thermopressedagromaterial as a function of atmospheric relative humidity (Figure 2) is of Type III. This type of isotherm, characteristic of multimolecular adsorption (gradual thickening of the adsorbed layer), with weak interactions between the porous medium and the adsorbed gas, is consistent with the composition of the agromaterial. The sigmoidal shape of the isotherms at 25°C confirms that the agromaterial consists of biopolymers, particularly fibers (cellulose, hemicellulose and lignin) (Geneau, 2006).The mass of water adsorbed by the agromaterial increases as water activity increases. However, the rate of increase is not uniform. The graph of the equation expressing f'(Peleg) as a function of relative humidity (Figures 4) provides a clear understanding of how the mass of adsorbed water changes as a function of relative humidity (Peleg, 1993).

$$f''(\text{Peleg}) = \frac{d^2X_{eq}}{da_w^2} = (A\,B(B-1)a_w^{B-2}) + (C\,D\,(D-1)\,a_w^{D-2})$$

$f''(\text{Peleg})$: the second derivative of the Peleg model equation ($X_{eq} = A\,a_w^B + C\,a_w^D$);
X_{eq}: Equilibrium adsorption predicted by the Peleg model;
a_w: Water activity;
A, B, C et D: Constants in Peleg's model.

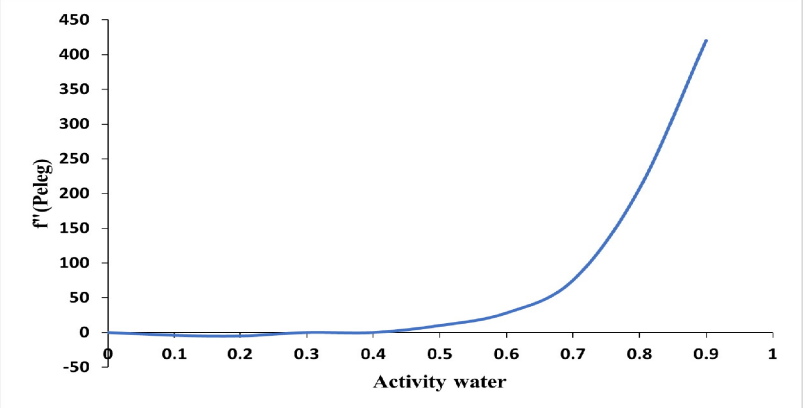


Figure 4: Water adsorption rate (f" (Peleg)) by the thermopressed agromaterial as a function of water activity

The curve plotting f''(Peleg) against water activity shows a positive region at moderate relative humidities (>50%) and a negative region at low relative humidities (<50%). This indicates that the rate of water adsorption as a function of relative humidity (RH) decreases in the range of low relative humidities and increases in the range of high relative humidities. At relative humidities below 40%, we observe a decrease in the rate of water adsorption as RH increases. For RH values below 30%, we can observe a slight inflection point between 0 and 20% RH. This could correspond to correspond to the formation of a monolayer of water on the readily accessible polar groups of the agromaterial. The adsorption rate increases gradually as the relative humidity of the environment rises when it exceeds 50%. Note that at high relative humidity levels (above 80%), the rate of water adsorption by the agromaterial increases exponentially, with a more pronounced increase in the case of this agromaterial. The second inflection point between 60 and 80% may indicate the completion of the formation of the hydration multilayer of the biopolymers (Geneau, 2006) and is more pronounced in the case of the agromaterial.

Water affinity of the agromaterial:-

In the GAB model, which provides a good representation of adsorption isotherms for many plant materials containing biopolymers, the coefficients X_0, C, and K can be related to the agromaterial's affinity for water. Indeed, X_0which measures the amount of water adsorbed to form the monomolecular layer and corresponds to the saturation of each primary adsorption site by a water molecule can be considered an index of the hygroscopicity of the agromaterial and its constituents. It should be noted that this initial adsorption phenomenon corresponds to water strongly bound to free polar sites directly accessible on the surface of the substrate such as starch, proteins, or fibers through the formation of hydrogen bonds or under the influence of Van der Waals forces. C, the Guggenheim constant, relates the formation of the monolayer to the heat of adsorption and can be considered an indicator of the agromaterial's ability to condense water vapor into strongly bound water. And K, a correction factor related to the total heat of adsorption of water multilayers (polymolecular layers that adsorb onto the initial monolayer), can be considered an indicator of the state of water in these multilayers, which are intermediate between solids and liquids. The closer K is to 1, the more the water contained in the multilayer will behave like liquid water (Pérez-Alonso et al., 2005).The thermopressed agromaterial therefore has low water affinity. Indeed, the low X_0 value (2.41) reflects its low hygroscopicity, and the amount of adsorbed watereven at a high relative humidity of 90% RH remains below 12%.

Conclusion:-

This study examined the water behavior of an agromaterial derived from the extrusion raffinate of neem seeds through the experimental determination of its adsorption isotherm and its mathematical modeling. The results show that this isotherm is of Type III according to the Brunauer-Emmett-Teller classification, indicating a low initial affinity between the agromaterial and water molecules at low water activities, followed by adsorption that increases significantly as water activity rises, suggesting a predominant multilayer adsorption mechanism. This type of isotherm, characteristic of multimolecular adsorption, is consistent with the predominantly fibrous composition of the agromaterial.

In terms of modeling, comparing experimental data with various commonly used mathematical models has demonstrated the superiority of the Peleg model, which most accurately describes the agromaterial's adsorption behavior across the entire range of water activity studied. Although the GAB model ranks second, it retains definite practical value due to the physical significance of its parameters, particularly the water content of the monolayer, which remains a key parameter for estimating optimal storage conditions. Taken together, these results confirm the low hygroscopicity of this agromaterial. also make an original contribution to our understanding of the water behavior of bio-based materials derived from underutilized oilseed byproducts, such as neem raffinate.

This work opens up several avenues for research: the study of the desorption isotherm and any hysteresis between the two curves; the determination of thermodynamic adsorption parameters (isosteric heat, sorption entropy) based on the effect of temperature; and the evaluation of the mechanical properties and durability of the agromaterial under conditions of varying humidity. Ultimately, these results could contribute to the development of value-added value chains for neem raffinate in sub-Saharan Africa, in line with a circular economy approach applied to the oilseed agroindustry.

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