MENTORSHIP, SCHOLARSHIP, AND SERVICE: A SPECIAL ISSUE HONOURING PROFESSOR ADAMU N. BABA-KUTIGI, FNIP ON HIS RETIREMENT
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Adsorption of Eosine Y Dye using Modified Eggshell Adsorbent for Sustainable Water Treatment

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ABSTRACT
Synthesis and application of modified eggshell-based adsorbent (MESBA) for the removal of Eosine Y dye from simulated wastewater as alternative technologies for wastewater treatment were investigated. The MESBA was characterised for physicochemical properties with the use of Fourier Transform Infrared (FTIR), Scanning Electron Microscope (SEM), and Energy Dispersive Spectrometer (EDS) as characterization tools respectively. The effect of adsorbent dosage on percentage dye removal was assessed. The isotherm data were fitted to Langmuir and Freundlich isotherm models by employing the linear and non-linear forms of the equations. MESBA has pH (7.80), moisture content (12.90%), ash content (5.80%), volatile matter (9.90%) fixed carbon (71.40%), bulk density (1.33 g/cm^3), particle size (300 µm) and surface area (800 m^2/g). The adsorbent possessed high carbon content with large surface areas in addition to the availability of functional groups within the adsorbent's surface that are good adsorption sites for the removal of ES-Y dyes from wastewater. The adsorbent percentage dye removal was dosage-dependent at optimum dosage of 0.8 g. MESBA has maximum percentage dye removal of 82% at optimum dosage. The isotherm model that fairly described the removal of ES-Y dyes from wastewater was Freundlich isotherm model with coefficient of determination values for both linear (R^2 =0.6746) and non-linear (R^2 = 0.8615) form of the equations. Largely, the prepared adsorbent is efficient, eco-friendly and economically feasible in remediating dye-polluted wastewater, ensuring water reuse and regulatory compliance.

Keywords: Regulatory Compliance, Water Reuse, Freundlich Isotherm, Dosage, Non-Linear

INTRODUCTION
In recent time, with the fast expansion in industrialization and population explosion, the consumption of various types of synthetic dyes has increased rapidly (Giwa *et al.*, 2015; Abdulsalam *et al.*, 2020). It necessitates a constant production of large quantities of dyes to meet the needs of various industrial sectors, nearly 700,000 million tons of several types of synthetic dyes are produced annually. These products are largely used in beverage, food, leather, plastics, pharmaceutical, cosmetics, paper and pulp, and textile industries (Alhawtali *et al*., 2023; Fito *et al*., 2023).

Dyeing processes, especially in textile industries consume a large quantity of water to colour their products, which generates a large amount of countless hazardous coloured wastewater to be disposed in most cases into the environment without definite treatment. It is reported that about 20% of aquatic pollution is traceable to wastewater from the textile industries (Baharim *et al*., 2023). The presence of dyes in water cause damage to the aquatic environment, it reduces the penetration of sunlight, affecting the process of photosynthesis, incessant fluctuation in water temperature, change the potentials of hydrogen ions, it increases the water turbidity and altering the food chain. Furthermore, synthetic dyes are classified as toxic, mutagenic, and carcinogenic to man and aquatic organisms (Alam *et al*., 2021). As a result of the harmful and dangerous effects of dyes, several conventional techniques have been adopted in treatment of dyes from wastewater before reaching aquatic life (Adeleke *et al*., 2023), such as chemical oxidation, chemical precipitation, membrane filtration, coagulation, electrochemical and photo-catalysis. These methods are effective, but with certain limitations such as cost, high skilled technicians, the use of chemicals and high energy consumption. On the other hand, adsorption proves to be efficient and promising due to its low-cost, simplicity of design, ease of operation, eco-friendliness and fast adsorption kinetics (Kuang *et al*., 2020; Rabeie *et al*., 2021).

The chemical formula for Eosine Y (ES-Y), the commonest form of Eosine employed in histology is $C_{20}H_6Br_4Na_2O_5$. ES-Y, is also identified as Acid Red 87 (AR-87). The structure of Eosine Y as shown in Figure 1, is a fluorescent dye, commonly used in histology and cytology as a stain, dyeing of fabrics in textile industry, fluorescent labeling in various biomedical applications, and its potential use in photodynamic therapy due to its ability to generate reactive oxygen species under light exposure (Giwa et al., 2015).

The egg shell (EGS) is the outer covering of egg, which is usually thin, fragile, and bristle. It is primarily composed of calcium carbonate, along with small amounts of protein and other minerals (Ahmed *et al.*, 2021). The colour of an EGS varies, depending on the species of bird that laid it, but it is commonly white or off-white. The EGS serves as a protective barrier that encases the egg's content, providing support and shielding the delicate internal structure. Despite its delicate appearance, the EGS is actually quite strong and capable of withstanding the weight of an incubating bird sitting on it (Ahmed *et al*., 2019a). However, it is still susceptible to cracking or breaking under pressure. The surface of an EGS is covered with tiny pores that allow for the exchange of gases. These pores enable the developing embryo inside the egg to breathe by allowing oxygen to enter and carbon dioxide exits. In terms of texture, the outer surface of an EGS can feel smooth, but it may also have a slight roughness or a grainy texture (Ahmed *et al*., 2021). The texture can vary depending on the species of bird and individuals. Eggshells (EGS) have been utilized for various purposes beyond their role in protecting the developing embryos. They are sometimes crushed into a fine powder and used as a calcium supplement or fertilizer. EGSs have also been employed in art and craft, as well as in traditional remedies and folk practices. Finally, EGSs have been employed in various environmental remediation technologies such as removal of heavy metals, adsorption of radioactive meals, adsorption of total nitrogen, fluoride and phosphorus from wastewater (Lu *et al*., 2017).

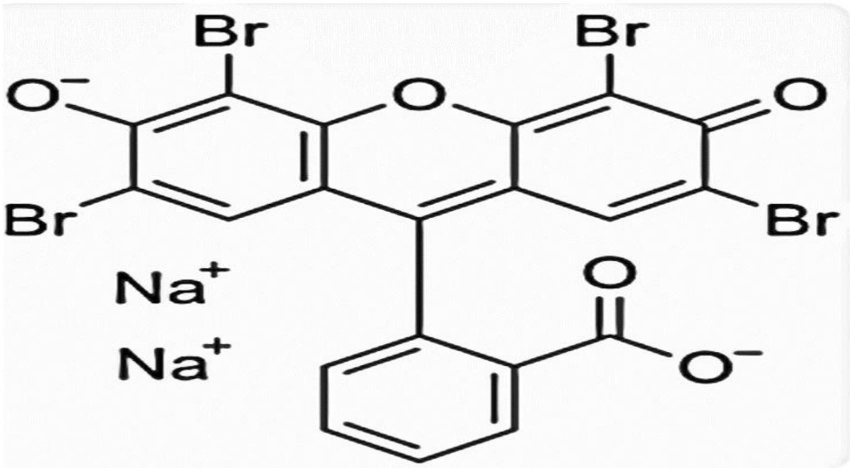

Figure 1: Molecular Structure of Eosine Y Dye

MATERIALS AND METHODS

Procurement and Preparation of Eggshell

Eggshell (ES) was obtained from PECA fast food Otuoke, Ogbia LGA, Bayelsa State, and was authenticated by Mr. Okpata Sunday (Voucher's number FUO-082), Department of Biological Sciences, Federal University Otuoke, Bayelsa State, Nigeria. The ES was washed thoroughly to get rid of dirt, debris and stones, followed by sun-drying to remove excess moisture. The ES was further oven dried at a temperature of 100 ^oC until a constant weight was attained. The oven-dried ES was subjected to crushing, and further ground to the desired particle size based on our previous works (Onawumi *et al*., 2021). The modification was carried out according to the method described by (Bello *et al*., 2017), with slight modification, and resultant product is regarded as modified eggshell-based adsorbent (MESBA). The modification was carried out by increasing the molarity of orthophosphoric acid (H_3PO_4) from 0.3 M to 0.5 M. A precise weighed 14.0 g of crude sample of ES was placed in a conical flask containing 250 ml of 0.5 M H_3PO_4. The substance of the container was entirely ground and warmed on a hot plate until a thick past was formed. The past of ES was moved into the crucible which was set in a furnace and warmed at 500 ^oC for 600 minutes. The sample was allowed to cool and washed severally with distilled water to a pH of 7.10 and further oven-dried at 100 ^oC for 4 h, and the resultant sample is code named, MESBA. This was put in an air-tight sealed glass bottle for further usage.

Characterization of MESBA

The Scanning Electron Microscope (SEM) of MESBA) adsorbent was taken with a ThermoFisher Scientific (Axia ChemiSEM, 120 x 120 mm^2 5-axis motorized eucentric, USA). The surface of the MESBA was considered with the microscope run at 10.0 kV. The samples were coated with a 10 nm thick layer of gold.

Fourier Transform Infrared Spectrometer (FTIR) (Thermo Fisher Scientific, Nicolet iS50, USA) technique of analysis was employed to study the functional groups in MESBA. The infrared spectra of MESBA were obtained by using MESBA mixed with potassium bromides at ratio 1;100 in a mortar and pestle. The mixture was taken in a disc of specific dimension to form pellet by pressing with a handpress machine, placed on the sample holder of IR spectrometer (Agilent Technologies, 4100 ExoScan, California, USA) operated at spectra range 4000 – 400 cm^{-1}.

The physicochemical properties and proximate composition of MESBA that were determined include: pH, moisture content (MC), volatile matter (VM), ash content (AC), fixed carbon (FC), bulk density (BD), surface area (SA), particle size (PS) by employing the methods described by Onawumi et al. (2021).

Preparation of Eosine Y Dye (ES-Y)

Furthermore, ES-Y was procured from Sigma Aldrich Chemical, Germany. ES-Y (1000 mg) was correctly weighed into 250 ml conical flask, and small quantity of distilled water was added and stirred uninterruptedly for total dissolution. The dissolved dye solution was transferred into 1000 cm^3 standard volumetric flask and carefully made up to mark with distilled water. The aqueous ES-Y solution was standardized on a UV-visible spectrophotometer (Agilent Technologies, Agilent 8453, California, USA). The ES-Y wavelength at maximum (λ_{max}) was found to be 525 nm, and was used to determine the absorbance of the serially working solutions of dye prepared from the stock solutions are: (10, 20, 30, 40, 50 mg/L). In addition, the absorbance of the ES-Y effluent solution after the adsorption processes was measured to provide means of evaluating the percentage dye removal (% R) by the adsorbent at a particular at equilibrium.

Effect of Adsorbent Dosage on the Adsorption Process

A batch adsorption study was carried out on the influence of contact on ES-Y dye removal using 100 ml dye solution in a 250 ml conical flask placed on a water bath shaker at a shaking speed of 150 rpm. The optimum initial dye concentration was 30 mg/L, 60 minutes contact time, 60 ^oC, and the adsorbent dosage was varied from (0.2 to 1) g at 0.2 g interval. After the adsorption experiments, the adsorbent was separated from dye effluent by centrifugation using centrifuge at room temperature at 2000 rpm.

The absorbance of aliquot part of dye effluent was read on UV-visible spectrophotometer and the concentration was interpolated from the working graph. Further, the percentage of ES-Y adsorbed on the surface of was determined according to Jabar and Odusote (2020), as reported by Alhawtali et al. (2023) as shown in equation 1.

$$\% R = \frac{(Co - Ce)100}{C_o} \quad (1)$$

The amount of ES-Y adsorbed per unit weight of MESBA was calculated as shown in equation 2 and 3 respectively:

$$q_e = \frac{(Co - Ce)V}{W} \quad (2)$$

$$q_t = \frac{(Co - Ct)V}{W} \quad (3)$$

% R = Percentage ES-Y dye removed
C_o = Initial dye concentration (mg/mg)
C_e = Equilibrium dye concentration (mg/g)
C_t = Concentration at time (t)
V = Volume of dye solution (L)
W = Weight of the adsorbent (MESBA) (g)

Effect of pH on ES-Y Dye

A batch adsorption study was carried out on the influence of contact on ES-Y dye removal using 100 ml dye solution in a 250 ml conical flask placed on a water bath shaker at a shaking speed of 150 rpm. The optimum initial dye concentration was 30 mg/L, 60 minutes contact time, 60 ºC, and the pH was varied from (2 to 10). After the adsorption experiments, the adsorbent was separated from dye effluent by centrifugation using centrifuge at room temperature at 2000 rpm.

The absorbance of aliquot part of dye effluent was read on UV-visible spectrophotometer and the concentration was interpolated from the working graph. Further, the percentage of ES-Y adsorbed on the surface of was determined according to Bello *et al*. (2017) as reported by Onawumi *et al*. (2021) shown in equation 1.

Langmuir Isotherm

From Langmuir isotherm model, the linear and nonlinear equations are given 4 and 5 respectively as;

$$\frac{C_e}{q_e} = \frac{1}{K_L Q_o} + \frac{C_e}{Q_o} \quad (4)$$

The linear form is given as;

$$q_e = \frac{K_L Q_o C_e}{1 + K_L C_e} \quad (5)$$

A plot of $1/q_e$ against $1/C_e$ gives a straight line with $1/Q_0$ as slope and $1/K_L Q$

Freundlich Isotherm Model

Freundlich isotherm model linear and nonlinear equations are given in equation 6 and 7 as;

$$\log q_e = \log K_F + \frac{1}{n} \log C_e \quad (6)$$

The plot of q_e against C_e gives the model parameters,
The linear form of Freundlich is given thus;

$$q_e = K_F C_e^{\frac{1}{n}} \quad (7)$$

Using the logarithmic form of Freundlich isotherm model given equation 7, a plot of
Log q_e against log C_e gives a straight line with 1/n as the slope and log K_F as intercept, from which K_F is calculated.

Isotherm models	Linear	Nonlinear	Plots	Reference
Langmuir	$\frac{C_e}{q_e} = \frac{1}{K_L Q_o} + \frac{C_e}{Q_o}$	$q_e = \frac{K_L Q_o C_e}{1 + K_L C_e}$	$\frac{1}{q_e}$ vs $\frac{1}{C_e}$	(Bello *et al*., 2017)
Freundlich	$\log q_e = \log K_F + \frac{1}{n} \log C_e$	$q_e = K_F C_e^{\frac{1}{n}}$	$\log q_e$ vs $\log C_e$	(Ayawei *et al*., 2017)

RESULTS AND DISCUSSION

Results

Table 1: Physicochemical Properties and Proximate Compositions of Modified Eggshell-Based Adsorbent (MESBA)

S/No.	Parameters	Mean ± SE
1	pH	7.80
2	Moisture content (%)	12.90
3	Volatile matters (%)	9.90
4	Ash content (%)	5.80
5	Fixed carbons (%)	71.40
6	Bulk density (g/cm^3)	1.33
7	Surface area (m^2/g)	800.00
8	Particle size (µm)	300.00

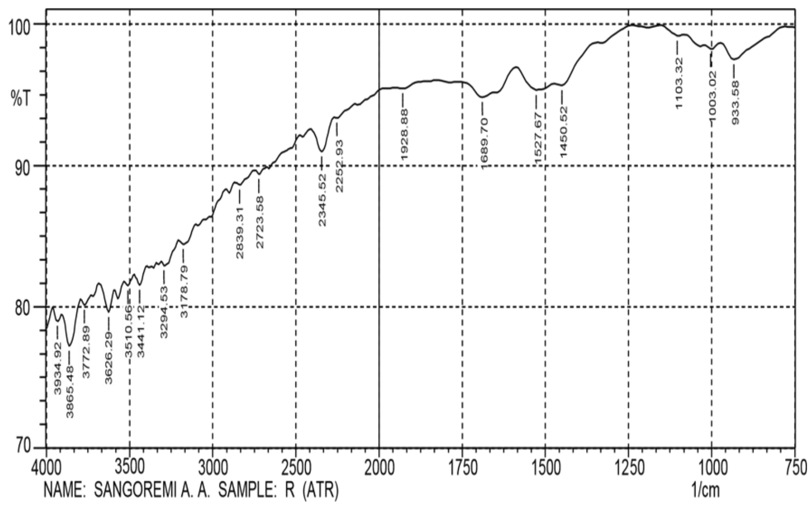

Figure 2: FTIR of Modified Eggshell-Based Adsorbent (MESBA)

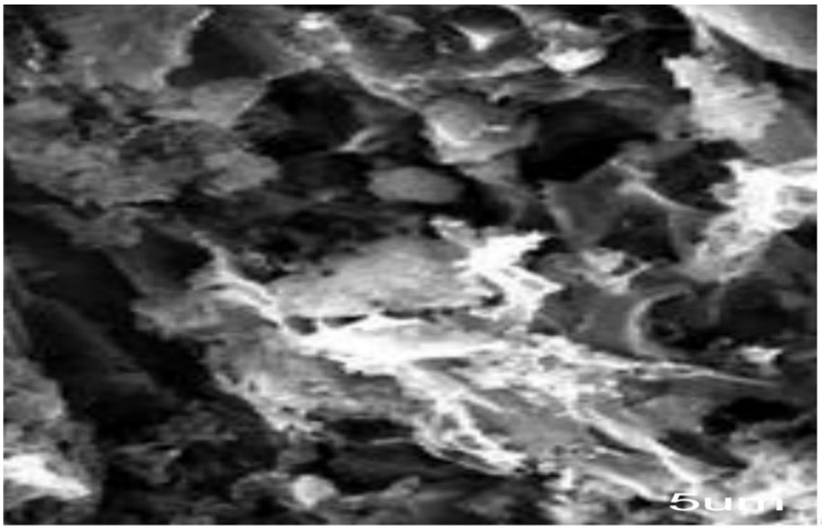

Figure 3: SEM Micrograph of MESBA

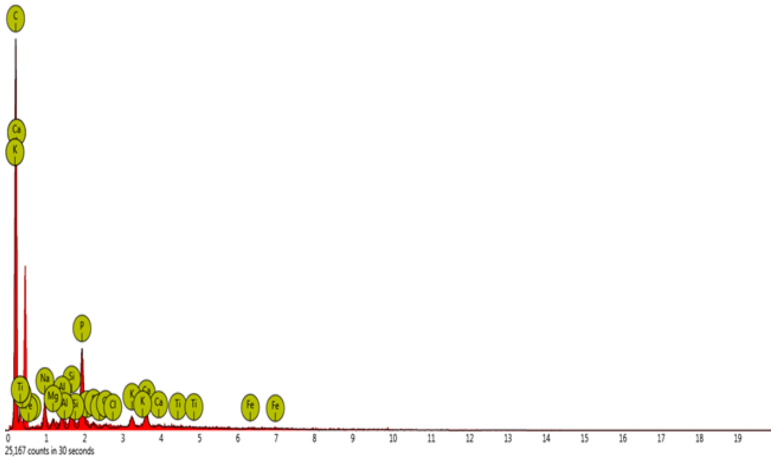

Figure 4: EDX Spectra of MESBA

Table 2: Elemental Composition of Modified Egg Shell Adsorbent

Element Number	Element Symbol	Element Name	Atomic Conc.	Weight Conc.
6	C	Carbon	93.67	84.43
20	Ca	Calcium	2.41	5.63
15	P	Phosphorus	1.15	1.99
19	K	Potassium	0.65	1.98
14	Si	Silicon	0.81	1.71
13	Al	Aluminum	0.58	1.18
12	Mg	Magnesium	0.32	0.58
26	Fe	Iron	0.12	0.49
16	S	Sulfur	0.17	0.41
17	Cl	Chlorine	0.13	0.35
22	Ti	Titanium	0.00	0.00

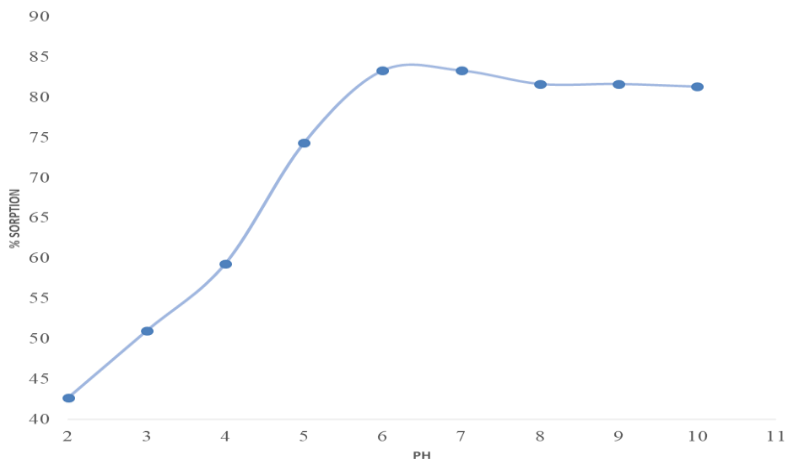

Figure 5: The Effect of pH on the percent Sorption of ES-Y onto MESBA

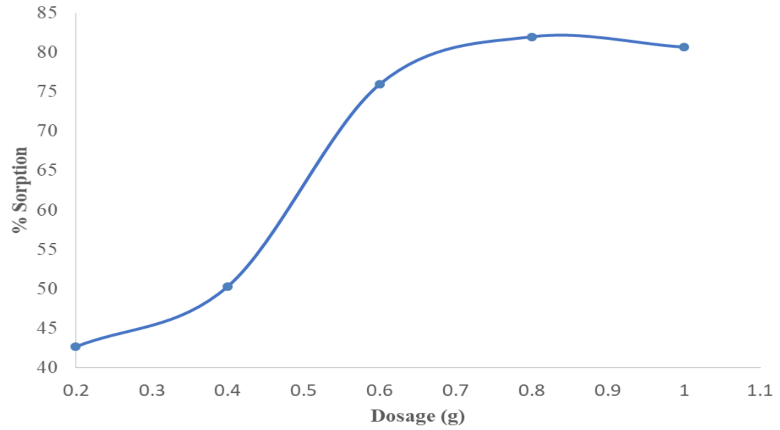

Figure 6: Relationship between Percent Sorption of ES-Y Dye onto MESBA Dosage

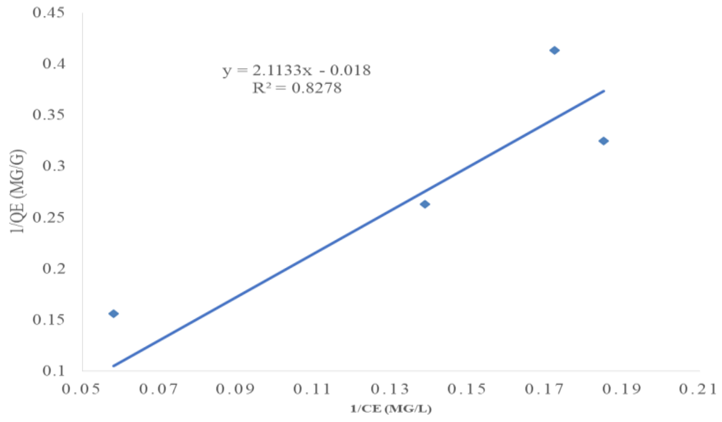

Figure 7: Langmuir Adsorption Isotherm for Eosine Y Dye

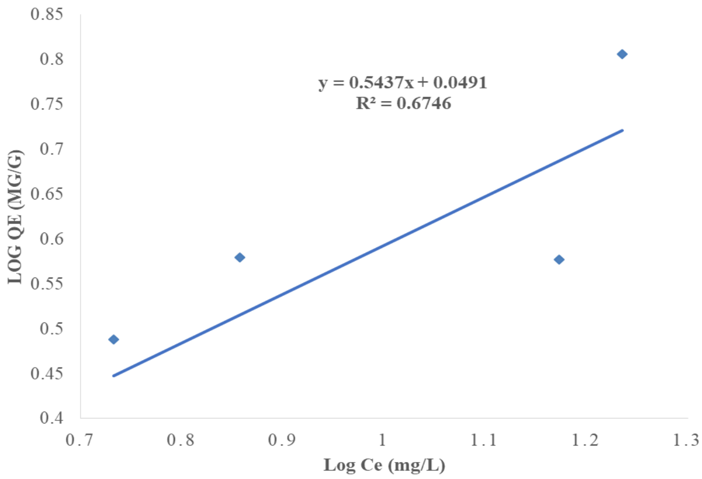

Figure 8: Freundlich Adsorption Isotherm of Eosine Y Dye

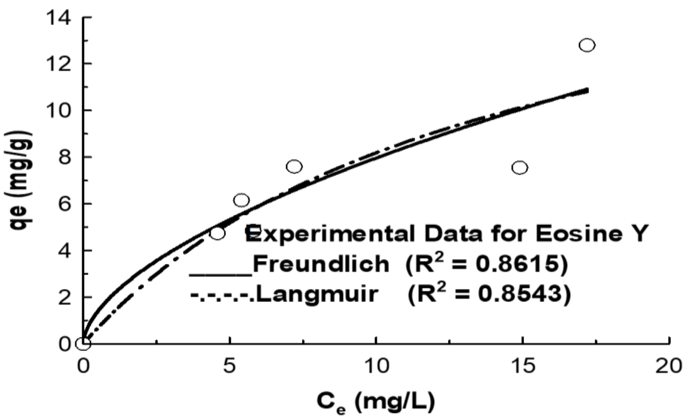

Figure 9: Nonlinear Langmuir and Freundlich Adsorption Isotherm for Eosine Y Dye

Discussion

Table 1 presents the physicochemical and proximate properties of the prepared adsorbent under study with the following outcomes: pH (7.80), moisture content (12.90%) volatile matter, (9.90%) ash content (5.80%), fixed carbon (71.40%), bulk density (1.33 g/cm^3), surface area (800 m^2/g), and particle size (300.00 µm). The physicochemical properties conform to those reported in the literature (Ajala and Ali, 2020), and as well in agreement with quality threshold range of the adsorbents as recommended by National Industrial Standard of Indonesia (SII) No. 0258-79, and National Standard of Indonesia (SNI) No. 06-3730-1995. Figure 2 shows the FTIR spectra of MESBA. The characteristic functional groups on the adsorbent's surface were identified from the spectra. The peak at 3865.48 cm^{-1} indicates the presence of an O-H stretching vibration of phenol or alcohols in lignin and cellulose of MESBA. Other peaks in the spectrum of the adsorbent are 3441.12, 3362.29, 2345.52, 1689.70, 1450.52, 1003.02, and 933.58 cm^{-1}. These are due to N-H, N-H, C≡N, C=O, CH_3, C-H, and CCl stretch respectively (Nandiyanto *et al.*, 2019).

Scanning Electron Micrographs (SEM)

Figure 3 shows the Scanning Electron Micrograph of the MESBA. The SEM image of the prepared adsorbent reveals the presence of holes on its surface. These cavities are available pores at the surface, where ES-Y dye molecules are captured from aqueous solution. The captured dye molecules traveled to fill the available pores on the adsorbent by diffusion of molecules from the aqueous solution to the MESBA's surface through the boundary layer. This was followed by migration of dye molecules from the adsorbent surface to the inner pores and finally adsorbed at the available vacant active sites on its surface. The adsorption of ES-Y on the surface of MESBA might be physical adsorption (physisorption), through mechanical adhesion of adsorbates on adsorbent. This agrees with our previous observations and that of other researchers (Unuabonah *et al.*, 2017).

Electron Dispersive X-ray Spectroscopy Analysis (EDX)

The EDX analysis is a method of elemental analysis associated with electron microscopy based on the generation of characteristic x-rays that divulges the presence of element present in a sample (Munagapati *et al*., 2018). Figures 4 reveals the EDX spectra of MESBA, while Tables 2 reveals the elemental composition of the prepared adsorbents respectively. The carbon content was 84.43% which demonstrates that the process of activation has enriched the carbon contents in the adsorbent. Other elements present in percentage atomic weight include: calcium (5.63 %), phosphorus, potassium and silicon are 1.99, 1.98 and 1.71% respectively, while others are in trace amount (Ushedo *et al.*, 2022).

Effects of Operational Parameter

The Effect of pH

Figure 5 discloses the effect of pH on percentage sorption. Several authors have submitted that pH is one of the important parameters affecting the amounts and types of electrostatic charges on the surface of the adsorbent, photo-degradation of dyes, ionization, and chemical speciation of the adsorbates (Ajala and Ali, 2020). From Figure 5, at low pH, the dye uptake was relatively low (42.67%). This is attributed to the competition of the cationic ES-Y dye molecules with protons (H^+) present in the acidic region. On the other hand, when the pH shifts to less acidic region, the dye removal efficiency increases significantly from 42.67 to 83.33%. This is attributed to the reduction in the acidic effect, thereby enhancing the electrostatic attraction between the functional groups on the surface of MESBA and ES-Y dye molecules for higher pH range (Budinoval et al., 2011). The optimum pH range for the present studies is 6 – 7 with maximum percentage dye removal of 83.33%.

Effect of the Adsorbent Dosage

Effect of Adsorbent dosage.

Figure 6 reveals the ES-Y percent sorption (%S) against dosage of MESBA. It was clear that an increase in dosage of the prepared adsorbent from 0.2 to 1.0 g led to an increase in ES-Y dye molecules removal from the wastewater, 42.67% to 82%. This is attributed to the increase in adsorption surface areas of MESBA, due to the availability of more vacant active sites on the surface of the adsorbent as dosage increases (Bello *et al*., 2017). The equilibrium was reached at 0.8 g of the adsorbent with percentage sorption of 82%, thereafter, there was a marginal decrease in percentage adsorption, which suggests that at this stage, the vacant adsorption sites were fully saturated that they could no longer hold more molecules of AS-Y dyes from the aqueous solution. This is a state of dynamic equilibrium, where the rate of adsorption is equal to the rate of desorption.

Adsorption Isotherm Model

Experimental isotherm data obtained in this study were analysed with two theoretical models: Langmuir, Freundlich, Jovanovich (two-parameter isotherm models) by employing both the linear and nonlinear model equations. The plots of the fittings of these theoretical models compared to experimental data are shown in Figures 7, 8 and 9 respectively. The heterogeneity factor (n) in the Freundlich model can be used to indicate the degree of favorability of adsorption (Abdullahi *et al*., 2022). The Freundlich constant, n should have values lying in the range 1 to 10 for classification as favourable adsorption. The value of n for the adsorbent used in the current study is greater than 1 but less than 10 indicating that the adsorption of ES-molecules onto MESBA is favourable. The same observation was made by (Unuabonah *et al*., 2022) on facile synthesis of new amino-functionalized agro genic hybrid Composite Clay adsorbents for phosphate capture and recovery from water. From the analysis of all the isotherms used in this study with respect to coefficient of determination for both linear (R^2 =0.6746) and non-linear (R^2 = 0.8615), and that the equilibrium data for the adsorption of ES-Y onto MESBA was best described by Freundlich isotherm model which predicts non-ideal and reversible adsorption on heterogeneous adsorption sites with no formation of monolayer on the adsorbent's surface (Nwabanne *et al*., 2022; Kachako *et al*., 2023). Furthermore, it was observed that the value of coefficient of determination of nonlinear isotherm model is greater than the linear form. The bigger (R^2 = 0.8615) value of nonlinear modeling as against linear method (R^2 =0.6746) in this study came to bear because studies had shown that the error structures of experimental data were usually changed during transformation of adsorption isotherm into their linearized forms (Unuabonah *et al*., 2017). It was against this backdrop that non-linearized regression analysis became inevitable, since it provided stress-free mathematically rigorous methods for determining adsorption parameters using original form of isotherm equations (Unuabonah *et al.*, 2017).

CONCLUSION

The prepared adsorbent possesses high carbon contents, low inorganic contents, high surface area, and heterogenous pore structures that make it a viable precursor for the removal of ES-Y from aqueous solution. The dye removal from the wastewater was pH and dosage-dependent with maximum removal efficiency of 82% and 83.33% at optimum conditions. The isotherm model that fairly described the removal of ES-Y dye from the wastewater was Freundlich isotherm model. The prepared adsorbent stands effective in remediating the dye contaminated wastewater to useable status.

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