

Research Article

Adsorptive Ability of Ethylene-1,2-Diamine Functionalized Jackfruit Seeds Adsorbent for Cationic Rhodamine B and Malachite Green Dyes from an Aqueous Media

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Abstract— The continued use of dye laden water is detrimental to human, animal wellbeing and the entire ecosystem even at permissible limits. Several decontamination techniques are available but are expensive and ineffective. Adsorption technology has gained interest because of its high efficiency, design simplicity and availability of materials. The study modified raw biomass using thionyl chloride and ethylene-1,2-diamine for uptake of Rhodamine B (RhB) and Malachite green (MG) dyes. Both raw and functionalized adsorbent were analyzed using FT-IR and FE-SEM. The FT-IR results showed functional groups of carbonyl ($-C=O$), carboxylate ($-COO^-$) (raw), hydroxyl (OH^-) and additional anchored amine ($-NH_2$) groups (functionalized adsorbent) as important sites for cationic dyes removal. The SEM results showed more porous adsorbent surface which improved its sorption ability. The maximum uptake obtained at optimal $pH=10$, 40 minutes, 35 mg and 40 mg/L was 22.61 ± 0.15 mg/g and 21.76 ± 0.09 mg/g for RhB and MG dyes respectively. The adsorption fitted Langmuir isotherm model ($R^2 > 0.95$) dictating a chemisorption process. The time data fitted pseudo second order model in explaining the sorption rate. The findings of the study revealed the potential of the functionalized adsorbent dyes adsorption from aqueous solution with no leaching of the secondary pollutants.

Keywords— Ethylene-1,2-diamine (EDA), Rhodamine B (RhB), Thionyl chloride ($SOCl_2$), Malachite green (MG), Chemisorption

1. Introduction

The continued demand for dyed products from pharmaceuticals, textiles, pulp and paper, tanneries, packed food industries, plastics, paint, electroplating, petroleum and cosmetics industries continues to grow every dawn [1]. Their extensive usage in diverse applications has generated coloured wastewater which is then discharged to the nearby water bodies [2]. The dye wastewater is detrimental to the wellbeing of an ecosystem when disposed prior to its purification [3]. Their presence in water causes the water coloring limiting the photosynthetic functions of the aquatic plants and fauna [4]. Most of the dyes are of synthetic origin with complex aromatic structures and non-biodegradable [5]. Rhodamine B and Malachite green dyes are examples of such dyes reported to be lethal, mutagenic and cancer-causing due to heavy metals and chlorides in their aromatic structures [6]. In addition, the dyes cause health issues such as allergy, hard breathing, skin irritation, sweating, nausea, confusion vomiting, mutations, high blood pressure and headache [7]. Their decontamination before discharge into the nearby surrounding is necessary. This study, therefore, explores the

use of the EDA functionalized adsorbent from Jackfruit seeds wastes in the removal of Rhodamine B and Malachite green from an aqueous media.

2. Related Work

A number of conventional techniques such as coagulation-flocculation, ion exchange, membrane filtration, electrochemical process, amongst others have been reported for the removal of dyes from aqueous solutions [8]. However, these methods are limited due to intensive energy requirement, hazardous by-products formation, operational costs and limited adaptability to a variety of dye effluents [9]. Adsorption has proven to be an efficient, feasible and effective treatment method as it utilizes low cost, non-toxic and readily available adsorbents [5].

Recently, agricultural waste by-products are reported to be inexpensive adsorbents, renewable and abundantly available [10]. Various adsorbent materials such as rice husk [11], palm fruits [12], *Aleurites Moluccana* seeds [13] and *Prosopis juliflora* seeds [14] have been used in Rhodamine B (RhB)

and Malachite green (MG) dyes removal from wastewater. The use of raw biomass as an adsorbent has however resulted to leaching of soluble organic compounds lowering its efficiency. The current study, a continuation of the work reported by [15] sought to use ethylene-1,2-diamine to chemically modify the Jackfruit seeds and utilized the functionalized adsorbent in the removal of Rhodamine B (RhB) and Malachite green (MG) dyes from an aqueous media.

3. Experimental Procedure

3.1 Chemicals and Reagents

The anal grade chemicals: Sodium Acetate (NaOAc), Hydrochloric acid (HCl), Sodium hydroxide (NaOH), Ethylene-1,2-diamine (EDA), Thionyl chloride (SOCl_2), Rhodamine B dye (RhB), N, N-dimethylformamide (DMF), Malachite green (MG) and Ammonia (NH_3) were all sourced from Sigma Aldrich, Kobian, Nairobi Kenya). The Jackfruits were collected from City park market in Nairobi, Kenya.

3.2 Adsorbent preparation

The Jackfruits were transported to Kenyatta University (KU) laboratories, washed thoroughly using distilled water, cut open and the seeds removed. The seeds were cut into small pieces and oven-dried at $105\text{ }^\circ\text{C}$ for 1 day to a constant weight. They were then ground into a fine powder. The modification of the raw powder was performed by chlorination then amination as described by Ndung'u *et al.* (2020). The powder (10 g) was first pre-activated at $50\text{ }^\circ\text{C}$ for 1 day and then suspended in DMF (200 mL) in a three-necked flask. Then, a 35 mL of SOCl_2 was slowly added while stirring the mixture under a reflux for 4 hours at $80\text{ }^\circ\text{C}$. The chlorinated material was then washed three times with 25 mL of NH_4OH solution (2M) until the pH (7). The chlorinated adsorbent was thoroughly washed with distilled water, filtered in a sintered glass crucible and *vacuo* dried. The chlorinated adsorbent (5 g) was suspended in DMF solvent, then EDA (25 mL) was slowly added and then refluxed for 4 hours at $80\text{ }^\circ\text{C}$ while stirring. Filtration then followed in a sintered glass crucible and the adsorbent *vacuo* dried at a room temperature (298 K). The raw and the EDA functionalized adsorbent were characterized using FE-SEM and TGA.

3.3 Dye stock solutions

A 1000 mg/L of RhB and MG dye stock solutions was made by dissolving 1.00 g of their respective dye salts in NaOAc buffer media to maintain the dye ionic strength. Then, fresh working solutions were prepared from the stock solutions by serial dilution. 0.1 M HCl and 0.1 M NaOH solutions were used for adjusting the pH of dye solutions. The molecular structure of RhB and MG dyes is shown in Figure 1.

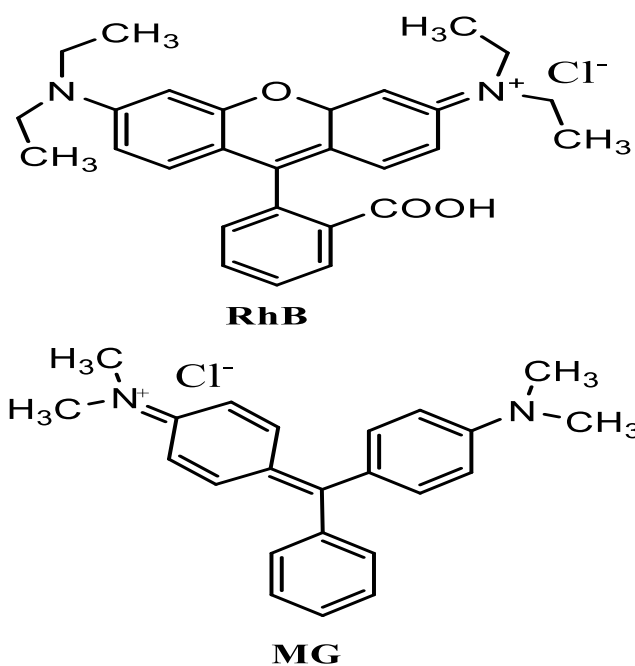


Figure 1: Molecular structure of RhB and MG dyes

3.4 Calibration of pH meter

To calibrate the pH meter, the pH electrode was first conditioned with 0.1 M KCl aqueous solution. A buffer solution of pH=4, pH=7 and pH=10 was prepared by dissolving their respective tablets in 200 mL of distilled water. The pH meter calibration was done before carrying any pH adjustments of the dye solutions.

3.5 Batch adsorption experiments

3.5.1 Point of zero charge (pH_{pzc}) studies

To determine pH_{pzc} , distilled water (100 mL) was taken in 100 mL screw cap bottles. The water pH was adjusted using 0.1 M HCl and 0.1 M NaOH from pH 1 to pH 10 as the initial pH. Then, the adsorbent (0.10 g) was added to all the screw cap bottles and agitated at 150 rpm for 24 hours. The resultant mixture was then filtrated and the pH of water at equilibrium was taken and considered as final pH. The plot of ΔpH against initial pH yielded a graph. The pH at which it is zero being considered as point of zero charge (PZC) value.

3.5.2 Batch studies

The influence of optimization parameters on uptake of RhB and MG dyes was explored by batch mode at 150 rpm using screw cap bottles (120 mL). The effect of pH (2 - 14), contact time (10 - 60 minutes), initial concentration (10 - 60 mg/L) and adsorbent dose (10 - 60 mg) was investigated in 20 mL of the test dye solution. A single parameter was varied while keeping others constant. The amount of dyes adsorbed was analyzed using a double beam UV-Visible spectrophotometer (Specord 200, Analytik Jena) set at maximum wavelengths (λ_{max}).

3.6 Data evaluation

The dye ions adsorbed at equilibrium was determined using Equation 1.

$$q_e = \frac{(C_i - C_e)V}{M} \quad (1)$$

where C_i is initial concentration (mg/L), q_e is maximum amount of dyes adsorbed (mg/g) at equilibrium, C_e is final concentration (mg/L), V is dye solution volume (mL) and M is adsorbent dosage (g).

3.7 Isotherm studies

The experimental data obtained at optimal pH, initial concentration, contact time and dosage was modelled using Langmuir and Freundlich isotherm to determine the maximum adsorption capacity of the functionalized Jackfruit seeds adsorbent. The linearized equation for Langmuir isotherm is expressed in Equation 2.

$$\frac{C_e}{q_e} = \frac{C_e}{q_m} + \frac{1}{K_L q_m} \quad (2)$$

Where q_m (mg/g) is the maximum amount of dye ions, C_e (mg/L) is the concentration of dye ions adsorbed at equilibrium and q_e (mg/g) is the adsorbed dye ions at equilibrium. K_L (L/g) is a Langmuir constant. The equation for Freundlich isotherm is given by Equation 3.

$$\ln q_e = \ln K_F + \frac{1}{n} \ln C_e \quad (3)$$

Where n and K_F correspond to removal capacity (mg/g) and adsorption intensity constant respectively.

3.8 Kinetic studies

The time data was fitted using pseudo-first-order and pseudo second-order kinetic models to determine the rate and adsorption mechanisms. The linearized kinetic rate equation for pseudo-first-order model can be written a given by Equation 4.

$$\ln (q_e - q_t) = \ln q_e - K_1 t \quad (4)$$

Where K_1 (min^{-1}) is the pseudo-first-order rate constant. q_e (mg/g) is the adsorption capacity at equilibrium and q_t (mg/g) is the adsorption capacity at time (t). Linearized rate equation for pseudo-second-order model is expressed using by Equation 5.

$$\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{1}{q_e} t \quad (5)$$

where k_2 : pseudo-second-order rate constant. The value of q_e and k_2 are calculated by plotting t/q_t against time (t).

4. Results and Discussion

4.1 Characterization

4.1.1 FT-IR

The FT-IR results as reported by [15] showed the presence of peaks at 3394.5 cm^{-1} (-OH/-NH₂), 1665.2 cm^{-1} (C=O) and

additional one at 1530.4 cm^{-1} which showed that amine (-NH₂) groups of EDA were successfully anchored onto the adsorbent surface. This improved the adsorption capacity of the functionalized adsorbent.

4.1.2 SEM

The SEM micrographs of raw and EDA functionalized Jackfruit seeds adsorbent are shown in Figure 2.

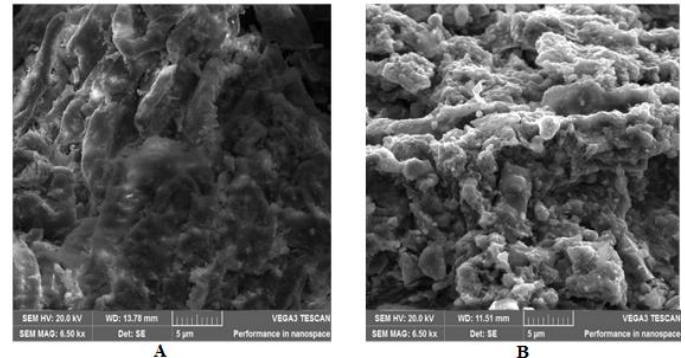


Figure 2: SEM micrographs of raw and EDA functionalized Jackfruit seeds adsorbent

The SEM micrographs (Figure 2) clearly reveal the surface texture and morphology of the raw and functionalized Jackfruit seeds adsorbents. Before chemical modification, the raw adsorbent had a more rigid, rough and less porous surface. The adsorbent however developed more pores on its surface and became rugged after chemical modification with EDA. This could be attributed to the partial destruction of cellulose, hemicellulose and lignin due to chemical treatments with thionyl chloride and ethylene-1,2-diamine. These changes in morphology of the functionalized adsorbent could improve its sorption properties.

4.2 Batch studies

4.2.1 The pH_{pzc} studies and pH effect

To study the effect of pH on RhB and MG dyes uptake using EDA functionalized Jackfruit seeds adsorbent, the studies on pH_{pzc} were first done and the results are presented by Figure 3.

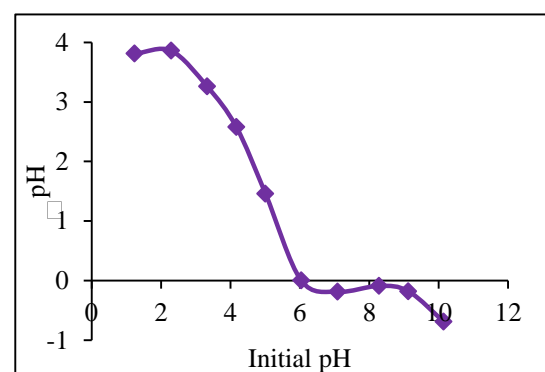


Figure 3: Point of zero charge (pH_{pzc})

It is noted that pH_{pzc} value of the adsorbent is 6.00. This implies that the net adsorbent surface is positive at $\text{pH} \leq 6.00$ and negatively charged at the adsorbent carries negative

surface charge at $\text{pH} \geq 6.00$. The results show that uptake of RhB and MG dyes is favored $\text{pH} \geq \text{pH}_{\text{pzc}}$. The results on pH effect on dyes uptake are presented in Figure 4.

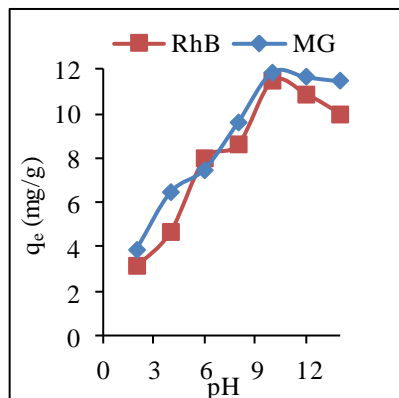


Figure 4: Effect of pH on RhB and MG dyes removal onto the functionalized adsorbent

From the results in Figure 4, the optimal pH for the adsorption of RhB and MG dyes is $\text{pH}=10$ with an uptake of 11.48 mg/g and 11.87 mg/g respectively. Lower adsorption at acidic media ($\text{pH} \leq \text{pH}_{\text{pzc}}$) was due to the presence of excess H^+ ions which competed with the cationic groups on the dyes for the adsorption sites lowering the adsorption. As a result, a higher mean uptake is attained at greater pH values $> \text{pH}_{\text{pzc}}$. Increased OH^- ions in the dye solution causes an electrostatic repulsion between the OH^- ions and the negatively charged adsorbent surface favoring the adsorption [13]. The results concur with those reported by [16].

4.2.2 Contact time

The parameter was investigated by varying the contact times (10 minutes - 60 minutes) and results are presented by Figure 5.

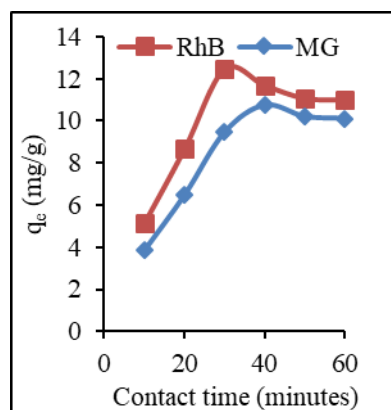


Figure 5: Effect of contact time on RhB and MG dyes removal onto the functionalized adsorbent

The results in Figure 5 showed the highest mean uptake of 12.46 mg/g and 10.74 mg/g was obtained at optimal contact times of 30 minutes and 40 minutes for RhB and MG dyes respectively beyond which a slight decrease was observed. Initially, adsorption was rapid due to availability of large number of active centers available for dyes removal [5]. As the agitation time exceed optimal, the number of active sites

becomes exhausted and consequently lowers the adsorption process [17]. These findings compare well with those reported by [5]. A contact time of 40 minutes was used for the subsequent experiments.

4.2.3 Dosage

The effect of dosage on RhB and MG dyes removal was investigated by varying the dose from 10 mg - 60 mg. The results are presented by Figure 6.

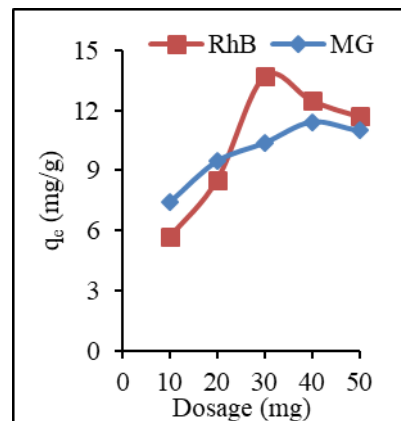


Figure 6: Effect of dosage on RhB and MG dyes removal onto the functionalized adsorbent

The results (Figure 6) showed that an addition of dose from 10 mg to 30 mg (RhB) and 40 mg (MG) increased the uptake of the dyes to 13.69 mg/g and 11.42 mg/g respectively. This due to increased binding sites for adsorbing the dyes [18]. The uptake decreases as the adsorbent doses increases beyond optimal. This is due to increased overlapping of the adsorption sites causing screening effect which reduces the number of effective sites [19]. A dosage of 35 mg was used for the subsequent experiments. The trend is coherent to the one reported by [3].

4.2.4 Initial concentration

The effect of dye concentration (10 - 60 mg/L) was investigated. The results are presented by Figure 7.

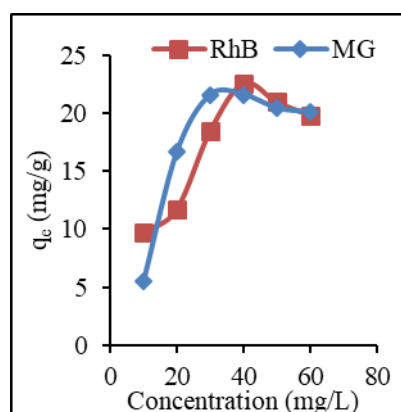


Figure 7: Effect of initial concentration on RhB and MG dyes removal onto the functionalized adsorbent

The results in Figure 7 showed that RhB and MG dyes sorption was maximum (22.61 mg/g and 21.76 mg/g) at optimal value of 30 mg/L and 40 mg/L respectively. The dyes

are adsorbed to specific adsorbent sites at the lower concentrations but the exchange sites are filled and the available active sites continue being used up on increasing the concentration of dyes [20]. In higher concentrations beyond optimal, the dye uptake decreases as the available adsorbent sites are completely used up leaving more dye ions left in the solution [21]. The results mirror those reported by [22].

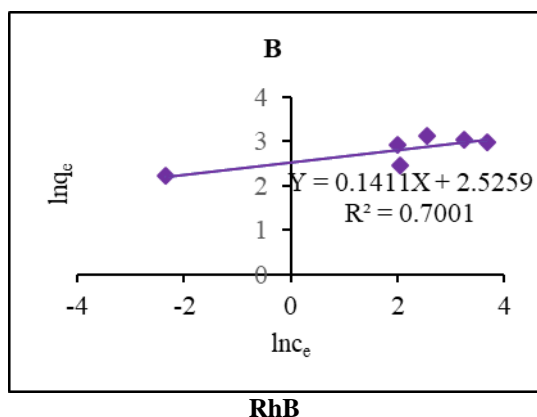
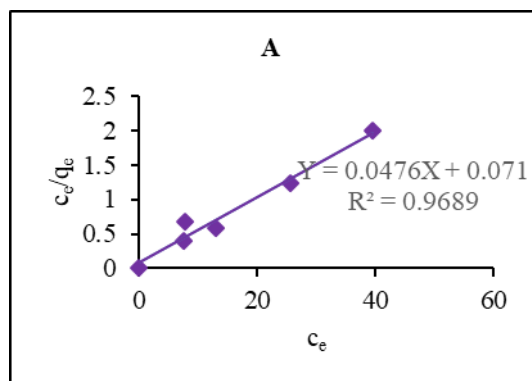
4.3 Isotherm studies

The Freundlich and Langmuir isotherms were used to analyze experimental data and results presented in Table 1.

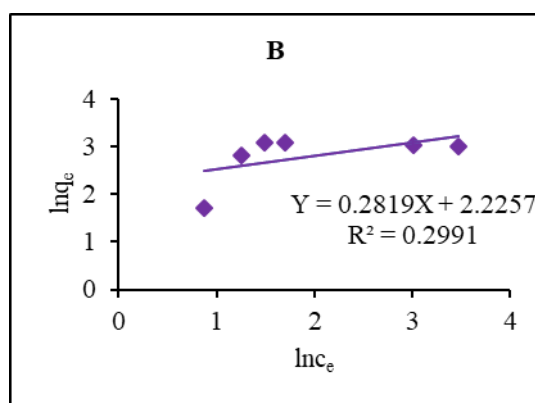
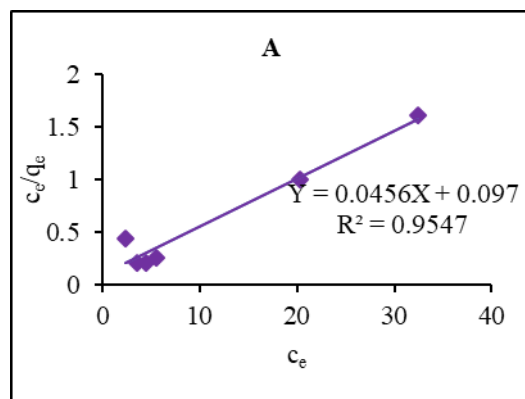
Table 1: Isotherm models for RhB and MG dyes removal onto the functionalized adsorbent

Dye	Langmuir				Freundlich		
	$Q_{max, exp}$ (mg/g)	$Q_{max, cal}$ (mg/g)	K_L (L/g)	R^2	$1/n$	K_F (mg/g)	R^2
RhB	22.61 ± 0.15	21.01 ± 0.07	6.70 x 10^{-1}	0.9689	1.41 $\times 10^{-1}$	12.50 \pm 0.18	0.7001
MG	21.76 ± 0.09	21.93 ± 0.13	4.70 x 10^{-1}	0.9547	2.82 $\times 10^{-1}$	9.26 \pm 0.05	0.2991

From the isotherm results, Langmuir plots showed a better correlation with the experimental data ($R^2 > 0.95$) than Freundlich isotherm plots as shown by the Table 1 and Figure 8. This showed that adsorption of RhB and MG dyes onto the adsorbent was well explained by Langmuir isotherm which assumes a monolayer adsorption and a chemisorption process [13]. The values of K_L were 6.70×10^{-1} (RhB) and 4.70×10^{-1} (MG) indicating the favorability of the sorption process. The findings are coherent to results reported by [4].



RhB



MG

Figure 8: Langmuir (A) and Freundlich (B) Isotherm plots for RhB and MG dyes

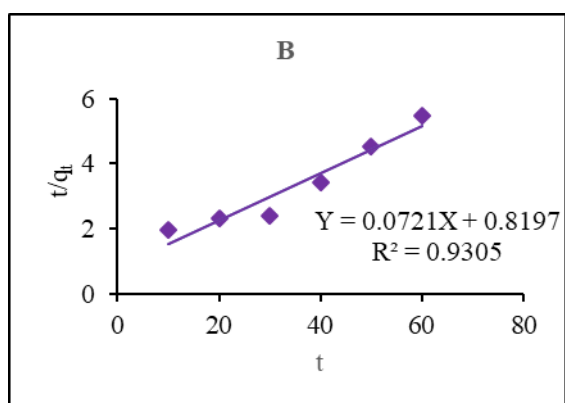
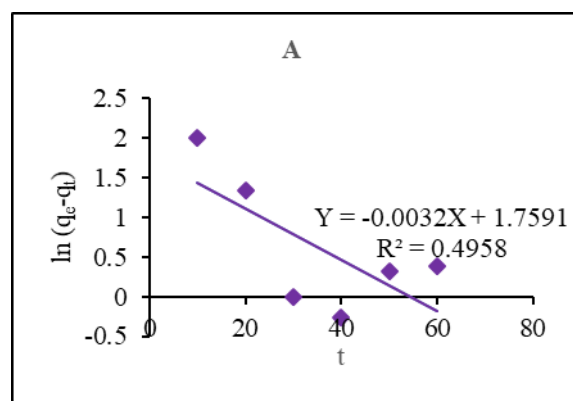
4.4 Kinetic studies

The kinetic parameters for adsorption of RhB and MG dyes ions is given by Table 2.

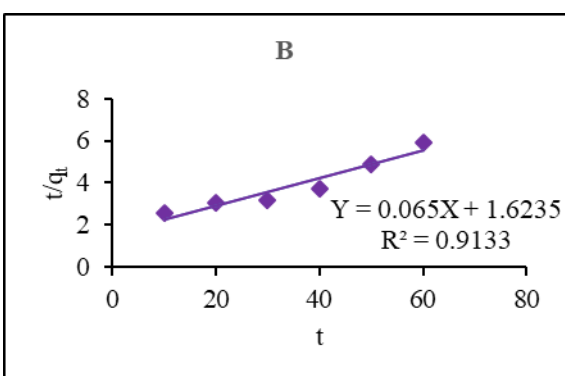
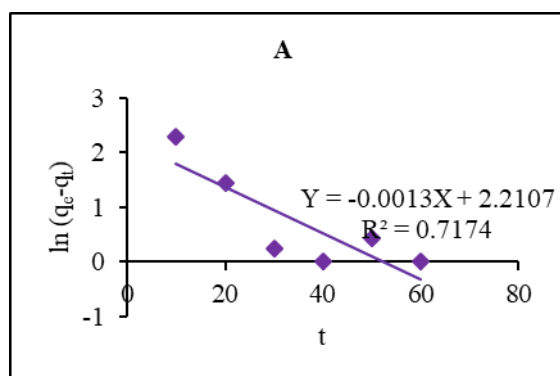
Table 2: Kinetic models for RhB and MG dyes removal onto the functionalized adsorbent

Dye	Pseudo-first-order				Pseudo-second-order		
	$Q_{max, exp}$ (mg/g)	$Q_{max, cal}$ (mg/g)	K_1 (min^{-1})	R^2	$Q_{max, cal}$ (mg/g)	K_2 ($\text{mg/g}/\text{min}$)	R^2
RhB	11.98 \pm 0.24	5.81 \pm 0.03	3.23 x 10^{-3}	0.4958	13.87 \pm 0.16	6.34 x 10^{-3}	0.9305
MG	13.74 \pm 0.39	9.12 \pm 0.17	1.30 x 10^{-3}	0.7174	15.38 \pm 0.02	2.60 x 10^{-3}	0.9133

The pseudo-second-order plots showed a better linearity of experimental data comparatively to pseudo-first-order plots (Figure 9). From the results in Table 2, the R^2 values for pseudo-second-order was 0.9305 and 0.9133 for RhB and MG dyes respectively. Also, the $Q_{max, exp}$ and $Q_{max, cal}$ values are closer compared to those in pseudo-first-order. The results revealed that pseudo-second-order best fitted the adsorption process. The model is based on the assumption that chemisorption is the rate determining step that controlled the adsorption process [23]. Similar conclusions were reported by [16].



RhB



MG

Figure 9: Pseudo-first-order (A) and Pseudo-second-order (B) kinetic plots for RhB and MG dyes

5. Conclusion and Future Scope

In the present work, raw biomass was chemically modified using thionyl chloride and ethylene-1,2-diamine. This was confirmed by FT-IR and FE-SEM results which showed a more porous adsorbent surface with the presence of functional groups of carbonyl ($-C=O$), hydroxyl (OH^-), carboxylate ($-COO^-$) (raw adsorbent) and additional anchored amine ($-NH_2$) groups (functionalized adsorbent) as important sites for Rhodamine B and Malachite green dyes removal. The batch results showed that the sorption was influenced by pH, contact time, dosage and initial concentration. The uptake of Rhodamine B and Malachite green dyes was well described by Langmuir isotherm model with a maximum adsorption capacity of 22.61 ± 0.15 mg/g and 21.76 ± 0.09 mg/g respectively. This implied a chemical interaction between Rhodamine B and Malachite green dyes and the adsorbent sites. The time data was best fitted using pseudo-second-order which describes that chemisorption was the rate determining step. The finding of the research study revealed the potential of the EDA functionalized adsorbent in dye wastewater treatment.

Data Availability

All the experimental data obtained from the research study is available in this manuscript.

Conflict of Interest

The author has disclosed no any financial or non-financial potential conflicts of interest.

Funding Source

Declared none.

Authors Contributions

The author was solely involved in all stages of the research study, manuscript preparation and submission to the Journal of Physics and Chemistry of Materials (JPCM).

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