



## ORIGINAL ARTICLE

# Modified Sawdust as an Efficient Adsorbent for the Removal of Eosin Y Dye: Optimization, Isotherm, Regeneration of Adsorbent, and Real Sample Studies

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## KEYWORDS

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**ABSTRACT:** In the current investigation, the utilization of sawdust was modified through chemical means employing diethylenetriamine. This modified form of sawdust served as an effective adsorbent for the purpose of adsorbing Eosin Y dye from aqueous samples. The chemical modification procedure was thoroughly examined, comparing the fourier transform of infrared spectroscopy (FTIR) and scanning electron microscopy (SEM) before and after the modification. Various factors including solution pH (ranging from 2 to 6), adsorbent dosage (ranging from 0.2 to 2.0 g L<sup>-1</sup>), contact time (ranging from 5 to 35 min), agitation rate, and initial dye concentration (ranging from 20 to 1000 mg L<sup>-1</sup>) were investigated. Remarkably, the modified sawdust exhibited a notable uptake capacity at ambient temperature and successfully removed approximately 96% of the Eosin Y dye with a mere 1.2 g L<sup>-1</sup> of adsorbent in just 5 minutes. Moreover, it was discovered that the adsorbent maintained its promising adsorption ability even after undergoing ten cycles of adsorption and desorption. The Freundlich isotherm model was employed to determine the adsorption parameters. This methodology was also implemented to eliminate dye from authentic samples of diverse water sources.

## INTRODUCTION

A large amount of dye is produced and consumed worldwide every year. Most of the dyes in question are derived from synthetic sources and possess toxicity, exhibiting potential carcinogenic and genotoxic properties. The introduction of dye-laden wastewater into aquatic environments detrimentally impacts photosynthetic activity, aquatic organisms, and human populations. Therefore, the effective and economic removal of toxic dye from industrial wastewater is an important task [1, 2]. Considerable work has been carried

out on the removal of dye from wastewater coming from the textile dyeing industries through coagulation [3], oxidation [4], and adsorption [5-9]. The process of adsorption is simple in operation, inexpensive, and without sludge formation [10]; therefore, it is widely used. Sawdust is an adsorbent [11] that has been used to remove several cationic dyes: methylene blue [12, 13], crystal violet [10], malachite green [14], etc. The chemical modification of sawdust could improve its ability the removal of anionic dyes according to the

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nature of interactions between dyes and modified materials. There has been much interest in the use of modified sawdust as an adsorbent substance to remove anionic ions: phosphate [15], reactive red-24 dye [16], and orange G [17].

Eosin Y (EY), an anionic dye, finds utility in the domains of textile dyeing, printing, leather treatment, printing ink, and fluorescent pigment production, among others. The discharge of untreated effluent harboring Eosin Y bears the potential to engender grave ecological predicaments by virtue of its profound chromaticity and toxic nature [18]. Some methods have been studied for the removal of EY from aqueous solutions: photooxidation [19], oxidation [20], cloud point extraction [21], solvent extraction [22], and adsorption onto i) coated sawdust by polypyrrole and polyaniline [18] ii) chitosan hydro beads [2] iii) chitosan nanoparticles [23] iv) modified bentonite [24] v) jute fiber carbon [25] vi) activated carbon [26] and vii) modified chitosan [27].

Diethylenetriamine was used to modify sawdust in this investigation. Next, EY dye was extracted from aqueous solutions using modified sawdust. To determine the optimum conditions, the effects of pH, adsorbent dosage, contact time, agitation rate, and electrolyte concentration on dye removal were investigated. Regeneration of adsorbent was studied. The results were very good. Langmuir and Freundlich's isotherms were employed. It was found that Freundlich was the best-fitted isotherm equation.

## MATERIALS AND METHODS

### *Materials and instruments*

All the chemicals utilized in the study were of analytical grade, and distilled water that had undergone double distillation was used. A stock solution of EY dye with a concentration of 1000 mg L<sup>-1</sup> was prepared by dissolving 0.50 g of the reagent (Merck) in water and diluting it to a final volume of 500 mL in a volumetric flask.

Absorption studies were conducted using a Genesis10s UV-Vis spectrophotometer (Miltonroy, American). The pH measurements were carried out using an Inolab wtw720 instrument (Germany). The morphology was visualized using SEM (Sigma VP, ZEISS, Germany).

The FT-IR spectra were recorded using an RX 700 FTIR spectrometer (PerkinElmer, United States).

### *Preparation of Adsorbent*

The sawdust provided by a local factory specializing in wood processing underwent a cleansing process involving the use of distilled water to eliminate any impurities present. Following this, the sawdust was subjected to overnight drying at a temperature of 60°C. For the subsequent step, 140 mL of concentrated grade hydrochloric acid (38%) obtained from Merck was combined with 10 grams of the sawdust. A duration of 2 hours was allowed for this step, after which 60 mL of diethylenetriamine from Merck was added to the mixture. The resulting product was then subjected to filtration, followed by washing with distilled water and subsequent drying in an oven at 40°C for a period of 24 hours [28].

### *Adsorption experiments*

In each adsorption experiment, a 50 mL solution of dye with a known concentration and pH was introduced into the modified adsorbent contained within a 250 mL Erlenmeyer flask under ambient conditions of room temperature (25±1°C). The mixture was then subjected to agitation at a rate of 80 rpm using a shaker apparatus. Following a 20-minute duration, the resultant mixture was subjected to filtration, and subsequently, the concentration of EY within the solution was determined via the spectrophotometric method, at a wavelength of 516.0 nm. The percentage of EY removal was subsequently evaluated by employing the equation provided.

$$\text{Removal}(\%) = \frac{C_0 - C_e}{C_0} \times 100 \quad (1)$$

In the given equation, C<sub>0</sub> represents the initial concentration of the dye prior to its admixture with the adsorbent, while C<sub>e</sub> denotes the concentration at equilibrium subsequent to the admixture.

## RESULTS AND DISCUSSION

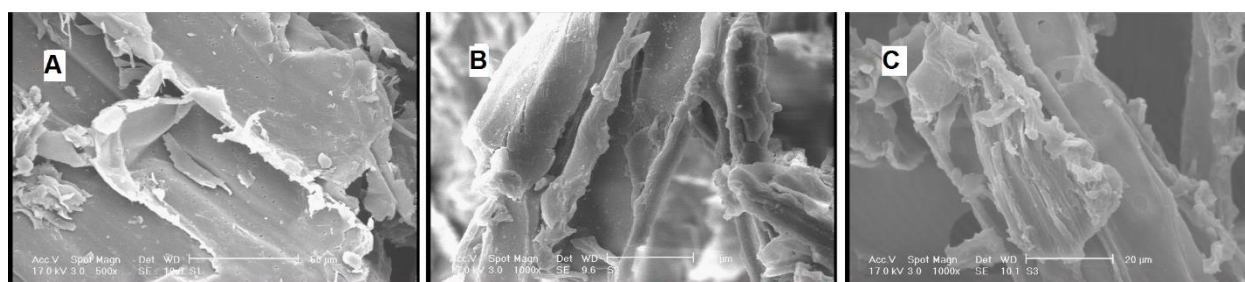
The preliminary experimental observations showed that modified sawdust removed EY dye from water samples

significantly (more than 90%) while the sawdust didn't have this ability ( $\approx 37\%$ ).

The surface morphologies of the sawdust, sawdust treated with HCl, and modified sawdust with diethylenetriamine were examined through the utilization of scanning electron microscopy (SEM). The micrographs displayed in Figure 1(a)-(c) depict the surface characteristics of each respective material. These micrographs serve as representative examples of the overall surface properties of the corresponding samples. Figure 1(a) corresponds to the original form of the sawdust, presenting a smooth surface punctuated with minuscule perforations. It is evident that, subsequent to the acid-based treatment (Figure 1(b)), the structure of the sawdust underwent disruption, resulting in the formation of microfibrils on its surface. Consequently, the surface developed pores along with coarse fibrous structures, thus facilitating a more efficient connection with the functional group. After the fabrication of the modified sawdust, the presence of the acid-treated sawdust chopsticks could no longer be observed, as they were coated with diethylenetriamine. In Figure 1(c), the

density of pores is significantly higher. However, the chemically modified sawdust exhibited a greater number of pores compared to its unmodified counterpart. Consequently, the adsorption capacity is enhanced, as adsorption is fundamentally a surface-based phenomenon.

The FTIR spectra of sawdust and modified sawdust were investigated in this study. The observed bands at 3422, 1705, and 1055  $\text{cm}^{-1}$  were attributed to the functional groups N-H, C=O, and C-N, respectively. A comparison of the FTIR spectrum of sawdust with that of modified sawdust revealed a more pronounced and narrower peak around 3422  $\text{cm}^{-1}$ , indicating the introduction of a significant amount of amino groups into the sawdust. Additionally, the FTIR spectrum of the modified sawdust exhibited the absence of the characteristic stretching vibration absorption band of C=O (observed at 1705  $\text{cm}^{-1}$ ) and an increased stretching vibration absorption band of C-N (observed at 1055  $\text{cm}^{-1}$ ). These findings provide confirmation that diethylenetriamine has been incorporated into the sawdust.

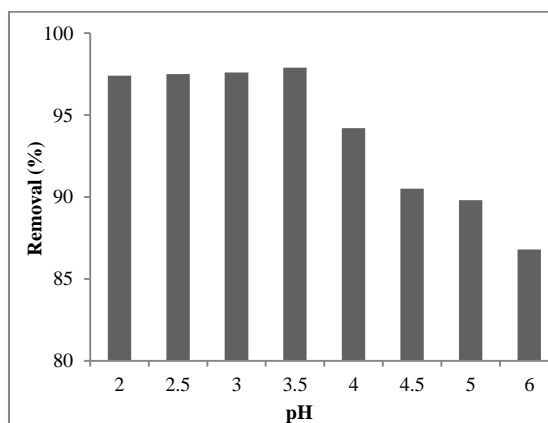


**Figure 1.** SEM images of (A) sawdust, (B) acidic sawdust, and (C) modified sawdust by diethylenetriamine.

### *The effect of pH*

The impact of the pH of the specimen solution was assessed through the manipulation of the pH in a series of 50 mL solutions that contained 100  $\text{mg mL}^{-1}$  of EY within the pH range of 2-6 using either HCl or NaOH. The highest removal of EY occurred at a pH of 3.5 (Figure 2). As EY dye precipitates under acidic conditions with pH values lower than 2, all investigations were conducted at a pH level above 2.0. In acidic conditions, the adsorbent's surface became positively charged due to the elevated concentration of  $\text{H}^+$  ions,

thereby enhancing the electrostatic attraction between the adsorbent and EY. At pH levels above 3.5, the concentration of  $\text{H}^+$  ions decreases, resulting in insufficient protonization of the adsorbent and a weakened interaction between the adsorbent and the dye. In alkaline conditions, the adsorption of EY was diminished, likely due to the presence of  $\text{OH}^-$  ions on the surface of the adsorbents, which competed with EY for adsorption sites [18].

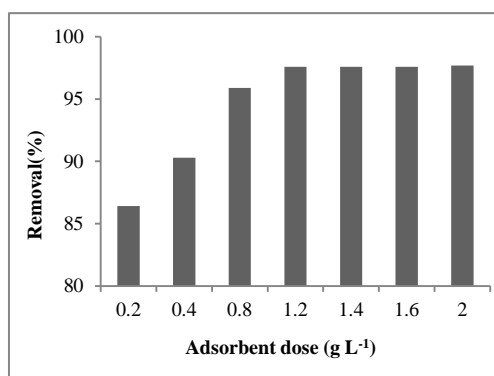


**Figure 2.** Effect of pH on EY dye removal (adsorbent dosage=0.0014 g mL<sup>-1</sup>, contact time=30 min, agitation rate= 135 rpm)

### *The effect of the adsorbent dose*

The impact of the quantity of the adsorbent on the elimination of EY dye is demonstrated in Figure 3. The percentage of elimination experienced an upward trend as the adsorbent dose increased, reaching a peak value before stabilizing. The augmentation in the adsorption of dyes as a result of the adsorbent dose was attributed to the heightened accessibility of the adsorbent's surface area for adsorption. The findings indicated that a quantity

of 1.2 g L<sup>-1</sup> of the modified sawdust was required to achieve a 98% removal of EY from the initial concentrations of 100 mg L<sup>-1</sup> while to remove 50 mg L<sup>-1</sup> eosin, the coated sawdust with polymer was necessary 16 g L<sup>-1</sup> [18], using jute fiber carbon is 4 g L<sup>-1</sup> to remove 100 mg L<sup>-1</sup> EY [25], using modified chitosan is 0.5 g L<sup>-1</sup> for removal 100 mg L<sup>-1</sup> EY [27] and using activated carbon is 1.0 g L<sup>-1</sup> to remove 100 mg L<sup>-1</sup> EY [26].



**Figure 3.** Effect of adsorbent dose on EY dye removal (initial dye concentration 100 mg L<sup>-1</sup>, pH=3.5, contact time=30 min)

### *The effect of contact time and Agitation Rate*

The investigation examined the impact of contact time within the range of 5-35 minutes. The findings revealed that, even within a brief span of 5 minutes, over 96% of the dye was successfully eliminated (Figure 4). However, to achieve a removal rate of approximately 98%, a time interval of 15 minutes was required. These outcomes surpass the outcomes of the most extensive adsorption

methods employed for the eradication of EY dye [18, 2-25, 27], with the exception of the employment of carbon active as an adsorption technique [26].

The agitation speed was maintained between 40 and 145 rpm, with the highest removal rate occurring above 60 rpm. Consequently, a stirring speed of 80 rpm was selected for subsequent experiments (Figure 5).

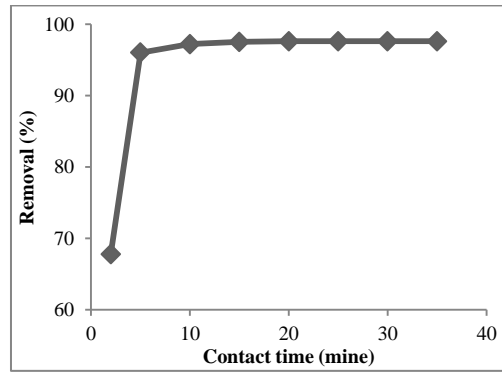


Figure 4. Effect of contact time on EY dye removal (adsorbent dosage=0.0014 g mL<sup>-1</sup>, pH=3.5, agitation rate= 135 rpm).

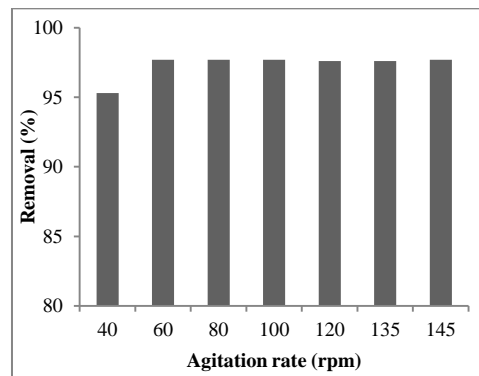


Figure 5. Effect of agitation rate on EY dye removal (initial dye concentration 100 mg L<sup>-1</sup>, pH=3.5, adsorbent dosage=0.0014 g mL<sup>-1</sup>)

### The effect of Electrolyte

The utilization of KCl and NaNO<sub>3</sub> as electrolytes was observed in this study. The findings indicated that an augmentation in the concentration of electrolyte up to 0.2 mol L<sup>-1</sup> does not yield a significant impact on the elimination of EY (the removal of dye decreases by a maximum of 2 percent and 6 percent for KCl and NaNO<sub>3</sub> respectively). Conversely, in numerous adsorption techniques, the inclusion of electrolyte leads to a substantial reduction in removal [29, 30].

### Initial concentration

The impact of the initial concentration of the dye on the eradication of the dye was examined utilizing the aforementioned ideal concentrations. The outcomes, as demonstrated in Table 1, indicate that a dye concentration of 20 mg L<sup>-1</sup> can be eliminated by 98.1%. This range (20-1000 mg L<sup>-1</sup>) is comparatively extensive, and the eradication percentage within this range was noteworthy.

Table 1. Removal for different initial concentrations of EY dye.

| Initial concentration (mg L <sup>-1</sup> ) | Removal (%) |
|---|-------------|
| 20  | 98.1        |
| 50  | 98.1        |
| 100   | 97.7        |
| 150   | 97.7        |
| 200   | 92.7        |
| 300   | 89.8        |
| 400   | 86.1        |
| 500   | 82.7        |
| 700   | 79.5        |
| 1000  | 50.0        |

### Regeneration of adsorbent

Regeneration investigations pertaining to the adsorbent hold significant value in industrial sectors due to their role in the exploration of adsorbent recycling potential and the recovery of sorbed materials. This particular research on regeneration studies employed a regeneration column in the form of a glass tube, measuring 70 mm in length and 6 mm in internal diameter. To initiate the study, the column was packed with 0.1 g of adsorbent, which was gently compressed using a flat glass rod. Subsequently, solutions of 50 mL each, containing 100 mg L<sup>-1</sup> of EY and with a pH value of 3.5, were introduced into the column. The removal was rather 98%. The column was eluted with 10 mL of 1 mol L<sup>-1</sup> HCl or 10 mL of 1 mol L<sup>-1</sup> NaOH. Then the column was washed with 50 mL of water. This cycle was repeated 10 times, in all cycles for both eluents (HCl or NaOH), the removal was about 98%. These results were better than desorption results using activated carbon [26].

### Isotherms of Adsorption

In the present investigation, the utilization of Langmuir and Freundlich's isotherms was employed to examine the adsorption of EY dye onto sawdust that had undergone modifications. These particular isotherms were attained under the previously optimized conditions, encompassing an initial concentration range of 100-1000 mg L<sup>-1</sup>, as well as a temperature of 25±2 °C. The determination of  $q_e$  was accomplished by means of equation (2).

$$q_e = (C_e - C_0) \frac{V}{W} \quad (2)$$

The adsorption capacity in an equilibrium (mg L<sup>-1</sup>) is represented by  $q_e$  in the given equation. The initial concentration of dye (mg L<sup>-1</sup>) is denoted by  $C_0$ , while the equilibrium concentration (mg L<sup>-1</sup>) is represented by  $C_e$ . The volume of the solution is denoted by  $V$ , and the weight of the adsorbent is represented by  $W$ .

In order to analyze the adsorption of dye in accordance with the Langmuir Isotherm, equation (3) was utilized.

This equation incorporates the constants of Langmuir, namely  $Q_0$  and  $b$ . The plot of  $C_e/q_e$  against  $C_e$  was employed to examine the sorption process. However, it was determined that the sorption process being investigated does not adhere to the Langmuir isotherm model. Consequently, no linear relationship was observed between the equilibrium concentrations of the sorbet in the solid and liquid phases, respectively.

$$\frac{C_e}{q_e} = \frac{1}{Q_0 b} + \frac{1}{Q_0} C_e \quad (3)$$

The Freundlich model posits a heterogeneous adsorption surface characterized by sites that possess varying adsorption energies that are not uniformly accessible. The Freundlich isotherm, which is more widely employed, is attained by means of equation (4), wherein  $K_f$  denotes the Freundlich constant and is contingent upon the adsorption capacity of the adsorbent. In the context of this isotherm, the logarithm of  $q_e$  was graphed against the logarithm of  $C_e$ .

$$\ln q_e = \ln K_f + \frac{1}{n} (\ln C_e) \quad (4)$$

The Freundlich equation was determined to be the most suitable isotherm equation, with an  $R^2$  value of 0.946. Several independent studies were conducted, and the values of  $K_f$  obtained for the adsorption of EY onto various adsorbents have been compiled in Table 2. It can be observed that the  $K_f$  for modified sawdust with diethylenetriamine is significant compared to other adsorbents.

Table 2 summarizes the constant parameters of the isotherm equations used in this adsorption process, as well as the correlation coefficient ( $R^2$ ) for conventional isotherms based on these equations and requirements. The suitability of the Freundlich model for interpreting the experimental data suggests that the adsorption of the proposed dye is limited to a monolayer coating and that the surface is relatively homogeneous with significant interaction with SY and Tar molecules.

**Table 2.** Calculated parameter values based on Freundlich isotherm model deferent adsorbents.

| Adsorbent                                | $K_f$ | $n$  | Ref.      |
|--|-------|------|-----------|
| Jute fiber carbon                        | 4.58  | 2.37 | 25        |
| Activated carbon                         | 75.51 | 4.14 | 26        |
| Modified sawdust with Polypyrrole        | 5.1   | 2.04 | 18        |
| Modified sawdust with Polyaniline        | 4.68  | 2.25 | 18        |
| Modified sawdust with diethylenetriamine | 35.29 | 1.28 | this work |

### Application

To evaluate the dependability of the proposed technique for eliminating EY, various concentrations of EY from tap water, river water, and industrial wastewater samples were utilized for removal assessment. In order to conduct this evaluation, a volume of 25 mL from each authentic sample was examined, and it was determined that there

was no presence of EY in any of the samples. Consequently, diverse quantities of EY were introduced to the samples, and the removal process was observed. The elimination of dye in all the samples exceeded 95%, which signifies the efficacy of this methodology across the board (Table 3).

**Table 3.** Removal of EY from water samples

| Sample                | Added EY (mg L <sup>-1</sup> ) | Removal (%) |
|-----------------------|--------------------------------|-------------|
| Tap water             | 50.0                           | 96.2        |
|                       | 100.0                          | 96.1        |
|                       | 150.0                          | 95.0        |
| River water           | 50.0                           | 99.8        |
|                       | 100.0                          | 97.2        |
|                       | 150.0                          | 95.0        |
| Industrial wastewater | 50.0                           | 97.4        |
|                       | 100.0                          | 96.7        |
|                       | 150.0                          | 96.2        |

### CONCLUSIONS

The current investigation assessed the efficacy of utilizing chemically modified sawdust, treated with diethylenetriamine, as an efficient adsorbent for the elimination of EY dye, an anionic dye, from various water samples. The SEM images clearly portrayed the emergence of numerous pores following chemical treatment, while the FTIR spectra indicated the occurrence of chemical modification. The experimental requisites (such as adsorbent preparation, adsorbent dosage, and contact time) outperformed several previously documented approaches. The experimental findings demonstrated that, during a single adsorption

process, quantitative removal (exceeding 82%) was achieved for dye concentrations ranging from 20 to 500 ppm. The adsorption equilibrium data conformed to the concentration ranges studied, as per the Freundlich model. Furthermore, the regeneration of the adsorbent was thoroughly examined, revealing that the adsorbent maintained its adsorption capacity even after ten cycles of reuse.

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### Conflict of interest

The authors declare that they have no conflicts of interest.

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