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Research Article

Adsorption Studies of Textile Dye (Chrysoidine) from Aqueous Solutions Using Activated Sawdust

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Chrysoidine is a type of industrial azo dye and a well-known toxicant. Due to its good dyeing characteristics, it is widely used for dyeing leather, paper, feather, grass, wood, bamboo, etc. Hence, it is very important to remove or reduce its concentration below the contamination level in the waste line by using low-cost technologies. Sawdust is a plentiful material available very cheaply from sawmills and woodworks. Therefore, the present work was conducted to study sorption ability of both raw sawdust and chemically activated sawdust carbon on the removal of chrysoidine from the aqueous solutions. Adsorption isotherms of the dye on sawdust were determined and correlated with usual isotherm equations like Freundlich and Langmuir. Experimental results have shown that sawdust has a high adsorption efficiency, and the adsorption of chrysoidine followed Freundlich's isotherm. Although raw sawdust proved to be slightly less efficient in comparison to chemically treated sawdust but in economic terms, raw sawdust is more cost-effective as the difference in the percent dye removal is less than the difference in the manufacturing costs. The influence of several parameters such as effect of temperature, adsorbent dose, adsorption time, etc., on the adsorption process was studied along with thermodynamic parameters such as enthalpy (ΔH°) and entropy (ΔS°).

1. Introduction

Wastewaters from textile industries are colored, which is mainly due to dyes used in textile industry. Chemical species present in textile effluents are of diverse nature, hence, posing a challenge to conventional physicochemical and biological treatment methods. Dyes are almost invariably toxic, and additionally a visible pollutant, so their removal from the effluent stream is ecologically essential. Recent estimates indicate that approximately 12% of synthetic dyes used each year are lost during manufacture and processing operations and that 20–35% of these input dyes enter the environment through effluents from the treatment of residual industrial water. Dyes are easily visible even in extremely diluted forms, posing aesthetic problems, and are toxic to human and aquatic life [1–5].

Numerous procedures, such as chemical coagulation using alum, lime, ferric chloride, and ferric sulphate, biosorption, oxidation methods using chloride and ozone, membrane separation, biological treatment, magnetic particles and adsorption, have been employed to remove dyes from industrial effluents [1, 3–7]. Adsorption requires less land area, least effect to toxic chemicals, greater flexibility in the design, and operation and superior removal of organic contaminants. Therefore, significant attention has been directed to adsorption as a process for color removal from wastewaters since it offers the most economical and effective treatment methods. Recently, various research groups have used different low-cost waste natural materials as an adsorbent for the separation of basic dyes. Nurchi et al. [2] investigated the sorption of chrysoidine on row cork and cork entrapped in calcium alginate, and the amounts adsorbed were about 0.27 and 0.29 mmol/g of chrysoidine in aqueous solutions at a pH of 7. Jain et al. [8] removed hazardous dye naphthol yellow S from wastewater using activated carbon and activated deoiled mustard. Mittal et al.

[9] investigated the adsorption of chrysoidine Y on bottom ash and deoiled soya. The dye sorption capacities of bottom ash (BET surface area = $870.5\,\mathrm{cm}^2\cdot\mathrm{g}^{-1}$) and deoiled soya (BET surface area = $728.6\,\mathrm{cm}^2\cdot\mathrm{g}^{-1}$) were determined as $3.61\times10^{-5}\,\mathrm{mol}\cdot\mathrm{g}^{-1}$ and $1.92\times10^{-5}\,\mathrm{mol}\cdot\mathrm{g}^{-1}$ at 30°C, respectively. Younes et al [10] and Pal et al. [11] have reviewed and studied biomass-derived activated carbons for adsorptive heat pump applications. Gupta et al. [12] studied the removal of indigo carmine dye from industrial effluents by deoiled mustard and charcoal. The adsorption experiments were carried out at 30°C, pH 3.0 for charcoal and pH 8.0 for deoiled mustard, and adsorbate concentration $2\times10^{-4}\,\mathrm{mol}\cdot\mathrm{L}^{-1}$. Amounts of adsorbates were found as approximately $0.33\times10^{-4}\,\mathrm{mol}\cdot\mathrm{L}^{-1}$ for $0.40\,\mathrm{g}\cdot\mathrm{L}^{-1}$ for charcoal and $0.25\times10^{-4}\,\mathrm{mol}\cdot\mathrm{L}^{-1}$ for $7.5\,\mathrm{g}\cdot\mathrm{L}^{-1}$ of deoiled mustard. Larous and Meniai [13] studied the use of carbonized sawdust as an adsorbent for phenol. Raghuvanshi et al. [14] have used chemically treated sawdust as a bioadsorbent for the removal of methylene blue dye.

Chrysoidine is a type of industrial azoic dye. Due to its good dyeing fastness, it is widely used for dyeing leather, paper, feather, grass, wood, bamboo, etc. Chrysoidine can cause acute and chronic toxicity to mammals when taken orally or inhaled, and its median lethal concentration (LC50, 24 h) for fish was 0.5 mg/L. According to American Dye Manufacturing Institute (ADMI), the basic dyes are generally more toxic than acidic or direct dyes [15].

The objective of this paper was to explore the removal of chrysoidine basic dye in an aqueous solution by adsorption on the sawdust. Chrysoidine removal has never been tried before by using chemically treated and raw sawdust. The influence of several parameters on adsorption such as contact time, dye concentration, temperature, etc., on the adsorption process was also studied.

2. Materials and Methods

Sawdust was grabbed from a nearby wood working factory and divided into two portions by weight. One part was chemically treated, while the second was kept for use in its raw form. The *first part* was treated with sulphuric acid in the ratio of 4 : 3 parts by weight. The material was kept in a vacuum oven for 24 hours at 150°C. The carbonized material was washed thrice with distilled water to remove any unreacted acid and dried at 100°C for 24 hours. The material was finely grounded and passed through sieve ISS 600 and stored. This material was used in adsorption experiments as chemically activated sawdust. The second part of the sawdust was saturated with the distilled water and washed repeatedly to remove the typical yellow color of the sawdust caused by lignin. Subsequently, it was treated with formaldehyde for 6 hours to remove the lignin. The material was dried in a vacuum oven at 60°C for 24 hours, and this dried material was grounded to fine powder and sieved through ISS 600. This material was used throughout as untreated sawdust adsorbent [14].

The dye chrysoidine is a basic dye, with CAS number 532-82-1, molecular weight 248.71, color index number 11270, $\lambda_{\max}$ 449 nm, and empirical formula (Hill Notation) $C_{12}H_{12}N_4\cdot HCl$. The structural formula is shown in Figure 1.

All reagents used in the present study were of analytical reagent (AR) grade and supplied by Sigma-Aldrich.

2.1. Adsorption Studies. In order to explore the adsorption efficiency of both versions of sawdust, a series of experiments was conducted. During the experiments, 1.0×10^{-4} kg to 1.0×10^{-3} kg of both adsorbents were taken separately in 250 mL conical flasks sealed with parafilm, on an electric rotary shaking machine, in 100 mL aqueous solution of the dye (variable concentrations) at different temperatures and time intervals. After predetermined time intervals, the solution was analyzed for the residual concentration of dye by using a Shimadzu UV-VIS spectrophotometer at 449 nm. A similar procedure was followed for another set of flasks containing same dye concentration but without sawdust to be used as blank. All the experiments were conducted in triplicate at neutral pH. The adsorption of the dye can be described in terms of dye removal:

$$\text{dye removal}\,(\%) = \frac{c_0 - c_f}{c_0}\times 100, \tag{1}$$

where c_0 is the initial and c_f is the final concentrations of the dye in the solution.

2.2. Adsorption Thermodynamics. The spontaneity of the adsorption process is normally described by changes in the standard enthalpy (ΔH°), Gibb's free energy (ΔG°), and entropy (ΔS°). Decrease in ΔG° normally indicates a spontaneous process and the opposite is true for a non-spontaneous reaction. The relations for the thermodynamic parameters are given as follows:

$$\Delta G^\circ = -RT\ln K_a, \tag{2}$$

where K_a is the thermodynamic equilibrium constant.

$$K_d = \frac{c_a}{c_e} \approx K_a, \tag{3}$$

where K_d is the adsorption equilibrium constant and c_a is the adsorbed concentration

$$\Delta G^\circ = \Delta H^\circ - T\Delta S^\circ, \tag{4}$$

$$\ln K_a = -\frac{\Delta H^\circ}{RT} + \frac{\Delta S^\circ}{R}. \tag{5}$$

A linear relation between $\ln K_a$ and inverse temperature is used to evaluate ΔH° and ΔS°.

2.3. Adsorption Isotherms. Adsorption analysis is normally performed using adsorption isotherms. Freundlich isotherm is the earliest known relationship describing the adsorption equation and is often expressed as

$$q_e = K_F c_e^{1/n}, \tag{6}$$

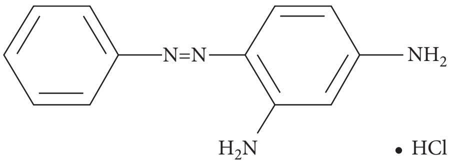


FIGURE 1: The structural formula of chrysoidine.

where q_e is the adsorption density (mg of adsorbate per gm of adsorbent), c_e is the equilibrium concentration (mg/L), K_F is the Freundlich constant, and n is an exponent [16].

The equation can be converted in a linear form by taking the log on both sides as

$$\log q_e = \log K_F + \frac{1}{n}\log c_e. \tag{7}$$

A plot of log q_e against log c_e yields a straight line, indicating the conformation of Freundlich's isotherm for adsorption. The constants can be determined from the corresponding slope and intercept.

Langmuir isotherm is another most frequently used adsorption isotherm [17]. It is described by the following relation:

$$q_e = q_{\max} \cdot \frac{bc_e}{1 + bc_e}, \tag{8}$$

where $q_{\max}$ is the maximum adsorption capacity and b is the Langmuir constant (L/mg).

This equation can also be converted into a straight line as

$$\frac{c_e}{q_e} = \frac{1}{q_{\max}b} + \frac{1}{q_{\max}}c_e. \tag{9}$$

A plot of c_e/q_e vs c_e gives a straight line. The slope and intercept gives the values of Langmuir constants.

Equations (2) and (4) can be straightforward but may lead to errors as mentioned elsewhere [13]. ISOT-Calc and other statistical and mathematical packages [18, 19] have been used to fit the nonlinear isotherm data. In this study, iterative Levenberg–Marquardt and nonlinear least squares was used to find the parameters. Nonlinear optimization was based on minimizing the objective function defined as

$$\mathrm{OF} = \sum w_i\left(q_{e_{\mathrm{exp}}} - q_{e_{\mathrm{calc}}}\right)^2, \tag{10}$$

where $q_{e_{\mathrm{exp}}}$ is the experimental adsorption density, $q_{e_{\mathrm{calc}}}$ is the modeled adsorption density, and w_i is the data weighing coefficient for every point (c_e, q_e).

The compatibility of an adsorbent-adsorbate pair can be indicated by a nondimensional parameter R_L. R_L is called the separation parameter, and it is derived from Langmuir constant

$$R_L = \frac{1}{1 + bc_0}, \tag{11}$$

where $R_L > 1$ indicates nonfavorable combination, $0 < R_L < 1$ favors adsorption, and $R_L = 1$ is for nonlinear adsorption, whereas $R_L = 0$ shows irreversible adsorption.

3. Results and Discussion

Figures 2(a) and 2(b) describe the effect of initial dye concentration on the rate of adsorption on sawdust both treated and untreated. It can be deduced that for any particular experiment, the rate of adsorption decreased with time until it gradually approached a plateau owing to the continuous decrease in the driving force (concentration) and also indicating that the adsorbent is saturated at this point. The saturation point was reached within 40 to 50 minutes. In the beginning, the adsorption process was found to be very fast, and a large amount of the total concentration of dye was removed in the first half an hour. Activated sawdust reached equilibrium slightly earlier than the raw sawdust. Moreover, the initial adsorption rate was high for initial dye concentration as resistance to dye uptake decreases when the mass transfer driving force increases. This observation indicated that the removal of dye is dependent upon the initial concentration of the solution.

The effect of various concentrations of treated and nontreated sawdust on adsorption is shown in Figures 3(a) and 3(b). Graphs show a decline in the dye concentration at a rapid pace as the sawdust quantity is increased. Chemically treated sawdust gave the greater removal at all levels of the adsorbent dose. In the beginning, the rate of dye removal was faster, which slowed down as the dose increased. This can be attributed to the fact that, at a lower adsorbent dose, the dye molecules are easily reachable, and therefore, removal per unit mass of adsorbent is higher. A larger surface area of the adsorbent particles and smaller size of adsorbate molecules favor adsorption.

The rate of adsorption is higher at the initial stage as sites are vacant for adsorption. Adsorption and desorption occur simultaneously, and an adsorption equilibrium is reached when isotherms are applied. With a rise in adsorbent quantity, there is a less corresponding increase in adsorption resulting from lower adsorptive capacity utilization of adsorbent. The results obtained from above experiment indicate that chemically treated sawdust has a large potential as an adsorbent for dye removal as compared to raw sawdust.

It has been reported that if the solubility of the adsorbate increases with an increase in temperature, then the chemical potential decreases and both these effects, working in the same direction, cause a decrease in adsorption. Conversely, if the temperature has the reverse effect on the solubility, then both the said effects will act in the opposite direction, and adsorption may increase or decrease depending on the predominant factor [14]. The adsorption rates of chrysoidine at three different temperatures (30°C, 40°C, and 50°C) were studied as shown in Figure 4(a) and 4(b). In case of raw sawdust, the rate of dye adsorption decreased with an increase in temperature from 30°C to 50°C with a 4 g/L dose in 5 minutes time from a 100 g/L dye solution. This behavior indicated that the process is exothermic in nature. This can be attributed to the predisposition of the dye molecules to escape from the solid phase to bulk phase with a rise in temperature of the solution. However, in the case of treated sawdust, the rate of dye uptake increases rapidly.

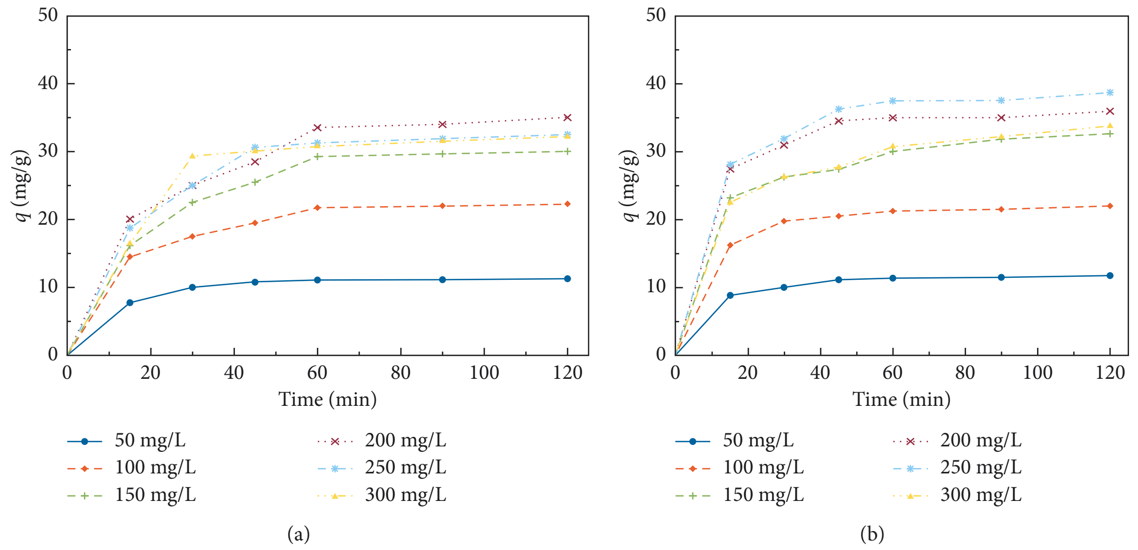


Figure 2: Effect of contact time on dye (dose 4 g/L) adsorption on (a) raw sawdust and (b) chemically treated sawdust.

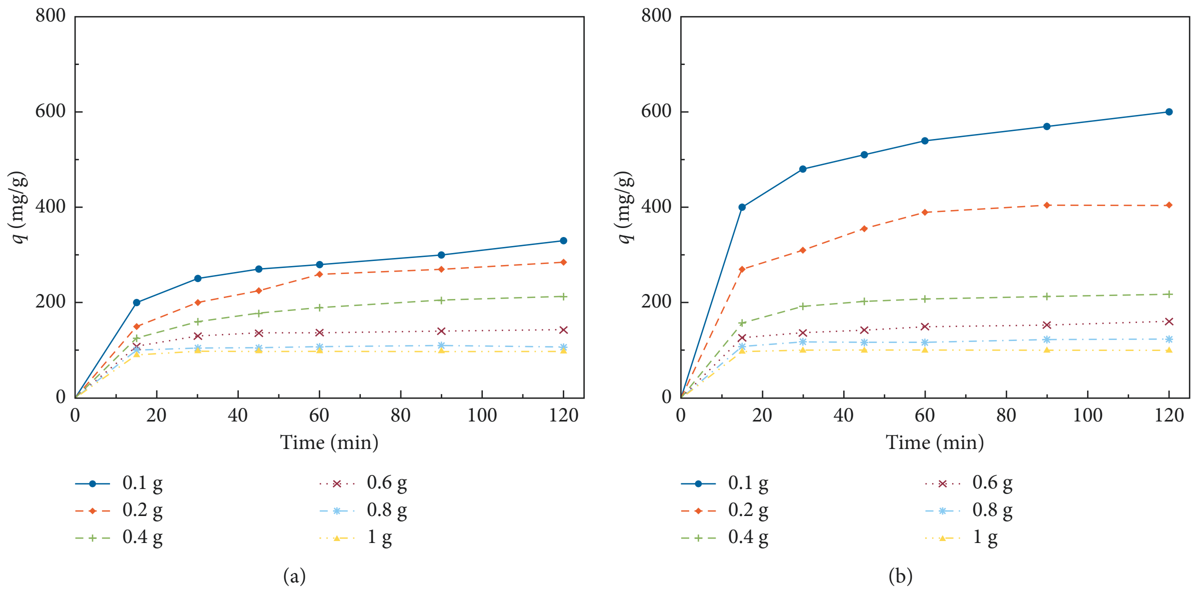


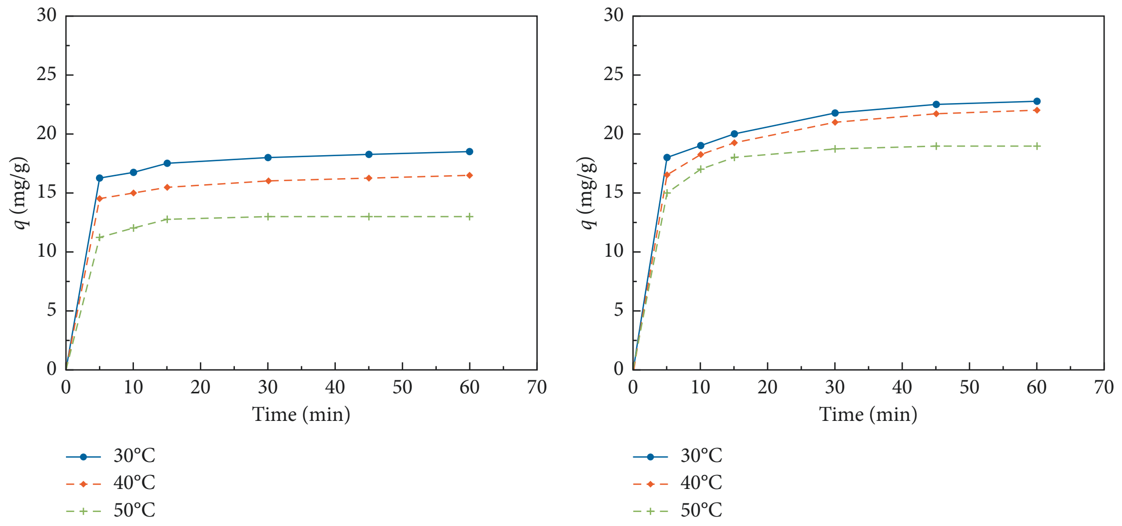
Figure 3: Effect of (a) raw sawdust dose (100 mg/L) and (b) chemically treated sawdust dose on dye adsorption.

The thermodynamic parameters (ΔH° and ΔS°) were obtained from the slope and intercept of the linear regression line fitted on the $\ln K$ vs $1/T$ data. The plot for raw and activated sawdust is shown in Figure 5, and the thermodynamic parameters values are given in Table 1.

From Table 1, it can be seen that ΔG° is negative, indicating that the adsorption is spontaneous. With the increase in temperature, ΔG° become less negative or the spontaneity decreases by increasing temperature. Compared to raw sawdust, activated sawdust showed more negative value for ΔG°, indicating more feasibility of adsorption. ΔH° is also negative showing the process is exothermic. And the process is physisorption as chemisorption proceeds with the enthalpy changes in the range of −80 to −200 kJ/mol. The negative value of ΔS° reveals the adsorption is ordered. Similarly, more negative value of ΔS° indicates decreasing randomness on activated surface than raw sawdust. Overall, it can be concluded from the obtained thermodynamic parameters that the activated sawdust betters in terms of feasibility, exothermicity, and ordered layering than raw sawdust.

For adsorption of chrysoidine on sawdust, there may be a possibility of intraparticle diffusion. In order to investigate this possibility, experiments were conducted and are depicted in Figures 6(a) and 6(b).

The plots (Figures 6(a) and 6(b)), with log (dye removal) (%) versus log time (min), for adsorption at three



(a) (b)

Figure 4: Effect of temperature on dye removal with (a) raw sawdust (conc. 100 mg/L; dose 4 g/L) and (b) chemically treated sawdust (conc. 100 mg/L; dose 4 g/L).

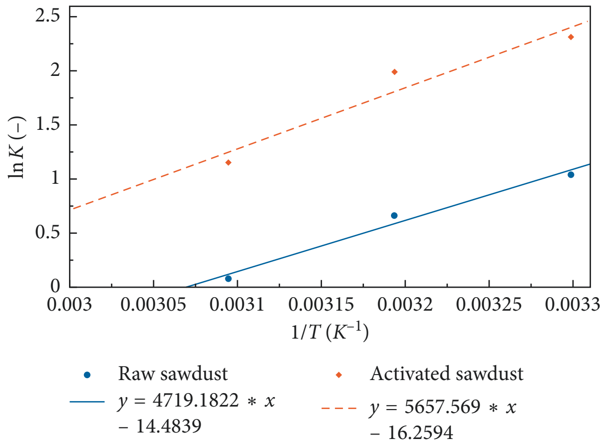


Figure 5: Ln K vs $1/T$ curves for raw and activated saw dust.

Table 1: Thermodynamic parameters at different temperatures.

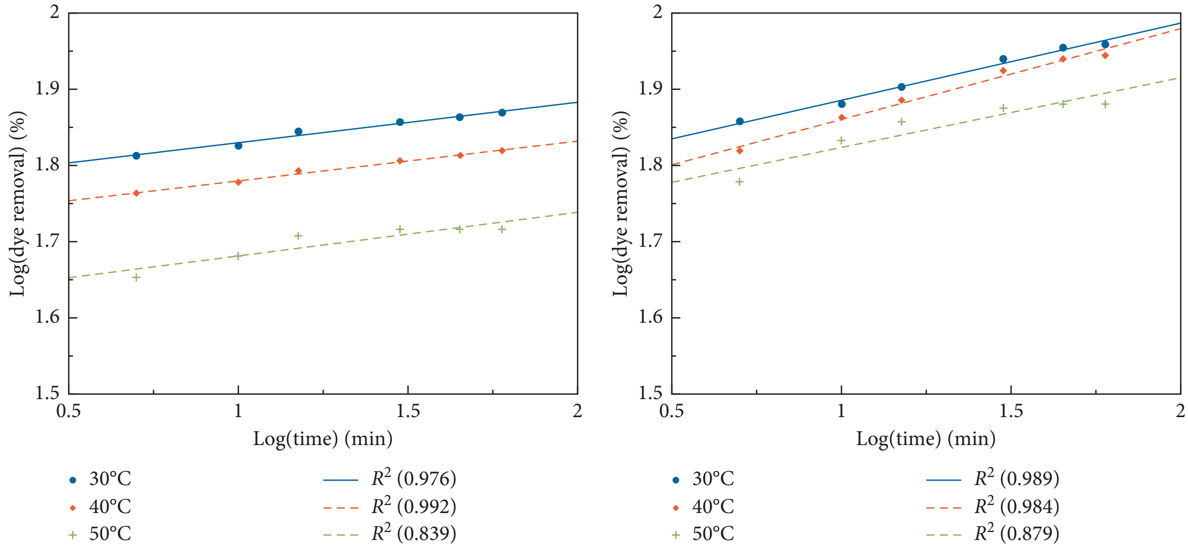
Temperature (°C)	Raw sawdust				Chemically treated sawdust			
	K_a (–)	ΔG°(kJ/mol)	ΔH° (kJ/mol)	ΔS° (J/mol-K)	K_a (–)	ΔG° (kJ/mol)	ΔH° (kJ/mol)	ΔS° (J/mol-K)
30	2.85	−2.64	−39.22	−120.40	10.11	−5.83	−47.03	−135.15
40	1.94	−1.73			7.33	−5.19		
50	1.08	−0.22			3.17	−3.10		

different temperatures ranging between 30 and 50°C, resulted in straight lines, which specify the existence of intraparticle diffusion. These plots are used to describe whether adsorption is controlled by diffusion in the adsorbent particles or the consecutive diffusion in the bulk of the solution [20–22].

3.1. Adsorption Isotherms. The two most frequently used adsorption isotherms are employed in Figure 7 for the adsorption of chrysoidine on raw and activated sawdust. It can be seen that the coefficient of determination (R^2) is lower in Freundlich isotherm. Therefore, it can be presumed that the adsorption of chrysoidine on raw and activated sawdust follows Langmuir adsorption model.

The parameters of the Freundlich and Langmuir linear isotherms are given in Table 2, and the same were used to compare the experimental curves. Lower values of objective function (OF) were obtained in case of Langmuir isotherm.

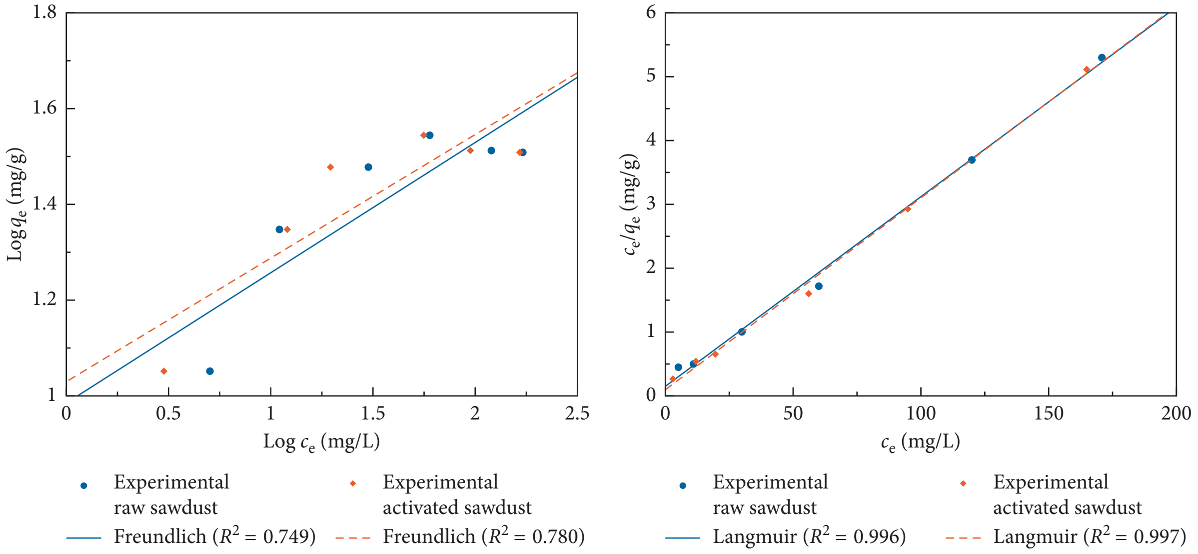
Similarly, Figure 8 shows the nonlinear curve fittings of Freundlich and Langmuir isotherms. Lower values of OF render Langmuir better model for the



(a)

(b)

Figure 6: Log (dye removal) (%) versus log time (min) for (a) raw sawdust and (b) chemically treated sawdust.



(a)

(b)

Figure 7: Freundlich (a) and Langmuir (b) linear isotherms of raw and chemically treated sawdust for chrysoidine removal.

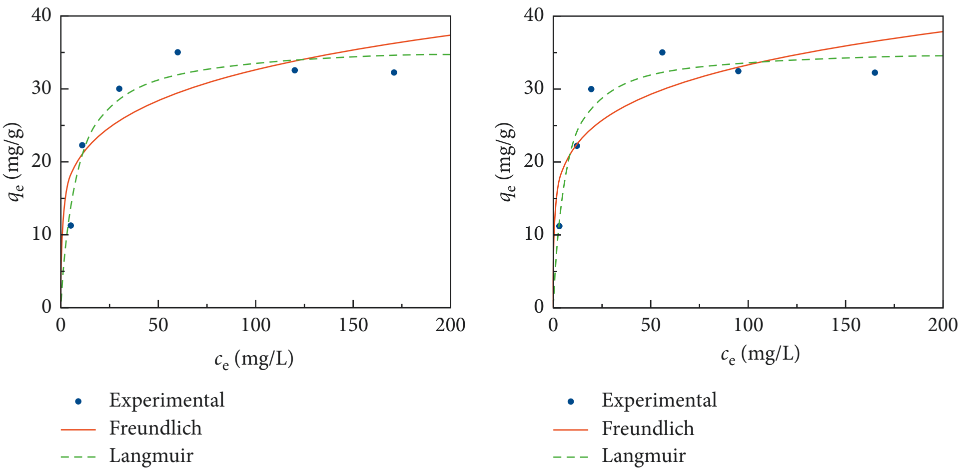
Table 2: Comparison of the optimized Freundlich and Langmuir isotherms parameters for the adsorption of chrysoidine on raw and chemically treated sawdust.

Sawdust	Freundlich			Langmuir			
	K_F (mg$^{(1-1/n)}$ g^{-1}·L$^{1/n}$)	n	OF (mg^2/g^2)	q_{max} (mg/g)	b (L/mg)	b (L/mol)	OF (mg^2/g^2)
Raw	9.689	3.690	145.74	33.670	0.208	51732.51	51.25
Activated	10.708	3.887	146.78	33.333	0.310	77101.34	53.54

adsorption of chrysoidine on raw and activated sawdust than Freundlich.

The parameters of Freundlich and Langmuir obtained from nonlinear fitting are reported in Table 3. The comparison of linear and nonlinear methods clearly reveals that the OF values are lower in case of nonlinear method.

Langmuir constant "b" estimated from the nonlinear method was then used to calculate R_L. Evolution of R_L with



(a)

(b)

Figure 8: Freundlich and Langmuir nonlinear isotherms of (a) raw sawdust and (b) chemically treated sawdust for chrysoidine removal.

Table 3: Comparison of the optimized Freundlich and Langmuir isotherms parameters for the adsorption of chrysoidine on raw and activated sawdust.

Sawdust	Freundlich			Langmuir			
	K_F (mg$^{(1-1/n)}$ g^{-1}·L$^{1/n}$)	n	OF (mg^2/g^2)	q_{max} (mg/g)	b (L/mg)	b (L/mol)	OF (mg^2/g^2)
Raw	13.054	5.045	114.12	36.094	0.127	31586.68	27.66
Activated	14.168	5.387	111.47	35.603	0.172	42778.81	23.61

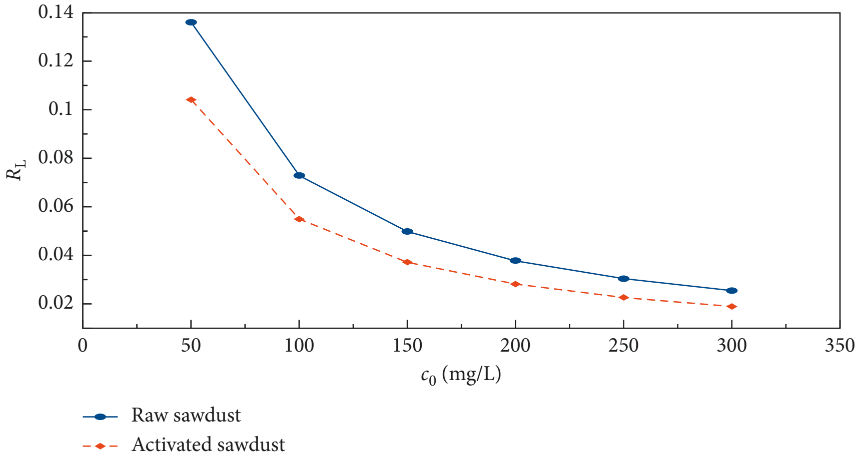


Figure 9: Effect of initial concentration on the Langmuir adsorption factor of raw and activated saw dust.

an initial dye concentration of raw and activated sawdust is shown in Figure 9. R_L of activated sawdust is lower (0.019 to 0.104) compared to raw sawdust (0.026 to 0.136), indicating better suitability of activated sawdust over raw sawdust for this adsorbent-adsorbate combination.

Recovery of the spent adsorbent is an important economic factor in determining the practicability of an adsorption system. However, sawdust is a wood by-product which is a waste material, available in plenty and bears no cost. Therefore, the recovery of adsorbent was not further pursued.

4. Conclusion

Sawdust, in both forms, proved to be an efficient adsorbent for chrysoidine dye from aqueous solution. This work has proved the potentiality of sawdust to be an effective adsorbent. The adsorption of chrysoidine followed Langmuir's isotherm. As sawdust is a cheap material and easily available, the process is expected to be economical. Although untreated sawdust proved to be less effective in comparison to treated sawdust, economically speaking, the percentage of dye removal is less than the difference in the commercial costs.

Data Availability

The adsorption data used to support the findings of this study are included within the article in the form of graphs. Data tables are not included just to avoid duplication.

Conflicts of Interest

The authors declare that there are no conflicts of interest regarding the publication of this paper.

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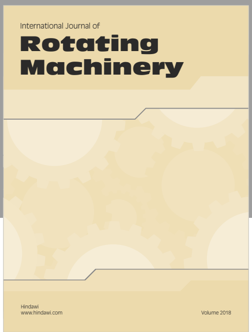
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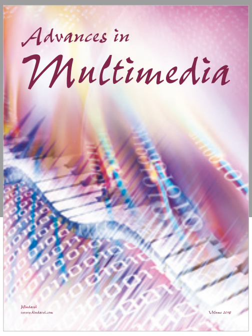
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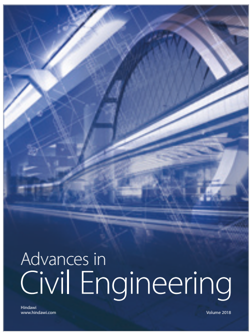
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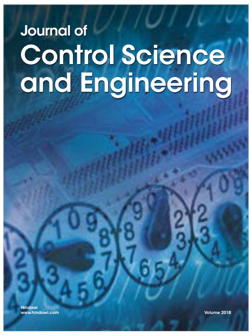
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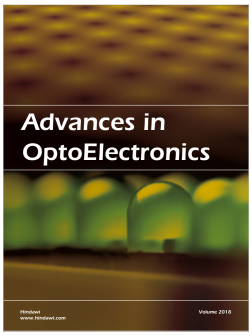
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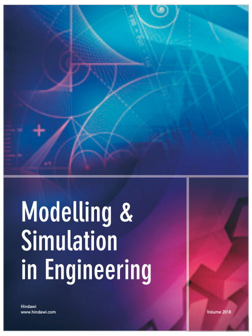
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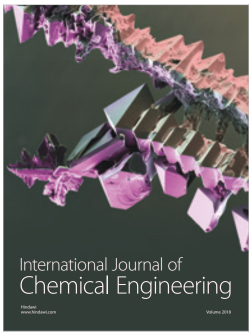
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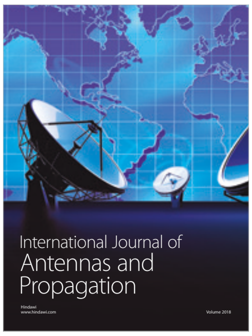
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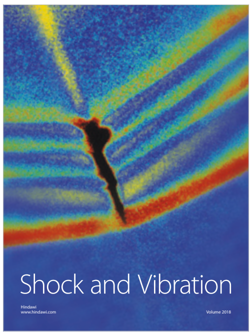
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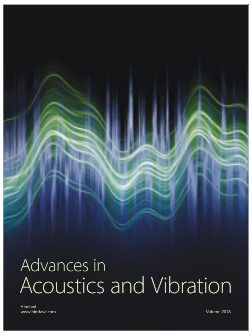
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