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Basic dye (methylene blue) removal from simulated wastewater by adsorption using Indian Rosewood sawdust: a timber industry waste

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Abstract

Dyes are usually present in trace quantities in the treated effluents of many industries. The effectiveness of adsorption for dye removal from wastewaters has made it an ideal alternative to other expensive treatment methods. This study investigates the potential use of Indian Rosewood (*Dalbergia sissoo*) sawdust, pretreated with formaldehyde and sulphuric acid, for the removal of methylene blue dye from simulated wastewater. The effects of different system variables, viz., adsorbent dosage, initial dye concentration, pH and contact time were studied. The results showed that as the amount of the adsorbent was increased, the percentage of dye removal increased accordingly. Higher adsorption percentages were observed at lower concentrations of methylene blue. Optimum pH value for dye adsorption was determined as 7.0 for both the adsorbents. Maximum dye was sequestered within 30 min after the beginning for every experiment. The adsorption of methylene blue followed a first order rate equation and fit the Lagergren equation well. Similar experiments were carried out with commercially available activated carbon to compare the results. Sulphuric acid treated sawdust or formaldehyde treated sawdust of Indian Rosewood can be attractive options for dye removal from dilute industrial effluents.

Keywords: Indian Rosewood; Sawdust; Adsorption; Batch mode; Methylene blue; Kinetics

1. Introduction

Colour is the first contaminant to be recognized in water and has to be removed from wastewater before discharging it into water bodies. Most of the industries in India, viz., textile, paper, printing, leather, food, cosmetics, etc. use dyes to colour their final product. In the dyeing section of a textile industry, about 1000 L of water is used for every 1000 kg clothes processed. Discharge of such coloured effluents imparts colour to the receiving water bodies (rivers and lakes) and interferes with its intended beneficial use. Colour impedes light penetration, retards photosynthetic activity, inhibits the growth of biota and also has a tendency to chelate metal ions which produce micro-toxicity to

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fish and other organisms [1]. It should be noted that the contamination of drinking water by dyes at even a concentration of 1.0 mg/L could impart significant colour making it unfit for human consumption.

Most of the used dyes are stable to photo-degradation, bio-degradation and oxidizing agents [2]. Currently, several physical or chemical processes are used to treat dye laden wastewaters. However, these processes are costly and cannot effectively be used to treat the wide range of dye wastewater. The advantages and disadvantages of some methods of dye removal from wastewaters are given in Table 1. The alum coagulation process is ineffective for the treatment of azoic, reactive, acidic and basic dyes. Conventional biological processes are effective for basic dyes removal only [3]. The stringent and rigid National Effluent Disposal Standards in India call for advanced treatment among which liquid phase adsorption has been found to be highly efficient for the removal of colour in terms of initial cost, simplicity of design, ease of operation and insensitivity to toxic substances. Activated carbon (powdered or granular) is the most widely used adsorbent because it has excellent adsorption efficiency for organic compounds, but its use is usually limited due to its high cost. Furthermore, regeneration using solutions produces a small additional effluent, while regeneration by refractory technique results in a 10–15% loss of adsorbent and its uptake capacity [4]. Consequently, a number of low cost, easily available materials are being studied for the removal of different dyes from aqueous solutions at different operating conditions (Table 2).

In our laboratory, the work is in progress to evaluate the possibility of the use of waste biomass for industrial wastewater pollution management. The studies on the use of Indian Rosewood sawdust as adsorbent are limited [6]. This is a common perennial tree present all over India. Timber of Indian Rosewood tree is widely used for furniture making and the waste sawdust so produced is generally used as cooking fuel due to its zero or negligible cost. The aim of the present study was to determine the optimum conditions for the removal of a dye, methylene blue, from simulated wastewater by formaldehyde treated sawdust and sulphuric acid treated sawdust carbon of Indian Rosewood.

2. Experimental

2.1. Adsorbents

2.1.1. Formaldehyde treated sawdust (SD)

Indian Rosewood (*Dalbergia sissoo*) tree sawdust collected from a local sawmill was washed with hot distilled water and then dried in sunlight until all the moisture evaporated. The material was ground to a fine powder in a still mill. The resulting material was sieved in the size range of 20–50 mesh ASTM. To immobilize the colour and water-soluble substances, the ground powder was treated with 1% formaldehyde in the ratio of 1:5 (sawdust:formaldehyde, w/v) at 50 °C for 4 h. The

Table 1
Advantages and disadvantages of the methods used for dye removal from industrial effluents [5]

Physical/chemical methods	Advantages	Disadvantages
Fentons reagent	Effective decolourisation	Sludge generation
Ozonation	No change in effluent volume	Shorthalf life (20 min)
Photochemical	No sludge generation	Formation of byproducts
NaOCl	Initiate azo-bond cleavage	Release of aromatic amines
Cucurbituril	Good sorption capacity for dyes	High cost
Electrochemical	Non-hazardous end products	High cost of electricity
Activated carbon	Highly effective for various dyes	Very expensive
Peat	Good adsorbent	Surface area is low
Silica gel	Effective for basic dyes	Side reactions in effluent
Membrane filtration	Removes all dyes	Concentrated sludge production
Ion exchange	No adsorbent loss	Not effective for all dyes

Table 2
Some low cost materials studied for dye(s) removal from aqueous solutions

Adsorbent(s)	Dye(s)	References
1. Bamboo dust, coconut shell, groundnut shell, rice husk	Methylene blue	[7]
2. Silk cotton hull, coconut tree sawdust, sago waste, maize cob	Rhodamine-B, Congo red, methylene blue, methyl violet, malachite green	[8]
3. *Parthenium hysterophorus*	Methylene blue, malachite green	[9]
4. Rice husk	Malachite green	[10]
5. Coir pith	Acid violet, acid brilliant blue, methylene blue, Rhodamine-B	[11,39]
6. Orange peel	Acid violet 17	[12]
7. Indian Rosewood	Malachite green	[6]
8. *Prosopis cineraria*	Malachite green	[13]
9. Banana and orange peels	Methyl orange, methylene blue, Rhodamine-B, Congo red, methyl violet, acid black 10B	[14]
10. Giant duckweed	Methylene blue	[4]
11. Banana pith	Congo red, Rhodamine-B, acid violet, acid brilliant blue	[15–18]
12. Orange peel	Congo red, Rhodamine-B, Procion orange	[19]
13. Carbonized coir pith	Acid violet, Rhodamine-B	[20]
14. Hardwood	Astrozone blue	[21]
15. Chitoson	Acid blue 25, basic blue 69	[22]
16. Chitin	Acid blue 25, basic blue 69	[22]
17. Biogas residual slurry	Congo red, Rhodamine-B, acid violet, methylene blue	[23,24]
18. Plum kernels	Basic red 22, acid blue 25	[25]
19. Rice husk	Safranine, methylene blue	[26]
20. Wheat straw, corn cob, barley husk	Cibacron yellow C-2R, Cibacron red C-2G, Cibacron blue C-R, Remazol black B, Remazol red-RB	[27]
21. Mahogany sawdust, rice husk	Acid yellow 36	[28]
22. Activated sludge biomass	Basic blue 3, basic violet 3, basic red 18, basic yellow 24, basic red 29, basic blue 47, basic blue 54	[29]
23. Fly ash	Acid orange 7, acid yellow 23, disperse blue 79, basic yellow 28, direct yellow 28	[30,37]
24. Perlite	Victoria blue, methyl violet	[31]
25. Fuller's earth	Methylene blue	[32]
26. Kaolinite	Methylene blue	[33]
27. Activated clay	Acid blue 9	[34]
28. Calcinated alunite	Acid blue 40, acid yellow 17	[35]
29. Cement kiln dust	Basic blue 3, basic red 22	[36]
30. Neem leaf powder	Brilliant green	[38]

sawdust was filtered out, washed with distilled water to remove free formaldehyde and activated at 80 °C in a hot air oven for 24 h. The material was placed in an airtight container for further use.

2.1.2. Sulphuric acid treated sawdust (SDC)

One part of dried SD was mixed with one part of concentrated sulphuric acid and heated in a muffle furnace for 24 h at 150 °C. The heated material was washed with distilled water and soaked in 1% sodium bicarbonate solution overnight to remove residual acid. The material was dried in an oven at 105 °C for 24 h and sieved in the size range of 20–50 mesh ASTM and used for the further study. SDC was characterized by adopting the standard procedures [40]. The various physico-chemical characteristics of SDC were: surface area = 98 m^2/g; apparent density = 1.45 g/ml; ash content = 1.68%; moisture content = 3.82%; CEC = 0.68 meq/g; water-soluble matter = 1.68%; acid soluble matter (4 N HCl) = 6.34%; 2 N NaOH soluble matter = 1.48%; and EC = 0.10 mS/cm. All adsorbents were dried at 110 °C overnight before the adsorption experiments.

2.1.3. Commercially available activated carbon (GAC)

GAC used in the present study was coconut shell based and was supplied by S.D. Fine Chemicals, Mumbai, India. It was used as such without further grinding and sieving. The general characteristics of GAC (as provided by the supplier) were: particle size = <300 mesh; surface area = 800 m^2/g; moisture = 3% (maximum); ash content = 2.5% (maximum); acid soluble = 2.5% (maximum); water-soluble = 1.5% (maximum), pH value = 6.5–7.5.

2.2. Preparation of simulated dye wastewater

The basic dye, methylene blue (S.D. Fine Chemicals, 85% dye content, chemical formula = $C_{16}H_{18}N_3SCl$; FW = 319; nature = basic blue; and λ_{max} = 665 nm) was used as such without further purification to prepare the simulated wastewater. An accurately weighed quantity of methylene blue (0.5635 g) was dissolved in double distilled water to prepare the stock solution (500 mg/L). Experimental solutions of the desired concentration were obtained by successive dilutions.

2.3. Experimental methods and measurements

In each adsorption experiment, 100 ml of dye solution of known concentration and pH was added to 0.4 g of GAC, SD or SDC in a 250 ml round bottom flask at 26±1 °C and the mixture was stirred on a rotary orbital shaker at 160 rpm. The samples were withdrawn from the shaker at predetermined time intervals, and adsorbent was separated from the solution by centrifugation at 4500 rpm for 5 min. The absorbance of the supernatant solution was estimated to determine the residual concentration. Residual dye concentration was determined using absorbance values measured before and after the treatment, at 665 nm with an Elico spectrophotometer (Model SL—150) using silica cells of path length 1 cm. The experiments were carried out at initial pH values ranging from 2–10; initial pH was controlled by the addition of dilute HCl or NaOH solutions. Kinetics of adsorption was determined by analyzing adsorptive uptake of the dye from aqueous solution at different time intervals. Two main system variables, initial dye concentration in the test solution and adsorbent dosage, were varied to investigate their effect on the adsorption kinetics. Blank runs, with only the adsorbents in 100 ml of double distilled water, were conducted simultaneously at similar conditions to account for any colour leached by the adsorbents and adsorbed by glass containers. Samples were diluted with double distilled water if absorbance values exceeded 0.900. Each experiment result was an average of three independent adsorption tests.

3. Results and discussion

3.1. Effect of adsorbent surface modification

Experiments were conducted with GAC, SDC and SD at constant adsorbent dosage (0.4 g/100 ml), pH (neutral), and temperature (26± 1 °C) for 3 h by varying methylene blue concentrations (50–500 mg/L). It is evident from Fig. 1 that GAC has more adsorption efficiency in comparison to SDC and SD at all initial dye concentrations studied. Decolouriztion of wastewater was 100%, 92% and 87.1% by GAC, SDC and SD, respectively, at 50 mg/L dye concentration. SDC and SD had almost similar adsorption efficiency if initial dye concentration in wastewater was up to 200 mg/L, but at higher dye concentrations

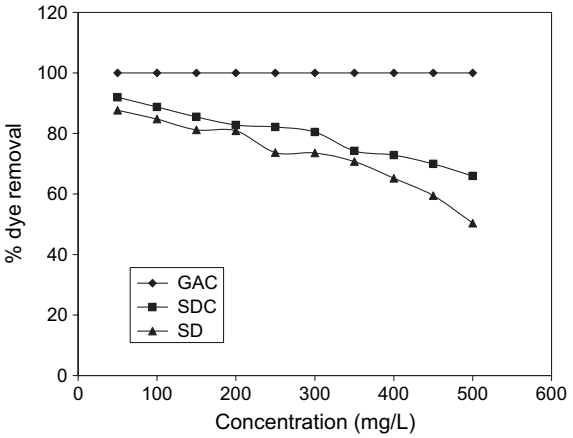

Fig. 1. Effect of adsorbent surface change on methylene blue adsorption (adsorbent dose = 0.4 g/100 ml; pH = neutral).

adsorption efficiency was in the order GAC > SDC > SD. At 500 mg/L concentration, GAC was 1.5 and 2.0 times more effective than SDC and SD, respectively. Whereas SDC was 1.3 times more effective than SD. The difference in adsorption capacity can be attributed to better physical structure and surface area of GAC than SDC and SD.

3.2. Effect of pH

To study the effect of pH on methylene blue adsorption on GAC, SDC and SD, the experiments were carried out at 250 mg/L initial dye concentration with 0.4 g/100 ml adsorbent mass at 26±1 °C for 3 h equilibrium time. Initial pH of dye solution was increased after the equilibrium time. The pH increase was lesser at lower pH values (2–4). It may be due to hydrolysis of the adsorbent in water, which creates positively charged sites. The dye adsorption by GAC was 100% in the studied pH range followed by SDC (78.3–80.2%) and SD (73.3–74.3%). The dye adsorption efficiency was not affected by pH except minor variations (Fig. 2) in the pH range of 2–10.

3.3. Effect of initial dye concentration

The influence of the initial concentration of methylene blue in the solutions on the rate of adsorption on GAC, SDC and SD was studied. The experiments were carried out at fixed adsorbent dose (0.4 g/100 ml) in the test solution, 26±1 °C temperature, pH (7.0) and at different initial concentrations of malachite green (50, 100, 150, 200 and 250 mg/L) for different time intervals (15, 30, 45, 60, 90 and 120 min). Dye removal by GAC was 100% at all the studied dye concentrations. Percent adsorption efficiency of SDC and SD decreased with increase in initial dye concentration in the solution (Table 3). Though the percent adsorption decreased with increase in initial dye concentration, the actual amount of dye adsorbed per unit mass of adsorbent increased with increase in dye concentration in the test solution. The unit adsorption for SDC was increased from 12.49 mg/g to 51.4 mg/g as the methylene blue concentration in the test solution was increased from 50 mg/L to 250 mg/L. Similarly, unit adsorption for SD was increased from 11.8 mg/g to 46.1 mg/g as the dye concentration in the test solution was increased from 50 mg/L to 250 mg/L. Maximum dye was sequestered from the solution within 15 min after the beginning for every experiment. After that, the concentration of methylene blue in the liquid phase remained almost constant. The equilibrium was established quickly within 15 min at all the studied concentrations by GAC. However, SDC and SD took about 30 and 45 min, respectively for equilibrium attainment. This may be due to the fact that SDC

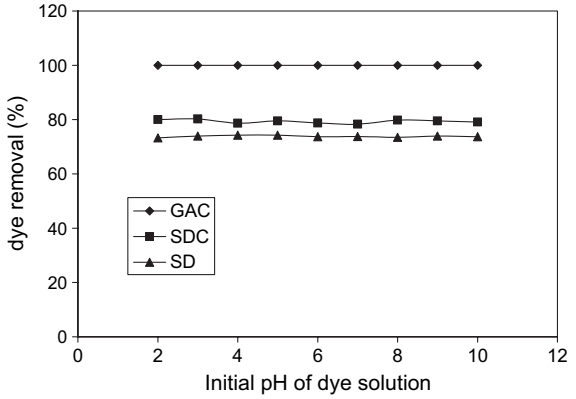

Fig. 2. Effect of pH on methylene blue adsorption (initial dye concentration = 250 mg/L; adsorbent dose = 0.4 g/100 ml; equilibrium time = 3 h).

Table 3
Effect of methylene blue concentration on dye adsorption

Initial dye concentration (mg/L)	Percent dye removal with time (min)					
	15	30	45	60	90	120
Sulphuric acid treated sawdust (SDC)						
50	99.3	99.4	99.5	99.7	99.8	99.9
100	95.7	97.2	97.6	98.8	98.8	98.9
150	87.2	87.6	88.0	89.0	90.1	91.3
200	82.9	83.9	84.2	85.2	86.9	87.4
250	80.0	80.2	81.0	81.1	81.8	82.2
Formaldehyde treated sawdust (SD)						
50	89.0	92.1	92.8	93.2	93.9	94.3
100	88.6	90.2	91.4	92.6	92.7	94.5
150	82.6	83.7	85.3	87.3	89.1	90.6
200	72.9	73.9	74.3	74.6	76.2	79.5
250	71.0	71.5	72.4	73.5	73.6	73.7

and SD have macro and micro pores. In the process of dye adsorption initially dye molecules have to first encounter the boundary layer effect and then it has to diffuse from boundary layer film onto adsorbent surface and then finally, it has to diffuse into the porous structure of the adsorbent. This phenomenon will take relatively longer contact time [28]. The time profile of dye uptake is a single, smooth and continuous curve leading to saturation, suggesting the possible monolayer coverage of dye on the surface of the adsorbent [28].

3.4. Effect of adsorbent mass

The adsorption of methylene blue on GAC, SDC and SD was studied by changing the quantity of adsorbent (0.2, 0.4, 0.6, 0.8 and 1.0 g/100 ml) in the test solution while keeping the initial dye concentration (250 mg/L), temperature (26±1 °C) and pH (7.0) constant at different contact times for 120 min. The percent adsorption was increased and equilibrium time was decreased with adsorbent dose (Table 4). The adsorption was 100% by GAC even at the minimum studied adsorbent dose (0.2 g/100 ml) within 15 min. The adsorption increased from 45.1 to 97.1%, as the SDC dose was increased from 0.2 g to 1.0 g/100 ml at equilibrium time (120 min). For SD, adsorption increased from 24.9 to 91.2% as the adsorbent dose was increased from 0.2 to 1.0 g/100 ml. Maximum dye removal was achieved within 30–45 min after which methylene blue concentration in the test solution was almost constant. Increase in the adsorption with adsorbent dose can be attributed to increased adsorbent surface area and availability of more adsorption sites. But unit adsorption decreased with increase in adsorbent dose. For SDC, unit adsorption was decreased from 56.4 mg/g to 24.3 mg/g as the adsorbent dose was increased from 0.2 to 1.0 g/100 ml in the test solution. For SD, unit adsorption was decreased from 31.1 mg/g to 22.8 mg/g as the SD dose was increased from 0.2 to 1.0 g/100 ml in the test solution. This may be attributed to overlapping or aggregation of adsorption sites resulting in decrease in total adsorbent surface area available to methylene blue and an increase in diffusion path length. Equilibrium time was lesser at higher adsorbent doses.

Table 4
Effect of adsorbent dose on methylene blue removal

Adsorbent dose (g/100 ml)	Percent dye removal with time (min)					
	15	30	45	60	90	120
Sulphuric acid treated sawdust (SDC)						
0.2	22.6	35.8	37.0	40.1	43.4	45.1
0.4	80.0	80.2	81.0	81.1	81.8	82.2
0.6	81.9	82.0	82.1	82.2	82.7	82.9
0.8	85.1	85.2	85.4	85.6	86.2	87.1
1.0	90.6	91.0	91.5	92.6	96.2	97.1
Formaldehyde treated sawdust (SD)						
0.2	11.9	22.8	23.1	24.2	24.6	24.9
0.4	71.0	71.5	72.4	73.5	73.6	73.7
0.6	75.6	77.5	78.2	79.3	79.7	80.4
0.8	80.2	81.1	82.8	82.7	83.6	83.9
1.0	85.5	86.9	90.2	90.6	91.0	91.2

3.5. Adsorption dynamics

The specific rate constants, k_{ad}, for the adsorption of malachite green on SDC and SD were calculated using the pseudo-first order Lagergren equation.

$$\log_{10}(q_e - q) = \log_{10} q_e - \frac{k_{ad} \times t}{2.303}$$

where q_e and q (both in mg/g) are the amounts of malachite green adsorbed at equilibrium time and at any time 't', respectively. The straight line plot of $\log(q_e - q)$ vs. t indicated the validity of Lagergren equation for the present system and also explained that the process followed the pseudo-first order kinetics. The values of k_{ad} calculated from the slopes of the plots were 0.0110 and 0.0128 min^{-1} for SDC at 0.2 g and 0.4 g adsorbent doses per 100 ml of the test solution and 0.0173 and 0.0254 min^{-1} for SD at similar adsorbent doses. These values are comparable with the results of other investigators [6,13,18,28].

4. Conclusion

The removal of methylene blue from simulated wastewater using GAC, SDC and SD has been investigated under different experimental conditions

in batch mode. The adsorption of methylene blue was dependent on adsorbent surface characteristics, adsorbent dose and methylene blue concentration in the wastewater. Maximum dye was removed within 30 min of the start of every experiment. The pH had very little effect on the methylene blue removal. Adsorption kinetics followed Lagergren first order kinetics model. GAC is an expensive material and regeneration is essential, whereas SDC and SD are cheap so regeneration is not necessary. Indian Rosewood is a common tree in India and its sawdust is easily available in the countryside at zero or negligible price. The data may be useful for designing and fabricating an economically cheap treatment process using batched or stirred-tank flow reactors for the removal of methylene blue from dilute industrial effluents.

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