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ABSTRACT

Bagasse fly ash, a waste generated in sugar industries in India, has been converted into an inexpensive adsorbent material and utilized for the removal of two basic dyes, rhodamine B and methylene blue. Results include the effect of pH, adsorbent dose, dye concentration, and presence of surfactant on the removal of rhodamine B and methylene blue. The adsorption data have been correlated with both Langmuir and Freundlich adsorption models. Thermodynamic parameters obtained indicate the feasibility of the process, and kinetic studies provided the necessary mechanistic information of the removal process.

Key Words. Adsorption; Basic dyes; Bagasse fly ash; Wastewater; Removal; Solid waste utilization

INTRODUCTION

Dyes are widely used in various fields, and their discharge into water causes environmental pollution. Effluents from industries such as carpet manufacturing, dyeing, pulp and paper, and textiles, are rich in color. And thus are easily detected. These effluents readily deteriorate the quality of the receiving waters

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and soil. The ingestion of such wastewaters by humans causes pain, skin irritation, vomiting, severe headaches, and acute diarrhea. Such effluents are also responsible for water-borne diseases exhibiting symptoms such as hemorrhage, nausea, dermatitis, ulceration of the skin and mucous membranes, kidney damage, and a loss of bone marrow leading to anemia (1, 2). The removal of color from wastewater can be carried out by flotation (3), chemical coagulation (4, 5), chemical oxidation (6), and by adsorption (7–12). Adsorption hold promise in the treatment of wastewater, as it is inexpensive, simply designed, easy to handle, and provides sludge-free cleaning operations. Commercially activated carbon has long been used as a standard adsorbent for color removal. In spite of its widespread use in various cleaning procedures, activated carbon remains expensive; therefore, the development of low-cost alternative adsorbents has been the focus of recent research. The efforts made in this direction include the use of clays (13, 14), metal hydroxides (15), bagasse pith (16–18), sunflower stalks (19), hardwood (20, 21), fertilizer, and steel wastes (12) for the removal of dyes from wastewater and other aqueous solutions.

The sugar industry is one of the most important agricultural industries in India. Bagasse fly ash, a waste material of sugar industries, causes a disposal problem. It is used as a space filler in building materials, but to date, no widely used application of this waste has been found. Earlier attempts successfully utilized wastes of some major industries for the removal of various pollutants (12, 22–24), and recently, bagasse fly ash was also converted into an effective adsorbent for the removal of metal ions (25) and phenols (26). The aim of the present research is to use the bagasse fly ash to remove two important basic dyes, rhodamine B and methylene blue, from aqeuous solutions. These dyes are widely used in various industries for coloring, as well as other purposes, e.g., rhodamine B after-treatment gives permanent dyeing properties and forms colored complexes with antimony, which is used for detecting antimony in biological fluids (27, 28). Methylene blue is used for biological staining, particularly for nerve tissues. It is also a reagent for testing for tubercular infection in milk and acts as an analgesic and antiseptic (29). The results of the removal of these dyes are discussed in this communication.

MATERIALS AND METHODS

All reagents used were of AR grade (Aldrich). Stock solutions of rhodamine B (1×10^{-3} M) and methylene blue (1×10^{-2} M) were prepared in double-distilled water.

Equipment

A pH meter (model CT No. CL46; Toshniwal, India) was used to measure pH. X-ray measurements were made by a Phillips X-ray Diffractometer employing

nickel filtered Cu-K_α radiation. Surface area of the sample was measured by a Model QS-7 Quantasorb Surface Area Analyser. Infrared spectra of the sample were recorded on an infrared spectrophotometer [Model (FTIR) Perkin Elmer 1600]. Absorbances were recorded on a DU-6, UV-VIS spectrophotometer.

Material Development

Bagasse fly ash, a solid waste, was obtained from a sugar manufacturer at Bijnor (U.P.) India. This material was treated with hydrogen peroxide (100 Volume) at 60°C for 24 h, until bubbling stopped, indicating the complete oxidation of adhering organic impurities. The resulting product was washed with distilled water, dried at 100°C for 24 h, and seived to desired particle sizes through 100–150, 150–200, 200–250, and 250–300 BSS mesh. Finally, the product was stored in a vacuum desiccator.

Estimation of Dyes

The concentration of coloring matter in each solution was determined by calibration plots drawn by recording the absorbance of dyes at different concentrations on a DU-6 spectrophotometer. All measurments were made at wavelengths corresponding to the maximum absorbance $\lambda max = 554$ nm for rhodamine B, and $\lambda max = 665$ nm for methylene blue. Furthermore, the effect of pH on the λmax of rhodamine B and methylene blue was also studied, and these values did not change with the change in pH.

Adsorption Studies

Adsorption studies were performed by batch technique to obtain rate and equilibrium data. A series of 50-ml stoppered test tubes were employed, each having 10 ml of dye solution of varying concentrations [rhodamine B (1×10^{-3} to 1×10^{-5} M) and methylene blue (1×10^{-2} to 1×10^{-3} M)] at desired pH and temperature. An optimized amount of adsorbent (10 gL^{-1} for rhodamine B, and 4 gL^{-1} for methylene blue) was then added into each test tube and agitated intermittently for 24 h. Preliminary investigations showed that equilibrium is attained in 8–10 h. Shaking for any time between 8–24 h gave practically the same uptake. As such, all adsorption experiments were run after equilibrating 10 ml of adsorbate solution with a fixed amount of adsorbent of 200–250 BSS mesh size for 24 h. The effect of pH was determined by studying the sorption of dyes at a fixed concentration over a pH range of 2.0–10.0. Adjustments to pH were made by dilute acid and dilute base. Sorption experiments were run at 30, 40, and 50°C to study the effect of temperature. The influence of an anionic detergent (Manoxol-1B) on the uptake of rhodamine B and methylene blue was studied as a function of detergent concentration at fixed pH. Stoppered glass tubes containing adsorbate solution along with the detergent and fixed amounts of adsorbent were equilibrated for

48 h with intermittent shaking. The supernatant obtained on centrifugation was analyzed for adsorbate concentration.

Kinetic Studies

Kinetic investigations of rhodamine B and methylene blue on bagasse fly ash were carried out by batch technique because of its simplicity. A number of stoppered glass tubes containing a definite volume (10 ml in each case) of solutions of adsorbates of known concentration (5×10^{-5} M of rhodamine B and 5×10^{-3} M of methylene blue) were placed in a thermostat-cum-shaking assembly. When the desired temperature was reached, a known amount of adsorbent was added to each test tube. At predecided intervals the solutions of the specified tubes were seperated from the sorbent materials and concentrations of rhodamine B and methylene blue were determined. Equilibrium was attained in about 8–10 h.

RESULTS AND DISCUSSION

The adsorbent developed from bagasse fly ash was found to be of "L" type as classified earlier (25); i.e., it lowers the pH when kept in deionized water. It is quite stable in water, salt solutions, acids, and bases. Chemical composition and characteristics of the bagasse fly ash determined by standard methods of chemical analysis (30) are SiO_2: 61.44; Al_2O_3: 14.50; CaO: 2.82; Fe_2O_3: 4.86; MgO: 0.71 and loss on ignition: 17.12 wt%. The porosity of the material was 0.36 (fraction) with a surface area of 440 $m^2 \cdot g^{-1}$. The X-ray diffraction pattern (Fig. 1) of the product provide d-spacing values that reflect the presence of minerals like goethite, mullite, hematitie, kaolinite, α-quartz, γ-alumina, etc. in the

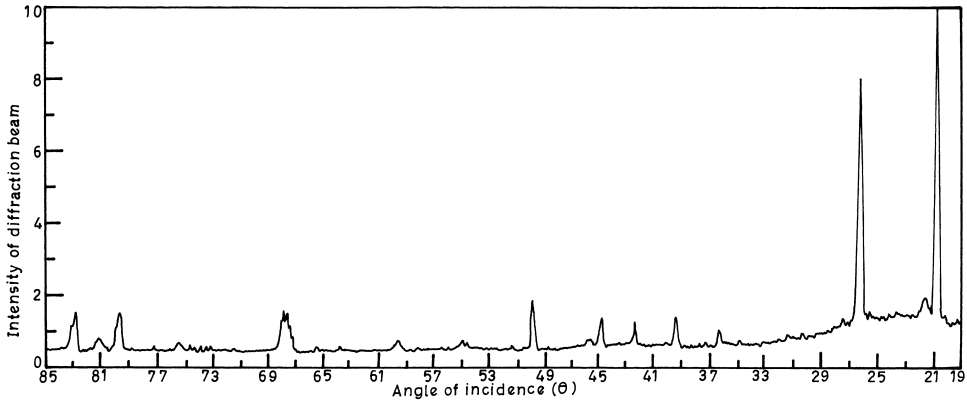


FIG. 1 X-Ray diffraction pattern of bagasse fly ash.

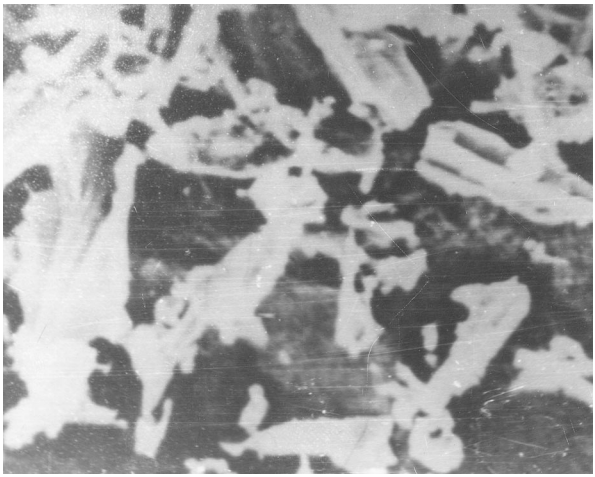
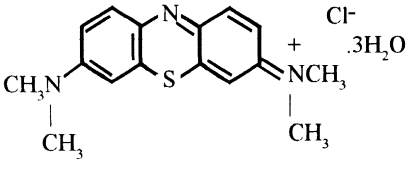


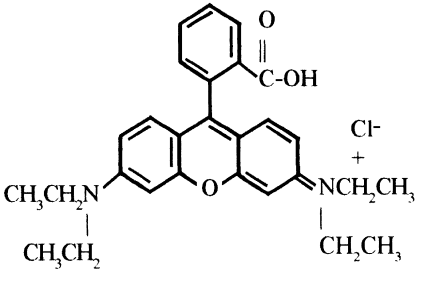
FIG. 2 SEM photograph of bagasse fly ash at 320 magnification.

waste material. Bands observed at 1172, 1157, and 680 cm^{-1} in the IR spectrum (not shown here) of bagasse fly ash were attributed to Si–O stretching vibrations, a characteristic of quartz. The adsorption bands at 3696 and 3670 cm^{-1} indicate the presence of kaolinite. A very weak band at 358 cm^{-1} was due to the presence of hematite. The presence of calcium silicate was suggested by a strong band at 470 cm^{-1} (31). The surface area of the adsorbent as calculated by the BET method was found to be 440 $m^2 \cdot g^{-1}$. A SEM photograph of bagasse fly ash (Fig. 2) at 320 × magnification clearly reveals the porous texture of the product. The material was so porous that it could be easily crushed by hand.

Figure 3 depict the variation of adsorption with pH. Rhodamine B is strongly adsorbed at pH 2, whereas methylene blue is strongly adsorbed at pH 8. The variation of adsorption with pH can be explained by considering the



Structure of methylene blue



Structure of rhodamine B

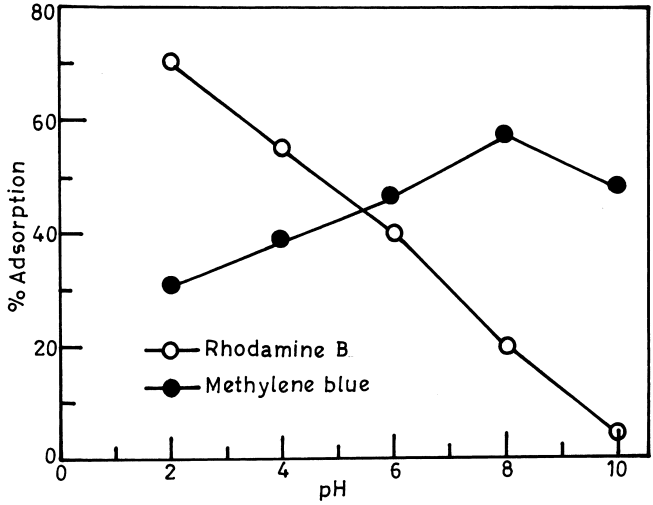


FIG. 3 Effect of pH on the adsorption of rhodamine B and methylene blue on bagasse fly ash.

difference in the structure of the two dyes as given below, as well as the point-of-zero charge (ZPC) of the bagasse fly ash (which is 5.8).

The main constituents of bagasse fly ash are silica and alumina. The ZPC of silica is 2.3, while that of alumina is 8.2 (25), and as such the surface of bagasse fly ash would have high positive charge density below pH 5.8, i.e., ZPC of the bagasse. Under these conditions the uptake of positively charged methylene blue would be low; with increasing pH, the negative-charge density on the surface increases, resulting in enhanced removal. The decrease in adsorption, with an increase in pH after pH 2, in the case of rhodamine B can be attributed to the presence of an acidic group in the dye that may dissociate as the pH increases, giving rise to a negative charge on the dye molecule.

To optimize the adsorbent concentration for the removal of rhodamine B and methylene blue from the solutions, adsorption studies were carried out with different adsorbent doses. After a few trial runs, it was observed that the amount of adsorbent affected the adsorption of adsorbate. Although the percentage of dye removal was higher at high adsorbent concentrations, a large quantity of the adsorbent dose was comparatively less efficient if the percentage of dye removal per gram of adsorbent was considered. Morever, large quantities of adsorbent create handling problems. In the case of rhodamine B, when the adsorbent dose was increased from 5 to 10 gL^{-1} the uptake went up by about 30%, while on further increasing it to 20 gL^{-1}, the uptake went up only by 2.0% (Fig. 4). Similarly for methylene blue, 4 gL^{-1} adsorbent dose was found to be optimum as the adsorption increased 20% when the amount was changed from 2

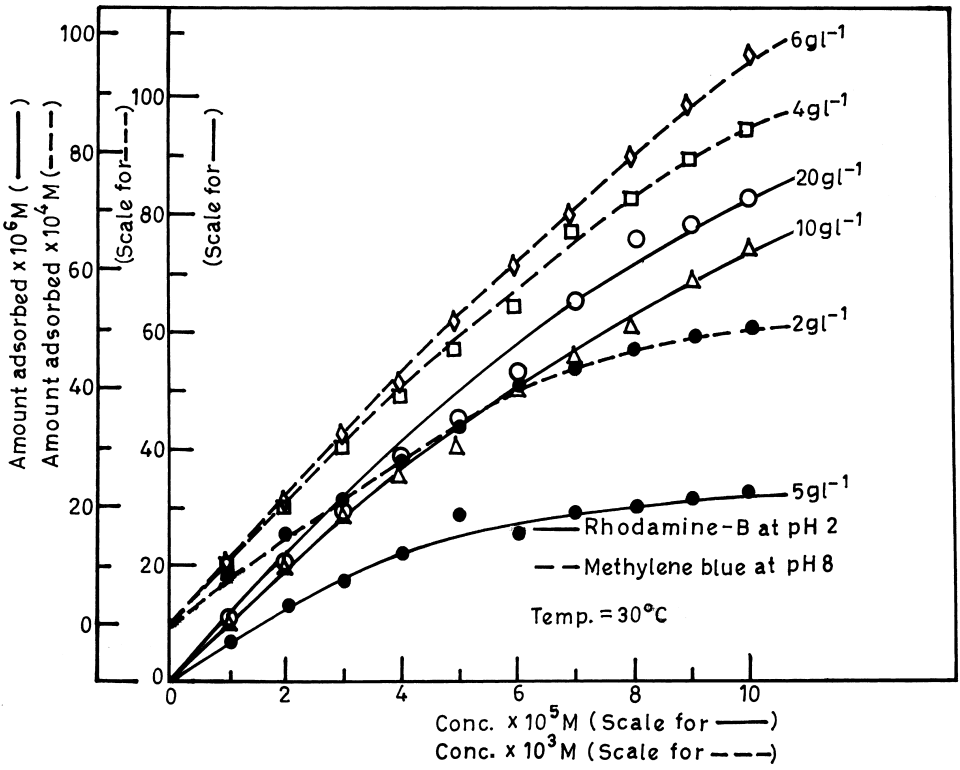


FIG. 4 Effect of adsorbent dose on the adsorption of rhodamine B and methylene blue on bagasse fly ash (temperature 30°C; particle size 200–250 BSS mesh).

gL^{-1} to 4 gL^{-1}, and 2.5% when it was increased to 6 gL^{-1} (Fig. 4). Hence, 10 gL^{-1} and 4 gL^{-1} of bagasse fly ash were used for the removal of rhodamine B and methylene blue, respectively. It is thus apparent that the adsorbent doses of less than 10 gL^{-1} and 4 gL^{-1} of bagasse fly ash for the removal of rhodamine B and methylene blue respectively gave very poor adsorption capacities.

Various fractions of particle size, e.g., 100–150, 150–200, 200–250, 250–300 BSS mesh, have been tried, and it was found that the removal of both the dyes increases with increasing mesh size. However, the difference in the percentage of removal with 200–250 and 250–300 BSS mesh was very small. Furthermore, because of the handling problems with smaller particle sizes, especially in the column studies, the particles of mesh size 200–250 were selected for the present studies. Hence, all the adsorption studies on both dyes were carried out on the adsorbent of particle size 200–250 mesh (63 μm diameter).

Adsorption isotherms were run at concentrations ranging from 10^{-4} to 10^{-5} M in the case of rhodamine B, and 10^{-2} to 10^{-3} M in the case of methylene blue, at different temperatures. These concentrations were chosen after much preliminary investigation. All the isotherms (Fig. 5) are regular and concave to the equilibrium concentration axis, indicating a favorable adsorption. The uptake is almost 100% at low dye concentrations and 60–80% at higher concentrations. This indicates the efficiency of the adsorbent for the removal of rhodamine B and methylene blue. Thus, the dyes can be removed 100% for concentrations $\leq 1.0 \times 10^{-5}$ M in case of rhodamine B, and $\leq 1.0 \times 10^{-3}$ M in case of methylene blue. Furthermore, the adsorption decreased with an increase in temperature, indicating that the process is exothermic.

Freundlich and Langmuir isotherms have been employed to study the adsorption behavior of rhodamine B and methylene blue. These two models

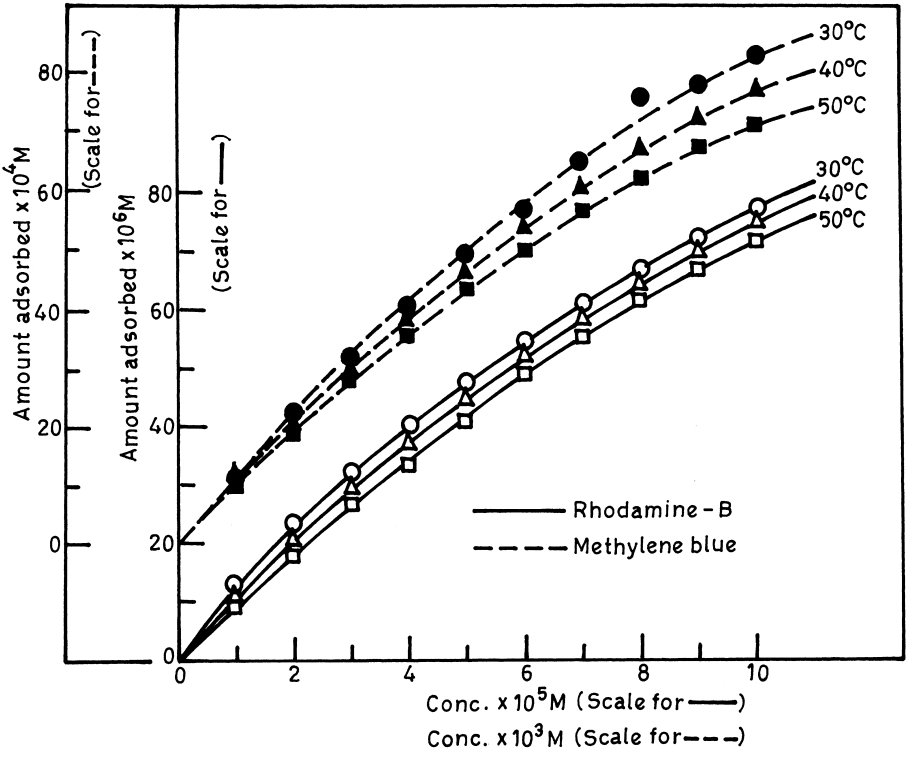


FIG. 5 Adsorption isotherms of rhodamine B and methylene blue on bagasse fly ash at different temperatures.

(Eqs. 1 and 2 respectively) are widely used, the former being empirical and the latter assuming that maximum adsorption occurs when the surface is covered by a uniform monolayer of adsorbate.

$$q_e = K_F C^{1/n} \tag{1}$$

$$q_e = \frac{Q^0 bC}{1 + bC} \tag{2}$$

where q_e is the amount of solute adsorbed per unit of weight of adsorbent (bagasse fly ash), C is the solute phase concentration, and Q^0 is the solid phase concentration corresponding to complete coverage of available adsorption sites. The value of K_F can be used as an alternative measure of adsorption capacity, while $\frac{1}{n}$ determines the adsorption intensity, and b signifies the equilibrium constant of the adsorption process.

It is seen from the linearity of plots in Figs. 6 (a, b) and 7 (a, b) that adsorption follows both models. The values of Freundlich and Langmuir constants are listed in Table 1. The adsorption capacity K_F is less for the rhodamine B–bagasse fly ash system than that for the methylene blue–bagasse fly ash system, whereas the intensity of adsorption, which is calculated from the slope $1\backslash n$, presents the reverse trend. The value of Q^0 (i.e., maximum uptake) increases with temperature increase, thereby confirming that the process is exothermic. The value of Q^0 is significantly higher for the rhodamine B–bagasse fly ash system compared to methylene blue on the same adsorbent.

The dimensionless separation factor, R (32) is calculated from the Langmuir isotherm by Eq. 3. The factor was 0.77 for rhodamine B and 0.04 for methylene blue. This indicates a favorable adsorption ($R < 1$).

$$R = \frac{1}{1 + b\, C_0} \tag{3}$$

The changes in standard Gibbs free energy (ΔG^0), enthalpy (ΔH^0), and entropy (ΔS^0) were obtained using expressions 4, 5, and 6 (25), which are given in Table 2.

$$\Delta G^0 = -RT \ln b' \tag{4}$$

$$\ln\left[\frac{b_1}{b_2}\right] = -\frac{\Delta H}{R}\left[\frac{1}{T_1} - \frac{1}{T_2}\right] \tag{5}$$

$$\Delta G^0 = \Delta H^0 - T\Delta S^0 \tag{6}$$

where b', b_1, and b_2 are Langmuir constants at 30, 40, and 50°C, and were obtained from the slopes of Langmuir isotherms. Other terms have their usual significance.

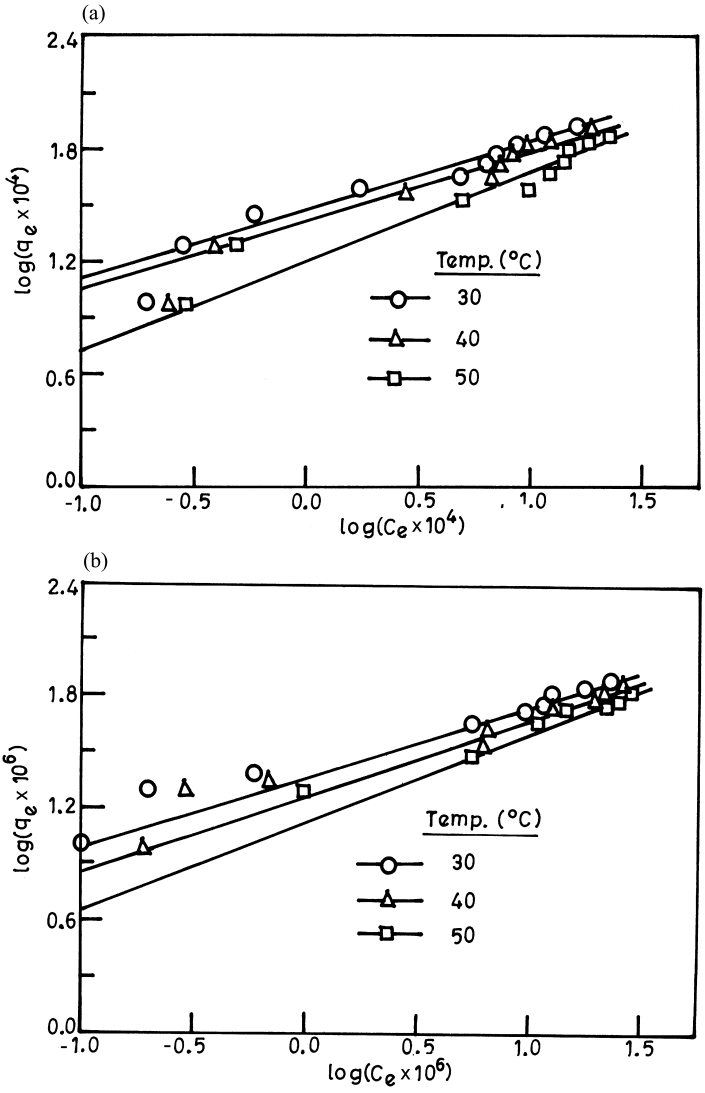


FIG. 6 Freundlich plots of rhodamine B and methylene blue at 30, 40, and 50°C on bagasse fly ash.

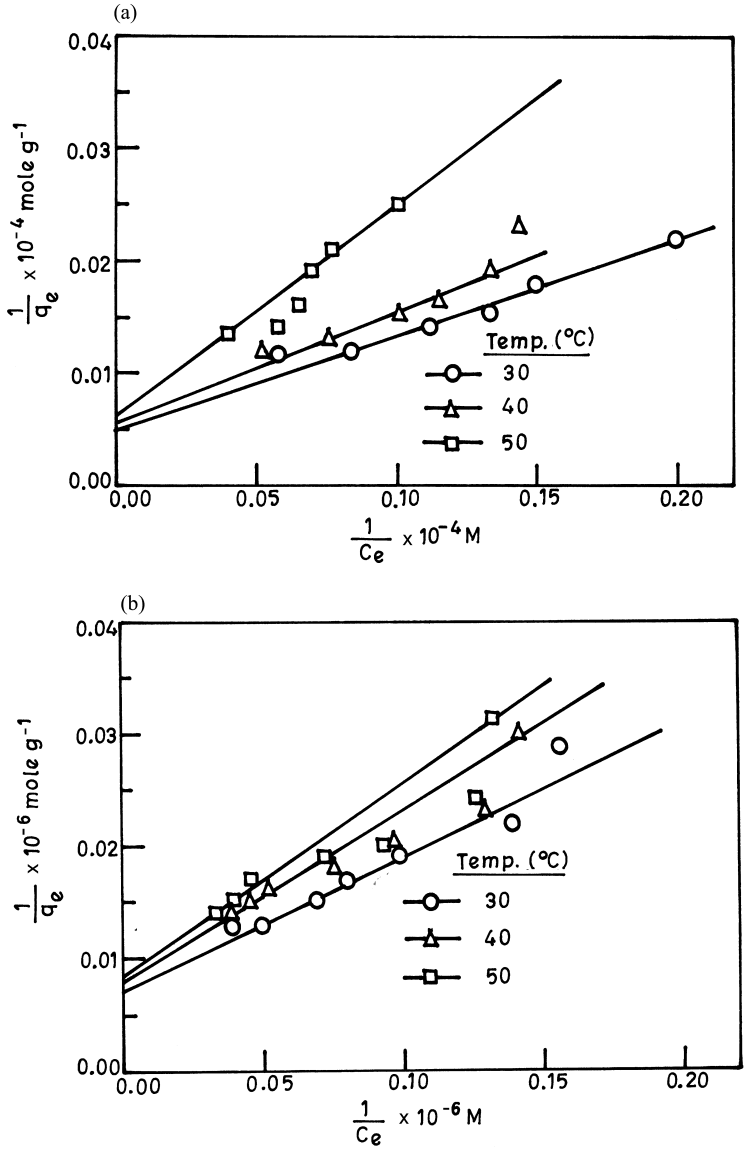


FIG. 7 Langmuir plots of rhodamine B and methylene blue at 30, 40, and 50°C on bagasse fly ash.

TABLE 1
Freundlich and Langmuir Constants for the Adsorption of

	n			$K_F \times 10^3$		
Adsorbate	30°C	40°C	50°C	30°C	40°C	50°C
Rhodamine B	2.54	2.46	2.17	0.102	1.38	0.219
Methylene blue	2.86	2.67	2.08	7.58	8.91	18.40

The negative-free-energy values indicate the feasibility of the process and its spontaneous nature without any induction period. Negative ΔH^0 values reflect the exothermic nature of the process, whereas small negative values of entropy change, ΔS^0, suggest that no noticiable change in the structure of bagasse fly ash takes place during the adsorption process.

Natural water is commonly contaminated by a large number of inorganic and organic pollutants from commercial as well as domestic effluents. Anionic detergent is the most likely of these contaminants to be encountered. The effect of the anionic detergent Manoxol-IB on the adsorption process has therefore been studied by equilibirating along with the detergent a fixed amount of adsorbent (10 gL^{-1} for rhodamine B, and 4 gL^{-1} for methylene blue) at optimum pH (2 and 8, respectively) and at fixed adsorbate concentrations. The results are presented in Fig. 8. The effect of ionic interactions on the adsorption process has been interpreted, using the ratio of the adsorption capacity of the ion in a single-component system (q_0) to that in a multi-component system (q_m) (33) such that if

$q_m/q_0 > 1$ adsorption is promoted by the presence of other interfering ions

$q_m/q_0 = 1$ no observable net interaction effect

$q_m/q_0 < 1$ adsorption is surpressed by the presence of other interfering ions

TABLE 2
Thermodynamic Parameters for Rhodamine B and Methylene Blue

	$-\Delta G$(k J mol^{-1})				
Adsorbate	30°C	40°C	50°C	$-\Delta H$ (k J mol^{-1})	$-\Delta S$ (J mol^{-1} K^{-1})
Rhodamine B	32.757	32.757	33.279	24.684	+27.0
Methylene blue	22.245	22.286	20.643	47.082	−82.5

Rhodamine B and Methylene Blue

$Q^o \times 10^3$ (mol g^{-1})			$b \times 10^{-3}$ (1 mol^{-1})		
30°C	40°C	50°C	30°C	40°C	50°C
14.3	12.5	11.8	44.4	29.3	24.1
0.202	0.182	0.167	0.684	0.524	0.218

The presence of surfactant does not effect the uptake of rhodamine B significantly (Fig. 8); it goes down by 1.5 percent ($q_m/q_o \approx 1$), while a decrease in the scavenging efficiency ($q_m/q_o < 1$) in the case of methylene blue is observed as the uptake goes down by about 10%.

Preliminary investigations on the rate of removal of dyes by bagasse fly ash (at optimum pH and adsorbent dose) indicate the process to be quite rapid. Nearly 80–85% of the sorption capacity is realized within the first hour of con-

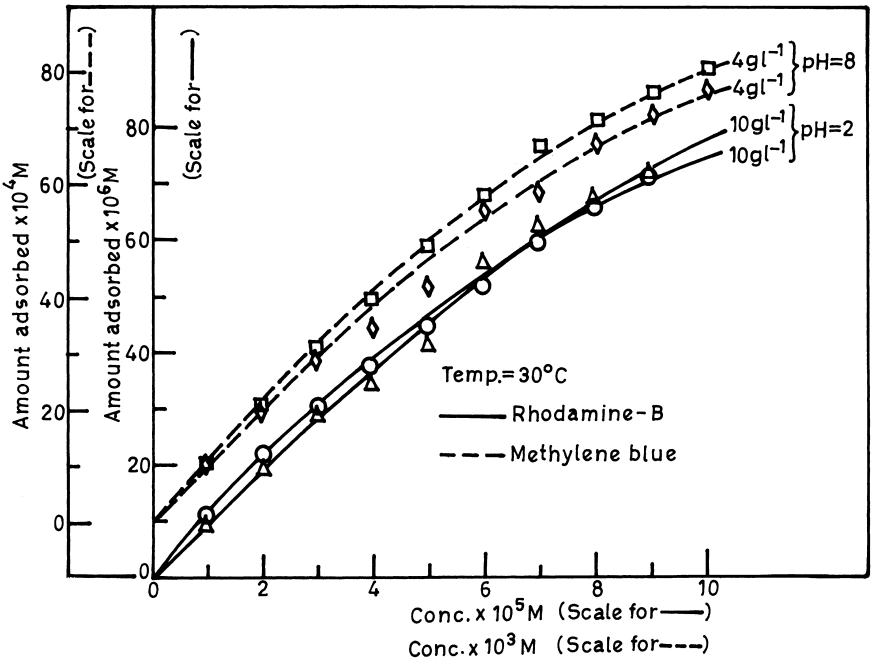


FIG. 8 Effect of manoxol-1B on the adsorption of rhodamine B and methylene blue.

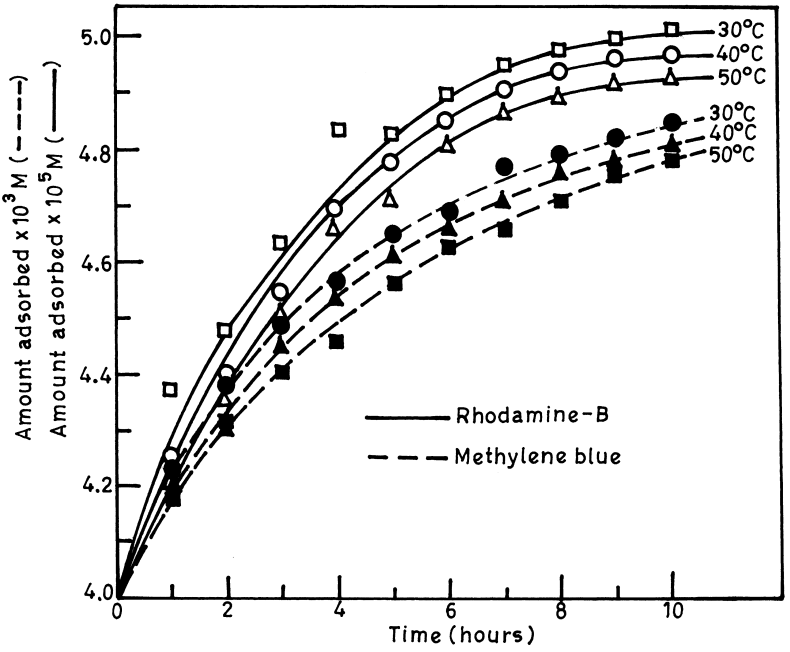


FIG. 9 Effect of temperature on the rate of adsorption of rhodamine B and methylene blue.

tact. The initial rapid adsorption gives way to a very slow rate of approach to equilibrium (reached in 8–10 h) as shown in Fig. 9.

To interpret the experimental data it is necessary to identify the steps involved in the process of adsorption that govern the overall rate of removal. Various parameters were calculated by using Eqs. 7 and 8 as given by Boyd et al. (34) and Reichenberg (35).

$$F = 1 - \frac{6}{\pi^2} \sum_{n=1}^{\infty} \frac{1}{n^2} \exp\left[\frac{-D_i t \pi^2 n^2}{r_o^2}\right] \tag{7}$$

$$F = 1 - \frac{6}{\pi^2} \sum_{n=1}^{\infty} \frac{1}{n^2} \exp[-n^2 B t] \tag{8}$$

where F is the fractional attainment of equilibrium at time t and is obtained by the expression (9).

$$F = Q_t / Q_\infty \tag{9}$$

$$B = \pi^2 \frac{D_i}{r_o^2} = \text{time constant} \tag{10}$$

where Q_t is the amount of uptake at time t, and Q_o is the maximum equilibrium uptake, D_i is the effective diffusion coefficient of adsorbate in adsorbent phase, r_o = radius of the adsorbent particle assumed to be spherical, and n is an integer. The values of energy of activation E_a have been determined by applying the Arrhenius-type expression (11).

$$D_i = D_o \exp\left[-\frac{E_a}{RT}\right] \tag{11}$$

The pre-exponential constant D_o (analogous to the Arrhenius frequency factor) gives the entropy of activation $\Delta S^{\#}$ (Eq. 12)

$$D_o = 2.72\, d^2 \left[\frac{kT}{h}\right] \exp\left[\frac{\Delta S^{\#}}{R}\right] \tag{12}$$

where d is the average distance between the successive exchange sites and is taken as 5 Å.

The D_i values for the rhodamine B–bagasse fly ash and methylene blue–bagasse fly ash systems at different temperatures are listed in Table 3. It may be argued that the effective diffusion coefficient, D_i in these systems, is mainly comprised of two components, which are due to simultaneous diffusion of the ingoing ions through the pores of different widths and different electronic fields along the diffusion path. The diffusion within the pores of wider widths and weaker retarding forces of electrostatic interaction (particularly at the surface of the adsorbent) accounts for the faster one, and the one within the pores of narrower-mesh widths and stronger retarding forces accounts for the slower component of D_i. Increased mobility of ions and a decrease in the retarding forces acting on the diffusing species result in the enhancement of D_i values with temperature.

The linearity test of Bt versus t plots (not shown) is used to distinguish between film and particle-diffusion-controlled rates of adsorption. Bt values were derived from Eq. 8 for the observed values of F, obtained from Reichenberg's table (35). In the case of rhodamine B, the Bt-versus-time plots were linear at

TABLE 3
Effective Diffusion Coefficients (D_1), D_0, E_a and $\Delta S^{\#}$ Values for Rhodamine B and Methylene Blue at Different Temperatures

	D_i (m^2 sec^{-1}) × 10^{11}			D_0	E_a	$-\Delta S^{\#}$
Adsorbate	30°C	40°C	50°C	(m^2 sec^{-1}) × 10^{13}	(k J mol^{-1})	(J K^{-1} mol^{-1})
Rhodamine B	4.08	3.88	3.67	2.30	4.36	81.75
Methylene blue	3.26	3.06	2.96	1.54	3.91	85.10

lower concentrations ($\leq 5.0 \times 10^{-5}$ M) and did not pass through the origin, indicating a film-diffusion process. At higher concentrations ($> 5.0 \times 10^{-5}$ M) the plots were linear up to a certain time and passed through the origin, indicating that the rate was controlled by particle diffusion. However, in case of methylene blue the plots were linear but did not pass through the origin up to a concentration of ($<1.0 \times 10^{-3}$ M), indicating a film-diffusion mechanism as a rate-controlling step up to this concentration, and beyond this concentration the plots were linear and passed through origin, signifying particle diffusion as a sole rate-determining step at these concentrations.

The values of E_a, D_o, and $\Delta S^{\#}$ for the diffusion of rhodamine B and methylene blue are also given in Table 3. The value of E_a is higher for the rhodamine B–bagasse fly ash system than for the methylene blue–bagasse fly ash system. The negative $\Delta S^{\#}$ values reflect that no significant change occur in the internal structure of bagasse fly ash during the adsorption of rhodamine B and methylene blue.

Cost Estimation

The uptake of rhodamine B and methylene blue on bagasse fly ash obtained from a local sugar company is quite comparable to other commercially available adsorbents. In India, the cheapest variety of commercially available carbon costs U.S. \$285 per ton^{-1}. The waste bagasse fly ash is available for U.S. \$2 per ton^{-1}, and considering the cost of transport, chemicals, and electrical energy used, the finished product would cost approximately U.S. \$12 per ton^{-1}. Recovery of dyes and the chemical regeneration of columns, if possible, may further reduce costs. Hence the developed adsorbent may be a good alternate to commercially available carbons for color removal because of its comparable efficiency and a significantly low cost.

CONCLUSION

Bagasse fly ash, a solid waste of the sugar industry, has been converted into an inexpensive adsorbent material. This product exhibits very good adsorption capacity for the removal of dyes. The uptake of rhodamine B and methylene blue is comparable to other commercially available adsorbents. The material under consideration is not only economical, but is a waste product. Hence, its use as an adsorbent on one hand would solve the problem of its disposal, and on the other hand would provide an effective adsorbent for the treatment of dye-contaminated water and wastewater.

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