J. Chem. Eng. Data **2010,** *55,* 5653–5657

Removal of Some Common Textile Dyes from Aqueous Solution Using Fly Ash

Debabrata Chatterjee,* Vidya Rupini Patnam, Anindita Sikdar, and S. K. Moulik

Chemistry and Biomimetics Group, Central Mechanical Engineering Research Institute, CSIR, Durgapur-713 209, India

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Fly ash samples collected from the Durgapur Thermal Power Station (DTPS) (Durgapur, West Bengal, India) were designated in regard to their respective collection points, that is, the discharge point (DP), the midpoint between the discharge point and the end point (MP), and the end point (EP) of the ash pond. F1 and F3 represent samples collected from field 1 and field 3 of the electrostatic precipitator (ESP), respectively. The fly ash samples were characterized physicochemically. The removal of some common textile dyes using the fly ash samples has been investigated spectrophotometrically as a function of fly ash dosage, dye concentration, and contact time at ambient conditions. The results of this investigation revealed that the sample F3 was the best among the fly ash samples collected from DTPS. Adsorption studies using a visible light irradiated system have been performed, and the spectral analysis of the photolyzed solution revealed that decoloration of the concerned dyes was greater compared to that observed in the dark under identical experimental conditions.

Introduction

Elimination of toxic and bioresistant organic pollutants from wastewater by their transformation into nonhazardous species is a demanding area of research. Dyes and pigments represent one of the problematic groups of chemicals that are hardly destroyed in conventional wastewater treatment plants.

The adsorption process perhaps provides an attractive alternative for the treatment of water contaminated with colorants, especially if the adsorbent is inexpensive and does not require any pretreatment step before its application. Although most commonly used adsorption agents in industry are activated carbon,[1,2] their high operating costs and regeneration of the spent carbon hamper their large-scale application.[3] Other materials which include peat,[4] chitin,[5] silica,[6] hardwood sawdust,[7,8] bagasse pitch,[9] paddy straw,[10] rice husk,[11] slag,[12] acid-treated spent bleaching earth,[13] palm fruit bunch,[14] barley straw,[15] bone char,[16] and natural zeolites[17] have also been examined, but their level of success is not much greater. In this content we are attracted to use fly ash for the adsorption of organic molecules as it is inexpensive and potentially viable. Although there has been continued interest in developing colorant removal processes using fly ash,[18−30] a consensus on the use of fly ash toward the removal of colorants or pollutants from industrial effluents has not yet been established. More importantly, the use of UV−vis (ultraviolet−visible) light for such processes has hardly been explored. Recently, we have reported the degradation of colorants over a visible light illuminated TiO_2 semiconductor particulate system.[31,32] The present work is conceived on the basis of the capability of fly ash toward adsorbing organic molecules and the vulnerability of surface adsorbed organic dyes toward aerial oxidation in the presence of light.[33] In this work fly ash samples were collected from the Durgapur Thermal Power Station (DTPS) (Durgapur, West Bengal, India) and have been used for the adsorption of various dyes of textile importance. The adsorption capacities of various fly ash samples were determined from parameters of adsorption isotherms. The results of the present studies explore the possibility of using fly ash for eco-friendly removal of dyes in textile wastewaters.

Table 1. UV−vis Spectral Data of the Dyes Selected for This Work

color index name of the dyes	λ_{max}/nm
reactive black B	599
reactive blue 3R	610
reactive Turkish blue 3RS	580
reactive yellow M3R	435
reactive orange M2R	480
reactive red M5B	540
reactive red 11	545
reactive yellow HE4G	440
reactive orange HER	496
reactive red HE7B	544
methylene blue	663
rhodamine B	557

Experimental Section

Materials. Fly ash samples were collected from different locations at DTPS, Durgapur. The samples are designated as DP, MP, EP, F1, and F3 in regard to their respective collection points, that is, the discharge point (DP), the midpoint between the discharge point and the end point (MP), and the end point (EP) of the ash pond. F1 and F3 represent samples collected from field 1 and field 3 of the electrostatic precipitator (ESP). The experimental work was carried out with the collected samples without further treatment. Different dyes, namely, vinyl sulfone-based (reactive black B, reactive blue 3R, reactive Turkish blue 3RS), monochlorotriazine-based (reactive yellow M3R, reactive orange M2R, reactive red M5B), and dichlorotriazine-based (reactive yellow HE4G, reactive orange HER, reactive red HE7B), were used in the present study. Some standard emissive dyes, methylene blue and rhodamine B, were also used in the present investigation. Dye samples were procured at a locally available commercial source, and some were collected from a textile industry (Exodus Knitwear Private Ltd., Bishnupur, Bankura, West Bengal). Spectral properties of dyes selected for this study are shown in Table 1.

Instrumentation. UV−vis spectral measurements were performed on a Perkin-Elmer (model Lambda 35) spectrophotom-

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* Corresponding author. Phone: +91-343-6510263. Fax: +91-343-2546745. E-mail: dchat57@hotmail.com.

10.1021/je100536e

Table 2. Physicochemical Characterization Data of Fly Ash Samples

parameter	DP	MP	EP	F1	F3
bulk density/kg$\cdot$m^{-3}	1.45	0.83	0.83	0.94	0.73
particle size/μm	97.5	40.7	35.6	49.1	19.2
BET surface area/m$^2\cdot$gm^{-1}	1.74	2.42	3.43	2.03	7.97
mineralogical properties (by XRD)	quartz phase[a]	quartz phase[a]	quartz phase[a]	quartz phase[a]	quartz phase[a]
magnetic separates/% (w/w)	1.6	0.27	0.28	0.6	1.9
nonmagnetic separates/% (w/w)	98.4	99.7	99.7	99.4	98.1
loss on ignition/%	12.6	9.1	5.7	8.4	10.6
pH	7.4	7.0	7.1	5.7	4.9
conductivity/μS$\cdot$cm^{-1}	80.7	179.1	176.1	171.7	118.7
TDS/mg$\cdot$L^{-1}	80.3	89.7	87.7	85.2	78.9
carbon/% (w/w)	3.2	2.7	1.6	3.2	2.1
sulfur/% (w/w)	0.03	0.01	0.02	0.02	0.02

[a] Usual quartz phase with trace amounts of mullite.

eter. The particle size, Bruanuer−Emmett−Teller (BET) surface area, and X-ray diffraction (XRD) analysis of the fly ash samples were carried at CGCRI, Kolkata using a Malvern laser particle size analyzer (Mastersizer 2000 MU, U.K.), surface area analyzer (1750 Sorpty, Carlo Erba, Italy), and Phillips diffractometer (model PW 1730), respectively. The particle size of the fly ash samples was measured in a water dispersant and calculated using the respective particle size distribution plot, and the value corresponding to $d(0.5)$ was considered. Conductance or total dissolved solid (TDS) measurements were carried out with a MC 226 Mettler Toledo conductivity meter, whereas pH measurements were undertaken by using a Mettler Toledo pH/ion analyzer (model MA 235). The analysis of heavy metals was performed on a Perkin-Elmer atomic absorption spectrometer (model AAnalyst 300). The analysis of carbon and sulfur was carried out with an automated carbon−sulfur analyzer (Horiba make; model EMIA-320 V). Multidistilled water obtained from a Monodest 3000E quartz multidistillation apparatus was used throughout the experiment. The pH of the system was adjusted by using HCl and NaOH.

Adsorption Studies. The adsorption of the colorants or pollutants selected in this work was performed by batch experiments. The batch technique was selected because of its simplicity. In a typical experiment, a solid fly ash sample (5 g) was taken in 100 mL of dye solution of varying concentrations in a bottle (250 mL) generally used for biological oxygen demand estimation. The bottles were agitated mechanically for 60 min at constant temperature. Shaking of the reaction mixture was carried out horizontally using a locally made shaker (TIMC, Kolkata). Thereafter, the suspensions were allowed to settle for 30 min for sedimentation and centrifuged for 15 min (12 000 rpm) using a Remi centrifuge (R-24). While studying the effect of shaking time at shorter time scales [(10 and 20) min], the resultant solution was taken directly from the reacting bottle through a membrane filter using a syringe (the tip of which was fitted with a membrane filter) and subjected to spectral measurement. The final equilibrium concentration (C_e) of the colorant in the supernatant liquid was measured spectrophotometrically (after proper dilution) at the absorption maximum (λ_{max}). The percentage removal of the dye was calculated using eq 1:

$$\text{percentage removal (\%)} = 100(C_o - C_e)/C_o \qquad (1)$$

where C_o and C_e are initial and equilibrium concentration (expressed in mg$\cdot$L^{-1}) of the dye, respectively.

Photocatalytic Studies. Photocatalytic experiments were performed using a 150 W xenon lamp (Oriel Illuminator 7340). In a typical photocatalytic experiment, an aqueous suspension (50 mL) of colorant of varying concentrations containing 100 mg of fly ash sample was taken in a glass reactor. The temperature of the reacting mixture was kept constant by circulating water through the outer jacket of the reactor. The preaerated reaction mixture was illuminated with a 150 W xenon lamp under continuous magnetic stirring. A filter solution containing sodium nitrite, copper sulfate, and ammonium hydroxide was used to eliminate light at < 420 nm. The distance between the lamp and the glass reactor containing the reaction mixture was fixed at 20 cm. Under these conditions, the photon flow entering the reactor, measured by chemical actinometry, was found to be $3.5\cdot10^{-6}$ einstein/s. The actinometer was irradiated under conditions similar to those used for the photoreaction, thus eliminating the need for making corrections for the reflectance and nonuniformity of the incident light beam. Oxygen was purged from time to time to replenish the reacting system. At a chosen interval of irradiation time, aliquots of the reaction mixture were withdrawn, centrifuged, and filtered by using a membrane filter (0.1 μm), and the extent of dye decoloration was determined spectrophotometrically at the absorption maximum (λ_{max}) of the concerned dye. Chemical oxygen demand (COD) measurements were carried out with a titrimetric method. The resultant solution (5 mL) obtained at the end of the photolysis was shaken with 10 mL of dichloromethane, and the organic layer was subjected to gas chromatography−mass spectra (GC-MS) studies on a GC-MS-MS (Thermo Electron Corporation) equipped with a PolarisQ mass detector. However, results were inconclusive in regards to the identification of any particular intermediate because of complicated mass spectra. Sulfate, nitrate, and ammonium ions were analyzed using a Mettler Delta ion meter (model MA 350) using corresponding ion-selective electrodes.

Results and Discussion

Physicochemical Characterization of Fly Ash Samples and Textile Dyes. Physicochemical characterization data of the five fly ash samples designated as DP, MP, EP, F1, and F3 are summarized in Table 2. All five fly ash samples showed almost identical XRD patterns. The major phase for all fly ash samples was quartz, with minor amounts of mullite, hematite, and magnetite. On the basis of the results of particle size and BET surface area measurements of the five fly ash samples (Table 2). it is apparent that the fly ash sample designated “F3” is promising for superior adsorption, because it has a smaller particle size and higher surface area. The composition of the various fly ash samples collected has been provided in the Supporting Information (see SI1). The presence of heavy metals, namely, Cd, Cr, Pb, Ni, Co, and Hg, in all fly ash samples was found to be appreciably low [(10 to 60) mg$\cdot$kg^{-1}], and the

Table 3. Adsorption (%) of Various Textile Dyes (Collected from Exodus Knitwear Pvt., Ltd.) over Various Fly Ash Samples[a]

fly ash samples	reactive black B	reactive blue 3R	reactive Turkish blue 3RS	reactive yellow M3R	reactive orange M2R	reactive red M5B	reactive yellow HE4G	reactive orange HER	reactive red HE7B
DP	54	77	64	76	72	74	46	60	67
MP	87	93	90	82	88	90	82	74	83
EP	98	100	95	87	97	99	88	82	98
F1	88	94	93	83	83	87	87	75	100
F3	100	100	100	100	100	100	100	100	100

[a] Fly ash taken = 5 g, volume of sample solution = 50 mL, concentration of dye = 100 mg·L^{-1}, time of shaking = 1 h, pH = 8.5, temp. = 25 °C.

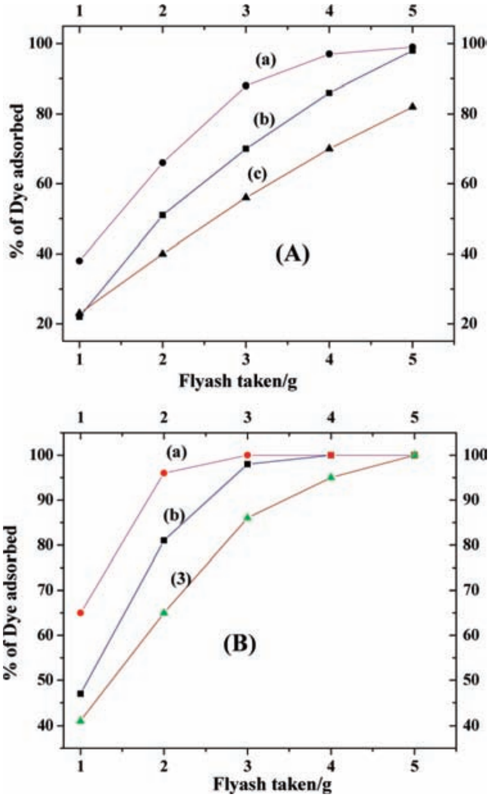

Figure 1. Effect of fly ash doses on the adsorption (%) of various textile dyes over fly ash samples (A) EP and (B) F3. (a) Reactive red M5B, (b) reactive black B, and (c) reactive orange HER. Volume of dye solution = 100 mL, shaking time = 1 h. Concentration of dye = 100 mg·L^{-1}, temp. = 25 °C.

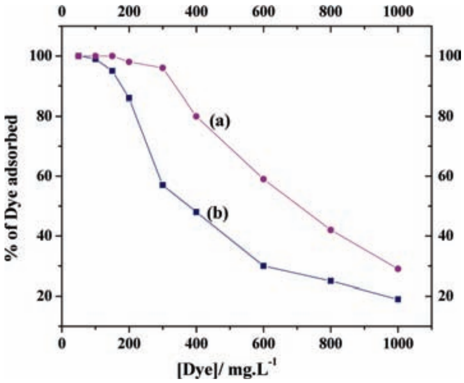

Figure 2. Effect of dye concentration on the adsorption (%) of reactive red M5B over fly ash samples (a) F3 and (b) EP. Volume of dye solution = 100 mL, shaking time = 1 h. Amount of fly ash sample taken = 5 g, temp. = 25 °C.

leaching of heavy metals from the fly ash samples was found to be very low (at the μg·kg^{-1} level), which is consistent with an earlier report on the leachability of heavy metals from coal fly ash.[34]

Adsorption Studies. Results of preliminary experiments concerning adsorption of the dyes (Table 3) selected for this work reveal that EP was the best performing sample among the fly ash samples collected from the ash pond, whereas F3 was the best among the samples collected from the ESP in regards to adsorption of the dyestuffs. However, the adsorbing capacity of F3 is much higher than that of EP. Therefore, further adsorption studies as a function of fly ash dose, shaking time, and dye concentration have been performed only with EP and F3. Results of the adsorption of the dyes studied by estimating the percentage of dye discoloration as a function of fly ash dosage are shown in Figure 1. In all cases the dye uptake by fly ash was enhanced with an increase in the amount of fly ash. For a given amount of fly ash, the percentage of unadsorbed dye increases with an increase in the initial concentration of dye as shown in Figure 2 for reactive red M5B. Figure 3 displays the results of the effect of shaking time on the extent of adsorption (%) of the dyes on EP and F3 samples of fly ash. No appreciable difference in the % adsorption of the three classes of dyes used in this study has been noticed in the concentration range studied. Since all dyes under investigation (Table 3) exist as ionic species, especially in the pH range close to that of the untreated effluent, no significant influence of pH on the adsorption was noticed. At such pH values, when the dyes are in the ionized form, adjacent molecules of the dyes on the hybrid adsorbent surface will repel each other to a significant degree, because of their equal electrical charge. In contrast, when the adsorbing species is not ionized, no such electrical repulsion exists, and thus the packing density on the surface can be much higher. This explains the common observation that the nonionized form of acidic and basic compounds adsorb more than their ionized counterparts. The above results clearly demonstrate that, without any pretreatment, the fly ash samples effectively adsorb the selected dye samples of textile importance. In particular, the results obtained with the fly ash sample designated as F3 are the best among all of the fly ash samples selected for this investigation. The higher dye removal efficiency shown by the F3 sample is probably associated with its larger surface area and smaller particle size. The capacity of F3 and EP toward the adsorption of azo-dyes is shown in Figure 1.

Photocatalytic Studies. Adsorption studies with a visible light irradiated system have also been performed to explore the possibility of dye removal from textile wastewater through adsorption on fly ash and followed by the photocatalytic degradation of the surface adsorbed dye. For this purpose, the fly ash samples EP and F3 were used. Spectral analysis of the photolyzed solution revealed that decoloration of the concerned dye is appreciably more as compared to that observed in dark under identical experimental conditions (Table 4). GC-MS analysis of the photolyzed solution for identification of the reaction intermediate(s) was difficult due to complicated GC-MS spectra; however, GC-MS peaks and their multiple MS-MS fragmentation patterns confirmed the presence of 1-amino-2-naphthol, sulfonated aminonaphthalenes, 1,2-naphthoquinone, and oxalic acid as intermediates in the photoirradiated reacting

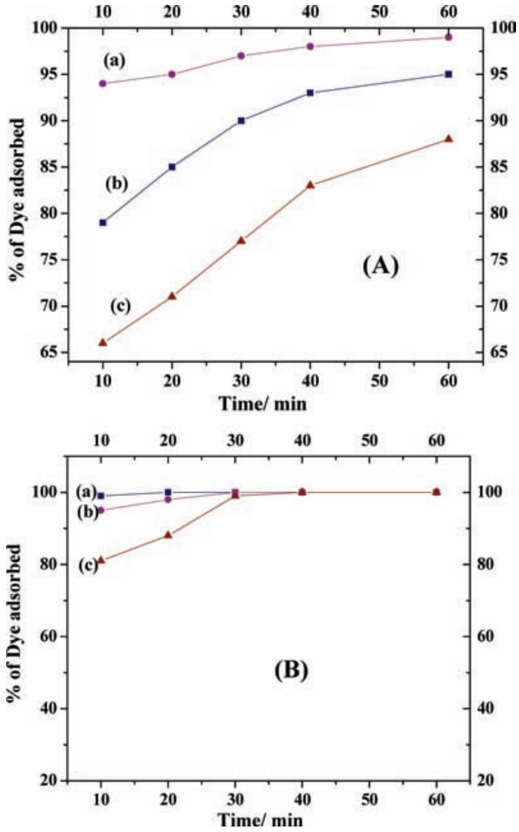

Figure 3. Effect of shaking time on the adsorption (%) of various dyes over fly ash samples (A) EP and (B) F3. (a) Reactive red M5B, (b) reactive black B, and (c) reactive orange HE. Volume of dye solution = 100 mL, [Dye] = 100 mg·L^{-1}. Amount of fly ash sample taken = 5 g, temp. = 25 °C.

Table 4. Results of Dye Removal over Visible Light Irradiated Fly Ash Samples

	% of dye removal			
	EP[a]		F3[b]	
dye	dark	light	dark	light
reactive red M_5B	53	71	18	38
reactive orange HER	23	40	11	28
reactive black B	22	19	14	39
methylene blue	65	85	15	35
rhodamine B	28	76	14	27

[a] Fly ash taken = 1 g. [b] Fly ash taken = 0.1 g; volume of sample solution = 50 mL, concentration of dye = 50 mg·L^{-1}, temp. = 25 °C.

system. Controlled experiments revealed no degradation product(s) in the absence of oxygen in the reacting system, thus confirming the essential role of oxygen in colorant decomposition over visible light illuminated fly ash samples. No change in the spectral pattern of the dye was observed when the air equilibrated aqueous solution of the respective dye was illuminated with visible light for 5 h. This necessarily confirmed that the dyes selected for this investigation do not undergo self-degradation under visible light illumination without a catalyst. Photocatalytic experiments carried out with emissive dyes, namely, methylene blue and rhodamine B, revealed better results as compared to the nonemissive reactive dyes (except for reactive red) in regards to their removal from the system (Table 4).

On the basis of the experimental results (Table 4) and considering Fe_2O_3 (present in the fly ash) participates in catalyzing the oxidation of the dyes at the excited state, the following working mechanism (Scheme 1) is reminiscent of semiconductor-mediated photocatalytic pollutant degradation[35] and is proposed for the visible light-assisted degradation of dyes adsorbed on the fly ash.

Scheme 1

$$\mathrm{FA} \xrightarrow{h\nu} \mathrm{FA}(h^+ + e^-_{CB}) \quad (1)$$

$$\mathrm{FA}(h^+ + e^-_{CB}) + O_2 \longrightarrow \mathrm{FA}(h^+) + O_2^- \quad (2)$$

$$O_2^- + H^+ \longrightarrow HO_2 \quad (3)$$

$$\mathrm{FA}(h^+) + \mathrm{D} \longrightarrow \mathrm{FA} + \mathrm{D}^+ \quad (4)$$

$$O_2^-/HO_2 + \mathrm{D}^+/\mathrm{D} \longrightarrow \mathrm{Product(s)} \quad (5)$$

(hv = UV-vis light; FA = flyash; D = Dyes selected for this work)

We have estimated the Fe content of the fly ash samples by adopting the reported procedure[36] and found that the percentage composition of Fe_2O_3 is 3.23 % and 3.54 % in EP and F3, respectively. The observed values of Fe_2O_3 composition are in close agreement with the range reported for Fe_2O_3 content in Indian fly ash.[37] Upon excitation with visible light, the semiconducting Fe_2O_3 electron is generated at the conduction band (eq 1), which is subsequently taken by molecular oxygen to produce a highly active superoxide radical (eq 2). This radical species repeatedly attacks the dye species, ultimately causing the degradation of the dyes (eq 5) under investigation. It is noteworthy here that the role of visible light irradiated hematite (Fe_2O_3) as a semiconductor photocatalyst in the oxidation of sulfite, oxalate, citrate, benzoate, and thiols has been well-documented in literature.[38,39] The extent of dye decoloration under the specified reaction conditions is not markedly changed for different dye molecules used in this investigation (Table 4). This perhaps suggests that the rate-controlling step in the above decoloration reactions is governed by the adsorption efficiency of dye molecules on to the surface of the fly ash samples.

Conclusion

In conclusion, the results of the present work reveal that the capacity of fly ash samples collected from DTPS (West Bengal, India) toward the adsorption of dyes can occur without any expensive pretreatment. In particular, the results obtained with the sample designated as F3 were the best among the fly ash samples selected for this investigation. The viability of using fly ash for the removal of colorants from textile effluents in the presence of visible light is implied. There is great scope for an increased understanding of the mechanistic possibilities concerning the involvement of sunlight and a cheap oxidant like H_2O_2, which should lead to further improvement in effluent treatment. Studies pertinent to this matter are in progress.

Acknowledgment

D.C. thanks Prof. Goutam Biswas, Director of the Central Mechanical Engineering Research Institute, for his encouragement.

Supporting Information Available:

Composition of the fly ash samples collected. This material is available free of charge via the Internet at http://pubs.acs.org.

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Received for review May 22, 2010. Accepted November 1, 2010. This work was supported by a research grant (No. T-122(232-05)FAUP/TIFAC) under the fly ash utilization program mission, TIFAC, Department of Science and Technology, Government of India, New Delhi.

JE100536E