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Anuradha Mishra[a]; Malvika Bajpai[a]; Sushant Pandey[a]
[a] Department of Chemistry, University Institute of Engineering and Technology, CSJM University, Kanpur, India

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Removal of Dyes by Biodegradable Flocculants: A Lab Scale Investigation

Anuradha Mishra, Malvika Bajpai, and Sushant Pandey
Department of Chemistry, University Institute of Engineering and Technology, CSJM University, Kanpur, India

Abstract: In the present communication, experiments were conducted to investigate the efficiency of mucilage isolated from fruits of Coccinia *indica* for the treatment of simulated textile wastewater samples containing direct dyes, direct fast scarlet (DFS) and direct fast yellow (DFY) and vat dyes, golden yellow (GY) and nyanthrene yellow (NY). This mucilage (Ku) is an ecofriendly and low cost anionic polysaccharide capable of reducing color from textile effluent through flocculation process. The flocculation efficiency of Ku was improved by grafting polyacrylamide onto it. The copolymer thus obtained (Ku-g-PAM) showed much better flocculation capacity than that of its precursor by reducing the flocculant dose and treatment time to half. The results showed that the maximum removal was obtained at acidic pH with both the flocculants. Statistical analysis showed that the change in percent removal with pH was highly significant in case of direct dyes while it was significant in case of vat dyes removal. The plausible mucilage-dye interaction and flocculation mechanism has been discussed.

Keywords: Natural polysaccharide, grafting, polyacrylamide, direct dyes, vat dyes

INTRODUCTION

The production and finishing of textiles is often one of the most important industries in many developing countries. Wastewater from textile industries creates a great problem of pollution due to the dyes contained therein. Due to biorecalcitrant nature of modern dyes, little dye would be decolorized or

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Address correspondence to Anuradha Mishra, Department of Chemistry, University Institute of Engineering and Technology, CSJM University, Kanpur 208 024, India. E-mail: anuradha_mishra@rediffmail.com

degraded, even if the wastewater were to be discharged to a conventional biological treatment plant. Environmental regulation in most European countries has made it mandatory to decolorize the dye wastewater prior to discharge (1), as dye molecules are toxic to organisms (2).

The treatment of dyes in industrial wastewater poses several problems, as the dyes are generally stable to light and oxidation (3). Several methods have been developed for the treatment of textile effluents such as, wet oxidation, advanced chemical oxidation, nanofilteration by membranes, adsorption using activated carbon and electrocoagulation using aluminium or iron soluble electrodes (4–10). Coagulation/Flocculation is widely used in water and wastewater treatment and may be accomplished by inorganic coagulants or organic polymers. Organic polymers usually are favored, although more costly, as they tend to produce less sludge than their inorganic counterparts.

Many natural polymers such as guar gum, starch, alginic acid (11) are often used as flocculants to find an ecofriendly wastewater treatment process. Recently, we have also reported the use of some natural as well as vinyl monomers grafted natural polymers for the treatment of various types of wastewater (12–14). In the present study, a brief evaluation of the effectiveness of pure Coccinia *indica* mucilage and its polyacrylamide (PAM) grafted copolymer as flocculants for color removal has been done. The variables studied are the flocculant dose, contact time and pH. Two-way analysis of variance (ANOVA) was applied for determining the statistical significance of the percent removal of the dyes.

EXPERIMENTAL

Materials and Methods

Kundoor mucilage was extracted from the fruits of Coccinia *indica*. The fruits were thoroughly washed with water, cut into pieces, and soaked in distilled water over night. The mucilaginous extract was filtered through muslin cloth. It was precipitated from the extract by addition of alcohol. The precipitate was washed with acetone 2–3 times and finally dried by keeping in oven at 40°C for 24 hours. It is easily soluble in cold-water.

Acrylamide, Ceric ammonium nitrate, hydroquinone, buffer tablets, and nitric acid (S.D.Fine-Chem Ltd.) were used as received. The dyes direct fast scarlet (DFS), direct fast yellow (DFY), golden yellow (GY), and nyanthrene yellow (NY) were of commercial grade and used without further purification.

Preparation of Ku-g-PAM Copolymer

Ku-g-PAM was synthesized by grafting acrylamide (AM) onto purified Kundoor mucilage by radical polymerization method in aqueous system

using ceric ion/nitric acid, redox initiator. The following procedure has been adopted in carrying out the reactions. One gram of Kundoor mucilage was dissolved in distilled water (200 mL) in an Erlenmeyer flask. The flask was then sealed with a septum stopper and nitrogen gas was flushed into the solution through hypodermic needle for 20 minutes. Then 0.2 moles of AM solution prepared in 100 ml distilled water was added into the solution through the stopper by hypodermic syringe with constant stirring using magnetic stirrer. The solution was stirred for 30 minutes while being bubbled with nitrogen. Ceric ion solution (0.05×10^{-3} moles dissolved in 1 N HNO_3 solution) was injected through the stopper by hypodermic syringe. The nitrogen flushing was continued for another 20 minutes; then the needles were taken out, and the flask was further sealed with teflon tape. The reaction temperature was maintained at 30°C by immersing the flask in constant temperature bath. The reaction mixture was stirred occasionally; the reaction was continued for 4 hours and then terminated by injecting 0.5 mL of saturated aqueous hydroquinone solution.

The reaction product was washed with ethanol and then dried. Ku-g-PAM copolymer was finally obtained by drying in a vacuum oven at 40°C for 24 h. The percent grafting (PG) was calculated by the standard equation (15). The structure of Kundoor mucilage and Ku-g-PAM was confirmed by Fourier Transform (FT) IR spectrum (Brucker Vector 22 spectrophotometer).

Flocculation Experiments

Jar test is the most widely used method for evaluating and optimizing the flocculation/coagulation processes (16). This study consists of batch experiments involving rapid mixing, slow mixing and sedimentation. The apparatus allowed six beakers to be agitated simultaneously. 300 ml of flocculant-dye solutions were agitated in a flocculator at 100 rpm for one minute and then 30 rpm was quickly established for ten minutes. After slow mixing, the beakers were carefully removed from the flocculator and were placed in a safe place for sedimentation phase to take place. The duration of sedimentation was kept constant at 10 minutes. The different dye concentrations chosen for jar experiments were in the range of 1 to 15 mg/L. In each case, 300 ml sample was taken with the optimum mucilage dose. At definite intervals, the supernatant solution was taken out, decanted, centrifuged and analyzed spectrophotometrically. Several contact time experiments were undertaken to assess the effect of system variables.

The known concentrations of dyes solutions were prepared in order to get optimal time and flocculant dose for the treatment of wastewater. The concentrations of dyes were analyzed using Perkin Elmer, Lambda 40, UV-Vis Spectrophotometer at wavelengths, so as to obtain maximum absorbance. All tests were done at room temperature to eliminate any temperature effects. The pH values for the dye-flocculant solutions were measured by

Microprocessor pH meter CP931. The percent dye removal was calculated from initial (C_o) and final/equilibrium (C_e) concentrations of test solutions as follows (17)

$$\% \text{ Dye Removal} = \frac{C_o - C_e}{C_o} \times 100 \cdots\cdots \text{equation} \quad (1)$$

Statistical Analysis

A two-way analysis of variance (ANOVA) was applied for determining the statistical significance of the percent removal of the dyes with different flocculants using Matlab software, The Mathwork, Inc., USA. Triplicates were done in each case and the experimental design was randomized design in a controlled experimental setup.

RESULT AND DISCUSSION

Characterization

IR spectrum of pure mucilage has peaks at 3311 cm^{-1} of –OH, at 2924 cm^{-1} of -CH_2 stretching, at 1623 cm^{-1} of enolic form, at 1146–1100 cm^{-1} of –C-O stretching. IR spectrum of Ku-g-PAM (PG = 68.60) was different than that of pure mucilage by showing additional peaks at 1670 cm^{-1} of–C═O of amide, at 1543 cm^{-1} of–NH bending, at 1383 cm^{-1} of –CN stretching, at 1237.67 cm^{-1} of –C-C-N asymmetric, and a broad out of plane–NH band between 800–600 cm^{-1}. Moreover, the broadening and shifting of band towards higher wave number from 3311 cm^{-1} in pure mucilage to 3419 cm^{-1} in Ku-g-PAM is also expected due to the overlapping of –NH of amide and –OH of mucilage.

Flocculation Studies

Effect of Flocculant Dose

Table 1 contains the data showing the effect of variation in the amount of flocculant dose i.e., Kundoor mucilage and Ku-g-PAM on the percent removal of the direct dyes, DFS and DFY and vat dyes, GY and NY. It was observed that within the range studied, the percent removal significantly increased with an increase in flocculant dose up to 10 mg/L in case of Kundoor mucilage whereas 5 mg/L dose of Ku-g-PAM was optimum dose for both the dyes. The increase in the percent removal with an increase in the amount of flocculant might be attributed to the availability of increased surface area or active sites for the adsorption. An increase in flocculant concentration beyond

Table 1. Percent removal of dyes on varying the flocculant concentration

		Percent removal of direct dyes using									
		Ku mucilage (mg/L)					Ku-g-PAM (mg/L)				
S. No.	Dyes	1	5	10	20	50	1	5	10	15	20
1	DFS	17.56	16.27[a]	11.34[a]	9.62[a]	8.10[a]	21.12	42.89[b]	38.6[b]	20.3[b]	17.2[b]
2	DFY	17.35	26.68[b]	34.69[b]	19.8[b]	11.6 [b]	26.72	48.1[b]	36.8[b]	29.5[b]	19.7[b]
3	GY	12.83	27.62[b]	37.8[b]	21.0 [b]	—	36.65	52.2[b]	48.9[a]	31.07 [b]	14.9[b]
4	NY	11.65	24.2[b]	34.5 [b]	23.8[b]	—	27.34	48.96[b]	38.6 [b]	25.40[b]	12.6[b]

P: [a] < 0.01 and [b] < 0.001.

optimum dose, however, showed the decrease in percent removal of dyes. In the present experimental conditions, it is very likely that the polymer bridging plays a large part in the flocculation process and the higher the dosage of polymer; the more likely is aggregation between colliding particles. This trend (increasing and then decreasing trend) in % removal is because of the fact that the optimum amount of polymer in the suspension causes larger amount of dye particle to aggregate and settle. However, an over optimum amount of polymer in dye solution would cause the aggregated particle to redisperse and would also disturb particle settling (18). This behavior could also be explained on the basis of much increase in the repulsive energy between the polymer and dye solution, which causes hindrance in floc formation.

Effect of Dye Concentration

The effect of variation of dye concentration on the percent removal of dyes by Kundoor mucilage and Ku-g-PAM is shown in Table 2. It is apparent that the percent removal of the dyes, DFS and DFY, decreased with an increase of dye concentrations from 1–15 mg/L. In case of percent removal of direct dyes with pure mucilage, a significant change in decreasing trend was observed on varying the dye concentration from 1–10 mg/L, whereas with Ku-g-PAM, the results obtained were highly significant up to 15 mg/L concentration of dyes. With Ku as flocculant, the percent removal of vat dyes, GY and NY, increased initially with increase in dye concentration from 1–5 mg/L. Further increase in dye concentration decreased the percent removal. Whereas with Ku-g- PAM, the maximum percent removal was observed for vat dyes, GY and NY, at initial concentration of 1 mg/L. Further increase in dye concentration showed the decreasing trend (Table 3). The reason for the above observation may be attributed to the larger increase in the denominator (C_o) value in comparison to that of the (C_o-C_e) value in equation (1). The explanation for this observation is also based on a particle-polymer-particle complex formation in which the polymer serves as a bridge. To be effective in destabilization, a polymer molecule must contain chemical groups, which can interact with sites on the surface of the colloidal particle (19). When a polymer molecule comes into contact with a colloidal particle, some of these groups adsorb at the particle surface, leaving the remainder of the molecule extending out into the solution. If a second particle with some vacant adsorption sites contacts these extended segments, attachment can occur. A particle-polymer-particle complex is thus formed in which the polymer serves as a bridge. If a second particle is not available, in time the extended segments may eventually adsorb on other sites on the original particle, so that the polymer is no longer capable of serving as a bridge. There is direct stoichiometric relationship between optimum polymer dosage and colloid concentration, and restabilization can occur due to any change in this stoichiometric ratio.

Table 2. Percent removal of direct dyes on varying the dye concentration

S. No	Dye concentration (mg/L)	Percent removal within 0 min.	30 min.	60 min.	90 min.	120 min.	180 min.
1. Direct fast scarlet with Ku as flocculant							
i	1	1.59	15.77^{b}	27.35^{b}	26.89^{a}	26.51^{a}	26.49^{c}
ii	2	1.42	7.66^{b}	16.27^{b}	16.09^{a}	15.31^{a}	15.3^{c}
iii	5	1.21	9.53^{b}	11.34^{b}	11.31^{c}	11.30^{c}	11.3^{c}
iv	10	0	6.42^{b}	9.62^{b}	9.38^{a}	8.97^{a}	8.92^{c}
v	15	0	4.13^{b}	8.10^{a}	8.07^{c}	7.61^{a}	7.60^{c}
2. Direct fast yellow with Ku as flocculant							
i	1	7.21	12.39^{b}	34.69^{b}	33.74^{a}	32.61^{a}	32.60^{c}
ii	2	10.01	19.87^{b}	32.21^{b}	32.00^{a}	31.18^{a}	31.18
iii	5	5.16	18.89^{b}	23.42^{b}	22.99^{a}	22.97^{c}	22.95
iv	10	4.25	13.45^{b}	16.91^{b}	16.90	15.24^{c}	15.23
v	15	5.29	9.08^{b}	12.32^{b}	11.47^{c}	11.62	11.60
3. Direct fast scarlet with Ku-g-PAM as flocculant							
i	1	18.53	42.89^{b}	36.81^{b}	35.43^{a}	31.60^{b}	31.60
ii	2	9.61	38.52^{b}	38.33	38.17^{a}	38.16	38.16
iii	5	7.23	26.19^{b}	25.68^{a}	22.43^{b}	21.56^{a}	21.49
iv	10	7.61	21.48^{b}	18.43^{b}	15.26^{b}	14.87^{b}	14.86
v	15	9.20	15.25^{b}	14.21^{a}	11.68^{b}	11.27	11.09
4. Direct fast yellow with Ku-g-PAM as flocculant							
i	1	29.82	48.10^{b}	45.32^{b}	44.21^{a}	39.73^{a}	39.73
ii	2	17.28	39.63^{b}	39.19^{a}	39.06^{a}	38.51^{b}	38.47
iii	5	25.36	32.51^{b}	28.78^{b}	25.42^{a}	24.96^{a}	24.95
iv	10	9.25	25.38^{b}	24.36^{b}	21.72^{b}	18.45^{b}	18.40
v	15	9.10	11.67^{b}	10.47^{b}	8.32^{b}	6.54^{b}	6.50

P: $^{a} < 0.01$, $^{b} < 0.001$ and $^{c} < 0.05$.

Effect of Contact Time

Tables 2 and 3 show the data for the effect of % removal of the dyes with contact time with Ku and Ku-g-PAM. The removal of the dye was rapid in the initial stages of contact time finally became constant. The treatment time affected the percent removal of direct and vat dyes significantly up to one and two hours, respectively; after that no significant change was observed with pure mucilage as flocculant. The maximum removal, with pure mucilage, was obtained in one hour for both the direct dyes and two hours for both the vat dyes with pure mucilage as flocculant. This shows that equilibrium is attained in three hours, which is irrespective of the concentration of dye solution. Whereas, with Ku-g-PAM, the maximum removal was

Table 3. Percent removal of vat dyes on varying the dye concentration

S. No	Dye concentration (mg/L)	Percent removal within 0 min.	30 min.	60 min.	120 min.	180 min.	240 min.
1. Golden yellow with Ku as flocculant							
i	1	0	15.21	28.86^{b}	37.80^{b}	36.82^{a}	36.78^{c}
ii	2	0	19.77	25.51^{b}	46.51^{b}	44.42^{b}	44.39^{c}
iii	5	0	28.85	46.62^{b}	52.40^{b}	51.63^{b}	51.54^{c}
iv	10	0	21.06	38.95^{b}	44.10^{b}	42.19^{b}	42.11^{c}
v	15	0	19.30	21.73^{a}	26.90^{b}	25.86^{b}	25.81
2. Nyanthrene yellow with Ku as flocculant							
i	1	0	27.89	30.61^{b}	34.5^{b}	33.96^{a}	23.85^{b}
ii	2	0	25.58	36.68^{b}	42.6	42.51^{b}	42.48^{c}
iii	5	0	36.62	42.0^{b}	57.3^{b}	55.48^{b}	55.36^{c}
iv	10	0	28.95	37.0^{b}	40.07^{b}	38.67^{b}	38.63
v	15	0	11.71	15.35^{b}	39.8^{b}	37.75^{b}	37.71
3. Golden yellow with Ku-g-PAM as flocculant							
i	1	26.61	48.91^{b}	46.65^{b}	45.62^{a}	45.17^{c}	45.07
ii	2	35.55	48.95^{b}	47.92^{b}	47.78^{c}	46.67^{a}	46.51
iii	5	19.67	36.81^{b}	35.56^{b}	34.98^{b}	34.17^{a}	34.08
iv	10	18.43	34.25^{b}	33.61^{b}	32.65^{b}	32.15^{c}	32.04^{c}
v	15	10.62	11.98^{b}	10.53^{b}	9.50^{b}	8.62^{b}	8.62
4. Nyanthrene yellow with Ku-g-PAM as flocculant							
i	1	24.43	38.65^{b}	36.61^{b}	35.54^{b}	35.29^{a}	35.26
ii	2	32.62	46.53^{b}	43.61^{b}	44.10^{b}	43.96^{a}	43.91
iii	5	15.35	32.85^{b}	32.05^{c}	31.92^{b}	31.07^{a}	31.08
iv	10	16.97	30.43^{b}	29.15^{b}	28.85^{b}	28.15^{a}	28.07
v	15	8.73	12.67^{b}	12.21^{a}	11.62^{b}	10.15^{a}	10.15

P: $^{a} < 0.01$, $^{b} < 0.001$ and $^{c} < 0.05$.

obtained within 30 minutes for both the direct and vat dyes and with gradual decrease, % removal became constant in about two hours time.

Effect of pH

Data related for removal of dyes as a function of pH is given in Table 4. It is apparent from the figures that the best % removal was seen at acidic pH in both the cases. The pH values seem to affect the percent removal of both the dyes significantly with both the flocculants.

It is well known that substances adsorb poorly when they are ionized. Usually, when the pH is such that an adsorbable compound exists in ionized form, adjacent molecules of the adsorbed species on the adsorbate surface will repel each other to a significant degree, because they carry the same

Table 4. Percent removal of dyes on varying the pH

S. No.	Flocculant	Dye	pH			
			4	6	7	9.2
1	Ku mucilage	Direct fast scarlet	34.46	28.52^{b}	19.27^{b}	7.35^{b}
2	Ku mucilage	Direct fast yellow	38.87	35.69^{a}	27.82^{b}	15.41^{b}
3	Ku mucilage	Golden yellow	68.00	59.00^{b}	36.00^{b}	32.00^{a}
4	Ku mucilage	Nyanthrene yellow	65.50	52.90^{b}	43.60^{b}	39.80^{b}
5	Ku-g-PAM	Direct fast scarlet	52.25	48.86^{a}	39.38^{b}	15.97^{b}
6	Ku-g-PAM	Direct fast yellow	55.53	49.62^{b}	38.56^{b}	17.21^{b}
7	Ku-g-PAM	Golden yellow	67.71	54.56^{b}	39.20^{b}	18.65^{b}
8	Ku-g-PAM	Nyanthrene yellow	64.42	52.98^{b}	36.67^{b}	15.42^{b}

P: $^{a} < 0.01$ and $^{b} < 0.001$.

electrical charge (forces of repulsion/attraction between actual ions are strong, as compared with weak forces such as Van der Waals forces). Thus, the adsorbing species cannot pack together very densely on the surface, and the equilibrium amount of adsorbed solute is only modest. In contrast, when the adsorbing species is in non-ionized form, no electrical repulsion exists, and thus the packing density on the surface can be much higher.

The increase in percent removal of GY and NY at acidic pH was attributed to this conversion of solubilized form into the non-solubilized form, which has the non-ionized structure (20). The maximum removal for DFS and DFY was observed at acidic pH as these dyes give anions in an aqueous solution and in the presence of H^{+} ions at acidic pH, these anions get neutralized. Hence, the maximum removal was expected at acidic pH (21) with pure mucilage. Whereas with Ku-g-PAM the higher percent removal observed at acidic pH might be attributed to the fact that in acidic conditions, the protonation of the amide groups occurred. The dyes, DFS and DFY, give anions when dissolved in water and these colored anions are absorbed and retained by protonated polyacrylamide chains forming an electrovalent bond between the dye and the copolymer.

CONCLUSIONS

From the present set of experiments, Flocculation, using Kundoor mucilage and its acrylamide grafted copolymer for color removal, was shown to be a simple and efficient treatment from an economic and technical point of view. The flocculation efficiency of these flocculants was tested for the removal of direct and vat dyes. It was concluded that the removal of direct dyes was faster as compared with that of vat dyes when Ku was used as flocculant, whereas, with Ku-g-PAM as flocculant, the optimum time for

removal of both types of dyes was 30 minutes. The optimum mucilage dose was 10 mg/L and 5 mg/L for Ku and Ku-g-PAM, respectively. The grafting of acrylamide chains improved the flocculation efficiency of the mucilage by reducing the flocculant dose and treatment time by half. The effective pH observed for dyes removal was acidic with both pure mucilage and its grafted copolymer. The possibility of industrial application of these biodegradable flocculants for removal of various types of dyes may be explored to evaluate their full potential.

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