Studies on Usage of Acrylamide Copolymers for Textile Effluent Treatment

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ABSTRACT: The copolymers of acrylamide–maleic anhydride and acrylamide–diallyl dimethyl ammonium chloride of different ionicities were used for the removal of color, solids, etc., from effluent generated from textile industry. The studies have been carried out to remove color generated in the effluent. To estimate dye content, we prepared standard curve using UV-vis spectrophotometer at different dye concentration following the Beer Lambert's Law. The copolymers of acrylamide–maleic anhydride and acrylamide–diallyl dimethyl ammonium chloride of known concentration were dosed in the known concentration of the dye. These polymers made flocks by adsorption with dyes and these colored flocks were filtered out. The intensity of the color of water went down on increasing the concentration of the polymers. The color of the textile effluent was removed to the extent of 97% using above mentioned polymers. © 2013 Wiley Periodicals, Inc. J. Appl. Polym. Sci. 129: 2536–2543, 2013

INTRODUCTION

The textile industry consumes considerable amount of water in the manufacturing process. The water is primarily used in dyeing and finishing operations in which the cloths are dyed and processed to finished products. In a typical dyeing and finishing mill, about 100 L of water are consumed on average for every ton of cloth processed.[1] The water used in the dyeing and finishing processes eventually ends up as waste water, which needs to be treated before final discharge.[2]

Normal textile dyeing and finishing operations are such that the dyestuffs used in a mill can vary from day to day, sometimes even many times a day, mainly because of the batch-wise nature of the dyeing process. Frequent changes of dyestuff and chemicals used in the dyeing process cause considerable variation in the wastewater characteristics,[3,4] particularly the pH, color, and wastewater BOD and COD values. A large pH swing is especially troublesome because the pH tolerance of conventional biological and chemical treatment systems is very limited. Hence, without proper pH adjustment, normal operation of the treatment processes is essentially impossible.

Strong color is another important component of the textile waste,[5] which is very difficult to deal with. Textile industries have shown a significant increase in use of synthetic complex and organic dyes as the coloring material. The dyes include a broad spectrum of different chemical structure based on hetero-cyclic groups such as aromatic amine (C_6H_5—NH_2), phenyl (C_6H_5—) and naphthyl ($C_{10}H_8$—). The large numbers of dyes are azo compounds (—N=N—), which are linked by an azo bridge. Color in waste water is an indicator of water pollution.[6] The waste water characteristics from a dye house are highly variable from day to day and even hour to hour, depending on the type of dye, the type of fabric, and the concentration of the fixing agents added. Treatment of such waste water is therefore essential but difficult. The discharge of dye from house waste water into the environment is aesthetically displeasing, impedes light penetration, damages the quality of the receiving streams, and may be toxic to treatment processes, to food chain organisms, and to aquatic life. The degradation of molecules of dyes in the environment by microorganism is likely to be slow, which means that it is possible for high levels of dye to persist and potentially accumulate.[7]

The combination of strong color and highly dissolved solid contents results in high turbidity of waste effluent. The dyestuffs are high-structured polymers and are very difficult to decompose biologically. Hence, there is relatively little change of these dyestuff molecules in an activated sludge process.[8] For color removal, decolorization using activated charcoal, polyalkylaluminum and their mixtures,[9] hydrogen peroxide, sodium hypochloride, and other chemical agents have been widely used in textile industries,[10–12] but none of them have been found satisfactory in complete removal of color from textile effluent.[13]

Traditional activated sludge and chemical methods (such as chemical coagulation and decolonization) have been widely used for dealing with the textile waste waters.[14] This study represents the attempts to utilize the process of chemical coagulation to treat waste water effluent from secondary waste water treatment plant of large dye in and finishing mill.[15–17] The waste water quality is enhanced to a level that meets the reuse standard of the industry.

EXPERIMENTAL

Chemicals and Materials

Acrylamide, maleic anhydride, potassium persulfate (KPS), sodium bisulfite (SBS), and sodium hydroxide were obtained from E-Merck, Fluka/Aldrich at Vadodara, Gujarat, India. All were of AR grade.

Synthesis of Acrylamide–Maleic Anhydride Copolymer.

Procedure. The polymerization was carried out in four-necked round bottom flask equipped with water condenser, thermometer pocket, mechanical stirrer, and nitrogen inlet. Oxygen hinders the propagation rate of acrylamide monomer and comonomer in polymerization, and one does not get higher molecular weight polymer, which is the requirement for color removal of textile effluent.[18,19]

Hence, the purging of nitrogen was made continued throughout the reaction to exclude O_2. An aqueous solution consisting of acrylamide and maleic anhydride was prepared. Similarly, KPS and SBS were dissolved in water. The monomer, comonomer, and initiator solution were added to round bottom flask simultaneously. KPS and SBS are used as initiator. The temperature of reaction was maintained at 70°C and the reaction was continued for 2 h.

Synthesis of Acrylamide–Diallyl Dimethylammonium Chloride Copolymer.

Procedure. An aqueous solution of acrylamide and diallyl dimethylammonium chloride containing a known amount of NaCl was prepared. Similarly, ammonium persulfate and potassium hydrogen sulfate in water were also prepared. The monomer, comonomer, and initiator solutions were prepared and added to the round bottom flask under nitrogen atmosphere maintaining the flask temperature at 4 ± 2°C using ice bath. All the solutions were fed into the round bottom flask and the reaction was continued at 40°C for 31/2 h. The resulting mixture was viscous solution which was diluted adequately with water to maintain 25% solid content. The solid polymers were prepared by precipitation using methanol.

Preparation of Calibration Curves.

The standard curves were prepared by plotting absorbance versus concentration of dyes as presented in Figure 1. It was observed that a straight line passing through the origin was obtained upto maximum dye color concentration in the range of 20–25 ppm, which follows Beer–Lambert's Law. This standard curve was used to estimate the dye in effluent.

CHARACTERIZATION

These copolymers were characterized using different analytical techniques.
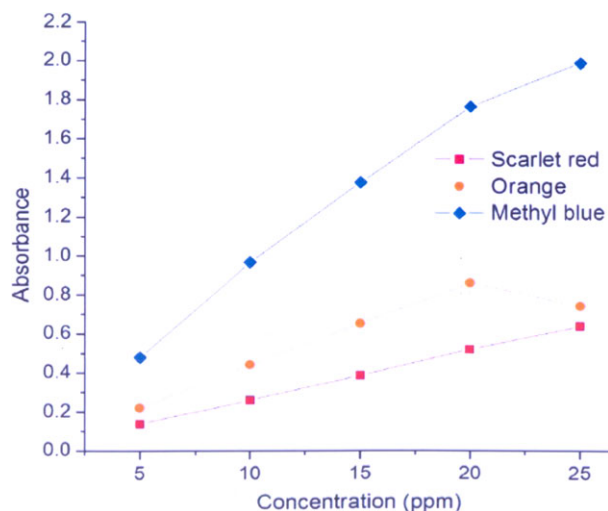
Figure 1. Plot of absorbance versus concentration of dyes. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

Viscosity Measurement

The viscosity of polymers was determined using Ubbelohde suspension level capillary viscometer AVB 400 SCHOTT-GERATE, Germany. The specification of capillary type No. 53110, capillary No.1 diameter of 0.63 mm Constant K (richwere) 0.01. The standard conditions of temperature at 25°C and atmospheric pressure were maintained during the measurement.[20,21]

(2) Infrared Spectroscopy

The presence of functional groups was determined using the IR spectroscopy. The IR of polymer samples was recorded on Perkin Elmer IR Spectrometer in the range of 500–4000 cm^{-1}. The polymer samples were dissolved in a mixture of Tetrahydrofuran (THF) and water. The films were casted and dried under vacuum. Characteristic peaks were analyzed and the spectrum was recorded.[22–25]

UV Measurement

The UV measurement was carried out using Simadzu 160-A UV spectrophotometer. A standard curve of individual dyes was made by determining maximum absorbance at particular wavelength. The absorbance of dyes, namely, scarlet red, orange, and methylene blue at different concentrations lower than the concentration that gives maximum absorbance were determined.[23–25]

Gel Permeation Chromatography

Number average molecular weight was determined by GPC of WATERS using water as an eluent. GPC was standardized with standard sample of polyacrylic acid having number average molecular weight of 40,000.

NMR Spectroscopy Analysis

The ^{13}C NMR spectra were recorded with a Varian 200 spectroscopy system operating at 50.30 MHz. Samples of about 40 mg were dissolved in $CDCl_3$ (Aldrich Chemical Co.) in 5 mm-o.d. sample tubes.

RESULTS AND DISCUSSION

Polyacrylamide is popular flocculating agent for waste water purification. But, homo polyacrylamide does not absorb color

Table I. Synthesis of Acrylamide-Maleic Anhydride Copolymer

| Sr. No. | Monomer | | Resulting reaction solution |
	Acrylamide (mol)	Maleic anhydride (mol)	
1	0.134	0.005	Viscous liquid formed
2	0.127	0.01	Viscous liquid formed
3	0.113	0.02	Viscous liquid formed
4	0.099	0.03	Viscous liquid formed

Temp – 70ºC, Time–2 hr, p^H–7.0
Concentration of initiators: KPS = 0.05%, SBS: 0.05%
Water (solvent) = 100 ml

or dye. The copolymer of acrylamide having cationic groups can attract the dye molecules and can absorb dyes from its aqueous solutions and can be utilized for the color removal from textile effluents.

Synthesis of Acrylamide–Maleic Anhydride Copolymer
Maleic anhydride on dilution with water gives bifunctional carboxylic (—COOH) groups. The polymers based on maleic anhydride are more polar in nature. They have wide industrial applications.[26] The copolymers of acrylamide—maleic anhydride were prepared by varying maleic anhydride content from 0.5 to 3%. The temperature, time, catalyst concentration, pH, and water content were kept constant.[27–29] The results are tabulated in Table I. The intrinsic viscosities of copolymers are presented in Table II.

Synthesis of Acrylamide–Diallyl Dimethylammonium Chloride Copolymer
Cationic polymerization has wide applications in developing products of industrial importance.[30–33] Copolymer of acrylamide and diallyl dimethylammonium chloride of different cationicity was synthesized varying the concentration of diallyl dimethylammonium chloride from 5 to 20 %. Polymerization parameter such as temperature, catalyst concentration, time, etc., was kept constant except the monomer concentration as shown in Table III.

These copolymers were prepared in the form of viscous solution and were used for testing the textile effluents for color removal. The intrinsic viscosity of these samples was determined and is presented in Table IV. It has been observed from the results that there is not much variation in viscosity values. It indicates that

Table III. Synthesis of Acrylamide–Diallyl Dimethylammonium Chloride (DADMAC) Copolymer

| Sr. No. | Monomer | | Resulting reaction solution |
	Acrylamide (mol)	DADMAC (mol)	
1	0.134	0.003	Viscous solution formed
2	0.127	0.006	Viscous solution formed
3	0.113	0.012	Viscous solution formed

Temperature 70°C, Time-3.5 hrs, p^H – 7
Concentration of initiators: APS = 0.05%, KHS: 0.05%
Water (solvent) = 100 ml

molecular weight (Mw) value will not be in wide range, which can be observed with as determined GPC values.

IR spectroscopy was used to identify the functional groups such as carbonyl, amide, and carboxylic groups to confirm the formation of copolymer as shown by IR spectra (Figure 2). For all the copolymers, a similar absorption spectrum was observed confirming thereby the presence of carbonyl groups (1170 cm^{-1}) related to C=O or C—O bonds resulted from presence of comonomers, amide groups (3416.57 cm^{-1}), CH$_2$ groups (2959.12 cm^{-1}), C—C bonds (1036.46 cm^{-1}), C—H vinyl in-plane bend (1414.36 cm^{-1}), —COO stretching (1566, 2355.80, and 2329.28 cm^{-1}), and C—H stretching (2959.12 cm^{-1}).

^{13}C NMR spectra were recorded for the acrylamide–maleic anhydride copolymer (composition 4 of Table I) and acrylamide–diallyldimethyl ammonium chloride copolymer (composition 3 of Table III) as shown in the Figures 3 and 4, respectively.

As per the NMR spectra shown in Figure 3 for acrylamide–maleic anhydride copolymer, the peak at 178 indicates the presence of acrylamide (>CONH$_2$) group and peak at 185 shows the presence of maleic anhydride group. The peak height at 178 and 185 supports the composition of copolymer. Similarly, in the NMR spectra in Figure 4, copolymers of acrylamide and diallyldimethyl ammonium chloride, the peak at 178 indicates the presence of acrylamide (>CONH$_2$) group and peak at 58 indicates the presence of diallydimethyl ammonium chloride group. The peak height supports the composition of copolymer.

Studies on Textile Effluent Treatment
Present studies have been carried out to treat the textile effluent by using acrylamide–maleic anhydride copolymer and acrylamide–diallyl dimethyl ammonium chloride maleic anhydride terpolymer

Table II. Intrinsic Viscosity of Acrylamide–Maleic Anhydride Copolymer

| Sr. No. | Monomer | | $[\eta]$ (dL/g) | M_n | M_w | M_w/M_n |
	Acrylamide (mol)	Maleic Anhydride (mol)				
1	0.134	0.005	5.35	9.21×10^4	1.57×10^5	1.7
2	0.127	0.01	4.75	7.88×10^4	1.33×10^5	1.69
3	0.113	0.02	4.80	7.99×10^4	1.34×10^5	1.68

Table IV. Intrinsic Viscosity of Acrylamide–Diallyl Dimethyl Ammonium Chloride (DADMAC) Copolymer

Sr. No.	Monomer		[η] (dL/g)	M_n	M_w	M_w/M_n
	Acrylamide (mol)	DADMAC (mol)				
1	0.134	0.003	5.65	8.9×10^4	1.59×10^5	1.79
2	0.127	0.006	6.25	8.6×10^4	1.5×10^5	1.74
3	0.113	0.012	6.45	8.5×10^4	1.53×10^5	1.80

with an objective of removal of color generated by dyes and the reduction of the suspended material. A standard curve of individual dyes was made by determining maximum absorbance at particular wavelength. It was observed that 50 ppm solution of scarlet red dye gave maximum absorbance at 514 nm. Similarly, 25 ppm solution of orange dye gave maximum absorbance at 490 nm. A 10 ppm solution of methyl blue solution absorbed maximally at 671 nm. The mixed samples of 7 ppm concentration absorbed maximally at 674 nm and 506 nm. The absorbance of dyes, nanely, scarlet red, orange, and methylene blue at different concentrations lower than the concentration, which gives maximum absorbance, was determined.

Studies on Color Removal Using Acrylamide–Maleic Anhydride Copolymer

The studies to remove the color of dyes such as scarlet red, orange, methylene blue, and their mixture were carried out using the copolymers of acrylamide–maleic anhydride and acrylamide–diallyl dimethyl ammonium chloride (DADMAC). A 1% solution of above dyes was made, and it was further diluted to make solution of 20 ppm concentration. The pH of the colored solution was acidic in nature. The quantity of the color absorbed by the copolymer was estimated using UV-vis spectrophotometer and the standard curves made from above dyes.

The color removal ability of all the copolymer compositions of acrylamide–maleic anhydride was measured with scarlet red dye with 20 ppm concentration keeping copolymer concentration at 1%.

Table V. Color Removal of Scarlet Red Dye by Acrylamide–Maleic Anhydride Copolymer

Sr. No.	Copolymer compositions	Dye concentration (ppm)	Color removal (%)
1	AAm-MA (95 : 5)	18.0	10
2	AAm-MA (90 : 10)	17.0	15
3	AAm-MA (80 : 20)	13.0	35
4	AAm-MA (70 : 30)	13.1	34.5

AAm – Acrylamide, MA – Maleic anhydride,
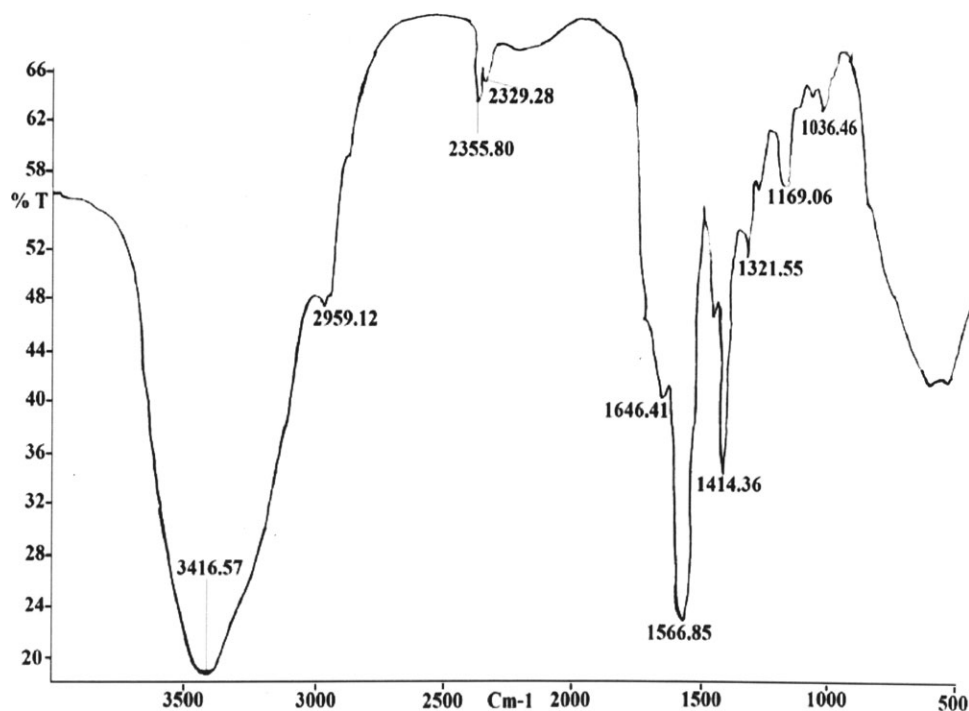
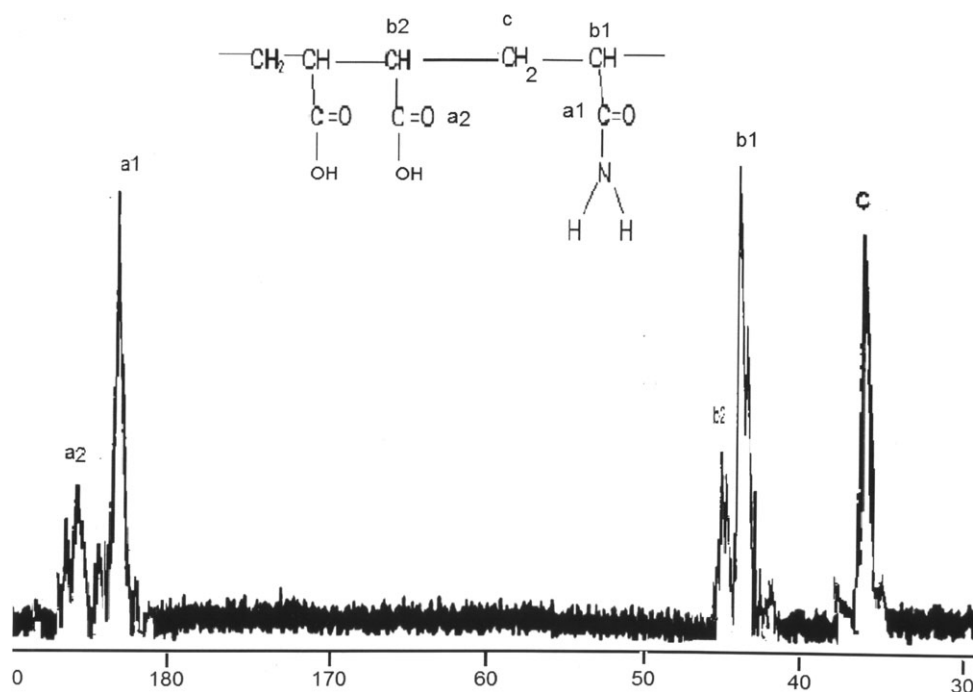

Figure 2. IR spectra of typical copolymer.

Figure 3. ^{13}C NMR spectra for the acrylamide–maleic anhydride copolymer.

It appears that the maleic anhydride place a major role in color removal. Beyond 20% maleic anhydride concentration in acrylamide–maleic anhydride copolymer, the color removal levels off hence copolymer composition of 80 : 20 was found to be optimum and studied further. The copolymer concentration dosing was varied from 1 to 2.5% in colored solution. The results obtained are presented in Table VI–VIII.

It is evident from the table nos. VI and VII that about 85% of the color due to scarlet red and orange dye is removed using 2.5% of the acrylamide- maleic anhydride copolymers. In case of methylene blue, 97% color is removed and water becomes mostly colorless. The majority of the color is removed at 2% copolymer concentration (Figure 5).
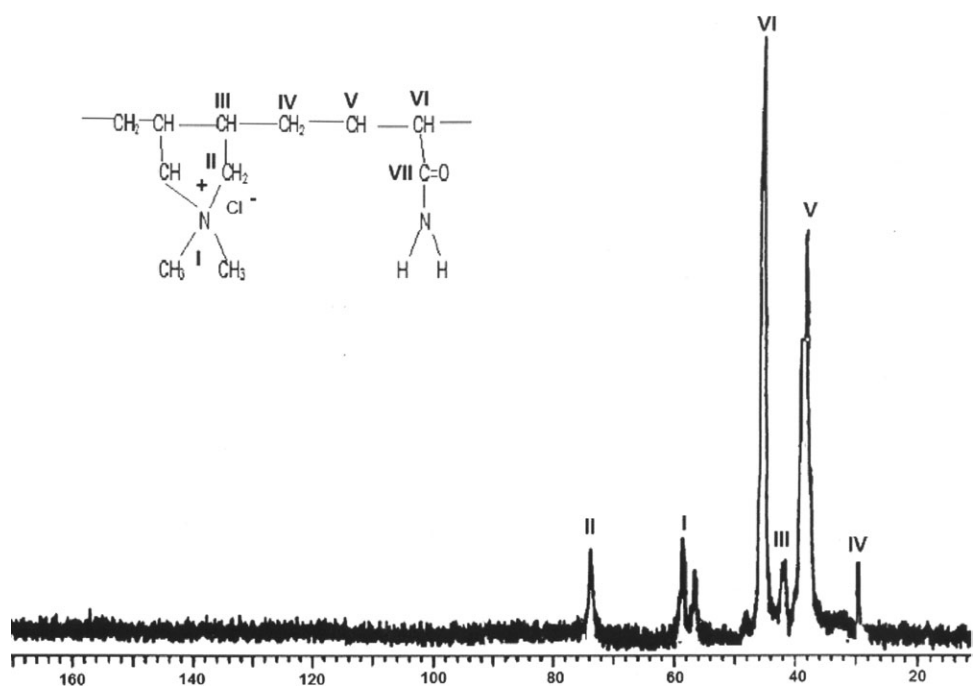

Figure 4. ^{13}C NMR spectra for acrylamide diallydimethyl ammonium chloride copolymer.

Table VI. Color Removal of Scarlet Red Dye by Acrylamide–Maleic Anhydride Copolymer (80:20)

Sr. No.	Copolymer (%)	Dye concentration (ppm)	Color removal (%)
1.	0.00	20.0	00.0
2.	1.0	13.0	35.0
3.	1.5	10.3	48.5
4.	2.0	3.2	84.7
5.	2.5	3.2	84.7

Studies on Color Removal Using DADMAC Copolymer

The color removal ability of all the copolymer compositions of DADMAC were measured with scarlet red dye with 20 ppm concentration keeping copolymer concentration at 1%. The results are shown in Table IX.

Similar studies as above were carried for the removal of color dyes scarlet red, orange, and methylene blue. The color is because of mixture of these dyes and effluent collected from textile industries. The results are given in Tables X–XII.

The 80 : 20 composition of DADMAC was studied further.

It is evident from the results that most of the color in all the three dyes is removed at polymer concentration of 2.5% of copolymer per 1000 ml of 20 ppm of dye concentration. The results of color removal using DADMAC copolymer concentra-

Table VII. Color Removal of Orange Dye by Acrylamide–Maleic Anhydride Copolymer (80:20)

Sr. No.	Copolymer (%)	Dye concentration (ppm)	Color removal (%)
1.	0.00	20.0	00.0
2.	1.0	14.3	27.0
3.	1.5	11.2	44.5
4.	2.0	3.4	83.0
5.	2.5	2.9	85.5

Table VIII. Color Removal of Methylene Blue Dye by Acrylamide–Maleic Anhydride Copolymer (80:20)

Sr. No.	Copolymer (%)	Dye concentration (ppm)	Color removal (%)
1.	0.00	20.0	00
2.	1.0	4.4	78
3.	1.5	3.2	84
4.	2.0	1.20	97
5.	2.5	1.21	97

Concentration of dye = 20 ppm, Quantity of colored water for treatment =1 Lt
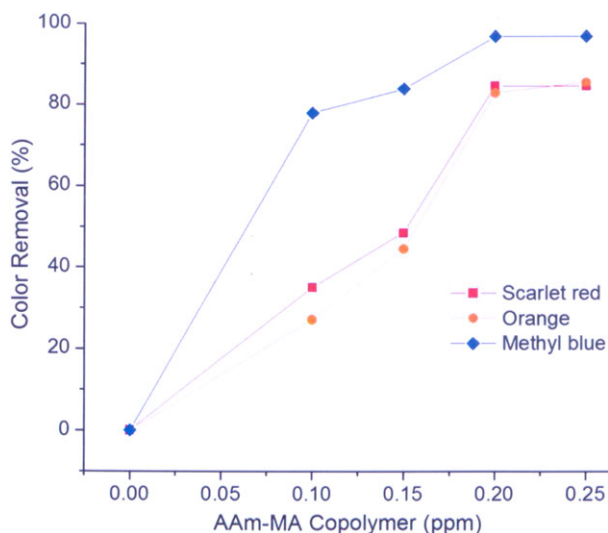
Figure 5. Color removal versus AAm-MA concentration. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

tions are plotted in Figure 6. The curves indicate that 80–90% of dye color is removed at 2% g of copolymers containing maleic anhydride and DADMAC. The results on color removal of textile effluent and mixture of dyes are given in Table XIII. The color of textile effluent was dark bluish and brown. Hence, it was difficult to estimate a particular dye component because it is a mixture of several unknown processing chemical such as pigments, dyes, lubricants, etc. However, the value was determined by comparison of garden color standards.

Table IX. Color Removal of Scarlet Red Dye by Acrylamide–Diallyl Dimethyl Ammonium Chloride

Sr. No.	Copolymer compositions	Dye concentration (ppm)	Color removal (%)
1	AAm- DADMAC (95 : 5)	8.9	55.5
2	AAm- DADMAC (90 : 10)	5.5	72.5
3	AAm- DADMAC (80 : 20)	3.8	81

Am – Acrylamide, DADMAC – diallyl dimethylammonium chloride

Table X. Color Removal of Scarlet Red Dye by Acrylamide–Diallyl Dimethyl Ammonium Chloride Copolymer (80:20)

Sr. No.	Copolymer (%)	Dye concentration (ppm)	Observed pH	Color removal (%)
1.	0.00	20.0	6.9	00.0
2.	1.0	3.8	6.8	81.0
3.	1.5	2.1	6.8	89.5
4.	2.0	1.2	6.60	94.0
5.	2.5	Not detectable		100.0

**Table XI.** Color Removal of Orange Dye by Acrylamide–Diallyl Dimethylammonium Chloride Copolymer (80:20)

Sr. No.	Copolymer (%)	Dye concentration (ppm)	Observed pH	Color removal (%)
1.	0.00	20.0	6.8	00.0
2.	1.0	2.0	6.7	90.0
3.	1.5	1.3	6.5	93.0
4.	2.0	1.0	6.5	95.0
5.	2.5	Not detectable		100.0

The most of the effluent available in textile industries have the pH in the range of 6–7. In view of this, we studied the color removal keeping the pH from 6.5 to 6.9. At lower pH, higher color removal ability of copolymers has been observed. At high pH, it is lesser. It may be because of different functionalities present in dyes.

Table XII. Color Removal of Methylene Blue Dye by Acrylamide–Diallyl Dimethyl Ammonium Chloride Copolymer (80:20)

Sr. No.	Copolymer (%)	Dye concentration (ppm)	Observed pH	Color removal (%)
1.	0.00	20.0	6.8	00.0
2.	1.0	3.0	6.7	85.0
3.	1.5	1.8	6.7	91.0
4.	2.0	0.5	6.5	97.0
5.	2.5	Not detectable		100.0

Concentration of dye = 20 ppm, Quantity of colored water for treatment =1 liter
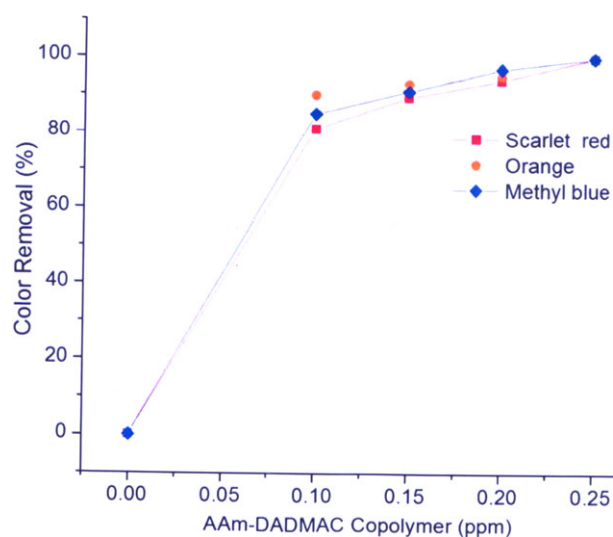
Figure 6. Color removal versus AAm-DADMAC concentration. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

Table XIII. Color Removal of Mixture Dyes (Scarlet Red, Orange, and Methylene Blue) by Copolymer

Sr. No.	Copolymer (g)	Color value of dye (mixed)	Color value of textile effluent
1.	0.00	4	6
2.	AAm-MA (80 : 20) (0.20 g)	2	3
3.	AAm-DADMAC (80 : 20) (0.25 g)	1	1
4.	AAm-MA-ADMAC (80 : 10 : 10) (0.25 g)	1	1

Concentration of dye = 7 ppm, Quantity of colored water for treatment =1 Lt, Color of textile effluent = Dark bluish brown, Appearance of textile effluent = Turbid.

CONCLUSION

The copolymers of acrylamide–maleic anhydride and acrylamide–dially dimethyl ammonium chloride were synthesized using potassium per sulfate and SBS initiators varying the monomer concentrations. The polymerization conditions were optimized to get the maximum conversion of monomer to copolymer. These copolymers were characterized using FTIR, NMR spectroscopy, and GPC for number and weight average molecular weight. Copolymers were found completely soluble in water. The FTIR and NMR analyses confirm the formation of the copolymer of acrylamide with maleic anhydride and dially dimethyl ammonium chloride. The GPC results confirm the formation of higher molecular weight of the copolymers.

These copolymers were used for studying the color removal of different dyes such as scarlet red, orange, and methylene blue dyes. It was observed that at lower pH, higher color content is removed using copolymers of acrylamide–maleic anhydride and DADMAC. At high pH, it is lesser. It may be because of different functionalities present in dyes. The 2% of the copolymers gives highest color removal from dye solution. The copolymers of acrylamide having 20% maleic anhydride and dially dimethyl ammonium chloride were most effective for color removal from dye solution. The copolymers of DADMAC are better than acrylamide–maleic anhydride copolymer in removing color from dye solution. The cationic nature of the comonomers enhances the attraction of the dye molecules toward the copolymers and thus enhance the removal of dye molecules from solution. These copolymers may find wide applications in removing dye color from textile effluents.

ACKNOWLEDGMENT

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