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Characterization and coagulation/flocculation treatment of coloured wastewater of institutional dyeing laboratory

Anilkumar Yadav[1,2]* & Sanjeev R. Shukla[2]

[1]Department of Textile Technology, Dr. B. R. Ambedkar National Institute of Technology Jalandhar, Punjab – 144008, India
[2]Department of *Fiber*s and Textile Processing Technology, Institute of Chemical Technology, Mumbai, Maharashtra - 400019, India
*E-mail: yadaval@nitj.ac.in

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This study focuses on the quantitative and qualitative characterization of coloured wastewater of an institutional dyeing laboratory and its treatment *via* coagulation/flocculation (CF), where the cumulative volume of wastewater is large and generally discharged without any treatment. Primarily woven fabric pieces of cotton, wool, silk, nylon, acrylic, and polyester are dyed using standard dyeing procedure maintaining the liquor ratio of 50. Total 23 dyes from the different class of dyes *viz.,* direct, reactive, acid, cationic, sulfur, vat, and disperse are used for dyeing of fabric samples. The generated colored wastewater is characterized for its volume, pH, total dissolved solids, turbidity, colour concentration, and biological and chemical oxygen demand. The qualitative characteristics of generated coloured wastewater influenced the preparation of two types of simulated coloured wastewaters which later subjected to CF using the coagulants (ferric chloride and a commercial polymeric coagulant). The pH of coagulation bath, coagulant dose and type influenced the color removal efficiency, where the polymeric coagulant performed better as compared to ferric chloride. Adaptable approaches and strategies have also comprehended in this study, which are adaptable to reduce the colored wastewater volume, thus leading to achieve the sustainability.

Keywords: Coagulation/flocculation, Colour removal, Dyeing, Ferric chloride, Polymeric coagulant

Introduction

Dyes, the complex organic molecules, depending upon their ionic nature are grouped into anionic (direct, acid and reactive), cationic (basic), and non-ionic (disperse) dyes, and based upon water solubility as water soluble (direct, reactive, metal complex (MC), acid, basic) and water insoluble (disperse, vat, and sulfur) dyes[1,2]. Dyes in large amounts are used in industries such as food, paper, plastic, leather, and textile[3,4]. Statistics reveals that the annual industrial production of synthetic colourants is nearly 8×10^5 tons, and approximately 10000 synthetic dyes and pigments are commercially accessible[5,6]. The market analysis report reveals that the global market size of dyes and pigments was accounted to USD 36.4 billion in 2021 and is expected to grow at CAGR of 5.2% from 2022-30[7]. In 2021, textile industries possessed the largest consumption of dyes accounting to 62%, and in that consumption of reactive dyes had the highest share of more than 56%. However, the consumption of dyes in textile industries is associated with generation of large volume of coloured wastewater additionally containing non-ecofriendly chemicals and auxiliaries. Annually 10-15% of the total dyes produced are discharged as the coloured wastewater into natural water reservoirs[8,9]. Consequently, the toxic, carcinogenic, and mutagenic nature of synthetic dyes cause serious environmental impact and human health issues[10–12]. Further, a number of synthetic dyes are bio-accumulating in nature, which additionally necessitates their effective removal from the colored wastewater prior to discharge into natural water resources[13].

Several colour removal methods such as physical (adsorption, coagulation/flocculation (CF), ion exchange, and membrane filtrations), chemical (electrochemical oxidation, ozonation, and fentonreagent), and biological (degradation using microbes, algae, and enzymes), owing to peculiar advantages and limitations have been reported over the decades[2,14–17]. Physical methods are highly appreciated and often applied due to their simplicity, high colour removal efficiency, minimal chemical requirements, and no need of living organisms as like in biological methods. Amongst physical methods, adsorption is highly efficient; however, adsorbents of high sorption capacity are very costly and their disposal on post-service is also a challenge[3,18]. Wherein, colour removal *via* CF which dates back to 1500 BC is a simple and economical method, and till date number of natural and synthetic coagulants have been described for the effective removal of colourants from wastewater[19–21]. Natural coagulants are based on plant (guar gum, gum arabic), animal (chitosan), and microorganisms. Nonetheless, synthetic coagulants are comprised of hydrolyzed metallic salts (ferric chloride, magnesium chloride, and alum), pre-hydrolyzed metal salts (polyammonium chloride, polyferric sulfate), and synthetic cationic polymers (polyamine, polyethyleneimine). Factors such as pH, temperature of coagulation bath and coagulant dose,[22,23] coagulant type,[24,25] coagulant-aid,[26,27] dye-structure,[28] presence of surfactant,[29] etc. have significant influence on the colour removal efficiency *via* CF. Further, the effect of mixing-speed during CF on floc-characteristics such as morphology, size distribution, and settling behaviour has also been studied[30].

It has been observed that though the terms, coagulation and flocculation have been defined distinctly, are used interchangeably and ambiguously[31]. In general, CF involves the addition of coagulants which change the physical state of suspended and dissolved solids, and facilitate their removal *via* sedimentation[32]. In addition to textile, CF has also been used for remediation of wastewater from agriculture[33], food[34], beverage[35], and petrochemical[36] industries. Industrial wastewater containing heavy metal ions[37,38], sewage wastewater[39] and laundry[40] have also been treated using coagulants. In this respect, as an element of novelty, we are focusing on treatment of coloured wastewater produced in the dyeing laboratory of textile educational institute *via* CF. This is of high significance, since, many students perform the dyeing experiments simultaneously, and large volume of coloured wastewater containing considerable amount of dyes and chemicals is produced and discharged without any treatment. The discharged of coloured wastewater without any treatment ultimately pollutes the local water bodies. Further, considering the well documented environmental impacts and health issues of coloured wastewater, it is highly necessary to characterize and treat the coloured wastewater of textile educational institute as well prior to discharging-off into local water bodies. Hence, practicing the remedial approach for treatment of coloured wastewater before discharging, an ethical and sustainable approach for academic institutions, will prevent environmental pollution.

This study, therefore, aims to characterize the coloured wastewater generated in the dyeing laboratory of a textile educational institute and its treatment using coagulants. At first, dyeing of natural and synthetic fabric samples was performed using the standard dyeing procedure and the generated colored wastewater was analyzed for its volume, pH, turbidity, etc. Based on the characteristics of generated colored wastewater, two types of simulated colored wastewater were prepared and treated using two different coagulants. Effects of pH, coagulant dose and type on the colour removal efficiency were studied. Further, measures for controlling the volume and characteristic parameters of coloured wastewater and for improving the colour removal efficiency are also proposed.

Experimental Section

Materials

All the commercial textile dyes, chemicals, and auxiliaries were used as received. Cumulative 23 dyes were used in this study, and those are listed in Table S1. The details of dyeing-chemicals, textile-auxiliaries, and dyeing-grade fabrics are summarized in section S1 to S3 (Supplementary Information). Ferric chloride hexahydrate ($FeCl_3.6H_2O$, henceforth written as $FeCl_3$) was purchased from Loba Chemie Pvt. Ltd., India. A commercial coagulant, Afilan-RAMF (henceforth written as RAMF, colourless clear liquid, pH = 7, of 1% aqueous solution) was procured from Archroma India Pvt. Ltd., Mumbai, India. Potable tap water supplied by Brihanmumbai Municipal Corporation (BMC), Mumbai, Maharashtra, India was used through all the experiments, otherwise mentioned specifically.

Methods

The overall methodology of this study is presented in Fig. 1. Firstly, the fabric samples of natural and synthetic fibers were dyed using the standard dyeing procedure. The resultant volume of coloured wastewater generated during dyeing was collected and characterized after homogenizing for overnight. Then, based on the characteristics of coloured wastewater, two types of coloured wastewater (I and II) were prepared and subjected to colour removal treatment *via* CF.

Fig. 1 — Overall methodology of this study

Dyeing of natural and synthetic fabric samples

The stock dye solution (1 wt.%, 20 mL) was prepared in the standard volumetric flasks using standard dye dissolution process. The standard dyeing procedure followed for dyeing of natural and synthetic fiber based fabric samples (each of 2 g) at liquor ratio (LR) of 50 is described in section S4. Dyeing of polyester was performed in high temperature (HT) dyeing machine, whereas dyeing of rest of the fabric samples was performed on waterbath dyeing machine. Direct, reactive, vat, and sulfur dyes were used for dyeing of cotton. Acid and basic dyes were used for dyeing of wool, silk, and nylon. Basic dyes were also used for dyeing of acrylic. MC and disperse dyes were used for dyeing of wool and polyester, respectively. Dyeing shades for cotton, with direct and reactive dyes were 0.5 and 5%, whereas, it was 10% for sulfur dye. For the rest, the dyeing shades were 0.5 and 3%, irrespective of dye class and fabric sample.

Colour removal study

For colour removal study *via* CF, two types of simulated coloured wastewater (**I** and **II**) were prepared and the respective composition is presented in Table 1. The simulated coloured wastewater **(I)** belonged to coloured wastewater of individual dye class. For example, simulated coloured wastewater **(I)** of direct dyes was comprised of CI Direct Red 23 and Red 31 dyes used in this study and other chemicals like NaCl and Na_2CO_3 used in dyeing. Same approach was followed to prepare the simulated coloured wastewater **(I)** of different class of dyes. The colored wastewater **(II)** had the mixture of cumulative 23 dyes, dyeing-chemicals (acid, alkali, salt, etc.), textile-auxiliaries (leveling agent, dispersing agent, etc.), and non-ionic detergent (surfactant). Colour removal study was performed on a flocculator (JK and PC Texlab Equipments, Mumbai, Maharashtra, India) at room temperature using coagulants, $FeCl_3$ and RAMF (coagulant doses were 0.25, 5, and 1 g/L). The CF study was performed using 100 mL simulated coloured wastewater adjusted to pH of 6, 8 or 10, using 0.1 N solution of H_2SO_4 and NaOH. Coagulants and simulated colored wastewater were homogenized firstly at 120 rpm for 2 min and then at 20 rpm for 20 min. Finally, the mixture was left undisturbed for overnight to achieve the maximum possible settling of flocks formed during the CF.

Characterization

The total amount of colored wastewater generated on completion of dyeing experiments was collected and its volume was quantified using a measuring cylinder of 1 L capacity. pH of coloured wastewater was determined using a digital pH meter (EQ-614A, Equiptronics, Mumbai, Maharashtra, India) at room temperature. Total dissolved solid (TDS, mg/L) was determined according to the ASTM D5907-13 method. 25 mL of coloured wastewater was filtered using Whatman filter paper (grade-42), weighed, and evaporated to constant weight at 110°C (hot air oven, Modern Industrial Corporation, Mumbai, Maharashtra, India). The TDS value was calculated

Table 1 — Composition of simulated colored wastewater (I) and (II)

Composition	Concentration (mg/L)								
	Simulated Coloured wastewater								
	(I)								(II)[b]
	Direct	Reactive	Acid	Cationic	MC	Vat	sulfur	Disperse	
Dye[a]	200	200	200	200	200	200	200	200	1200
Glauber's salt	4000	24167	1334	134	-	800	-	-	13975
CH_3COONa	-	-	600	600	800	-	-	-	4340
Na_2CO_3	100	8334	-	-	-	200	400	-	934
NaOH	-	-	-	-	-	500	-	100	49
$Na_2S_2O_4$	-	-	-	-	-	400	-	100	45
Na_2S	-	-	-	-	-	-	400	-	35
CH_3COOH	-	-	300	300	400	-	-	-	120
H_2SO_4	-	-	600	600	200	-	-	-	125
Detergent	-	1000	-	-	-	600	200	-	1490
Levelling agent	-	-	1000	1000	-	-	-	100	7
Dispersing agent	-	-	-	-	-	-	-	100	4
Carrier	-	-	-	-	-	-	-	200	3

[a] mixture of different dyes from a particular dye class, in equal weight ratio

[b] mixture of cumulative 23 dyes in equal weight ratio

using Eq. (1), where, W_1 was the initial weight of coloured wastewater and W_2 was the weight of the residue.

$$\text{TDS (mg/L)} = \frac{(W_1 - W_2)}{W_1} * 100 \qquad \ldots(1)$$

The suspended solids in the coloured wastewater were expressed as turbidity index (Nephelometric Turbidity Units, NTU). The turbidity of coloured wastewater was determined using digital turbidity meter, NT-4000, Spectralab Instruments Pvt. Ltd., Mumbai, Maharashtra, India. Colour concentration in wastewater was estimated by measuring the optical density (absorbance value) using the double beam UV-visible spectrophotometer (UV-1800, Shimadzu Corporation, Japan), where deionized (DI) water was used as the reference. To determine the colour concentration in wastewater, absorbance value of coloured wastewater was compared with that of dye solution of known concentration (≤0.02%). For estimating the exhaustion percentage of dyes, initial and residual dye concentrations were estimated using UV-visible spectrophotometer. The absorbance values were considered for the equal volume of dye bath before and after dyeing. The colour removal efficiency *via* CF was calculated using Eq. (2), where, C_o and C_f were the absorbance values of simulated coloured wastewater prior and after CF, respectively. The compactness of sludge/flocs produced during CF treatment was examined visually on standing of flocs for overnight.

$$\text{Colour removal efficiency (\%)} = \frac{(C_0 - C_f)}{C_0} * 100 \qquad \ldots(2)$$

The chemical oxygen demand (COD) of coloured wastewater was determined by closed reflux colourimetric method[41]. For COD determination, 2 mL of coloured wastewater was digested with 2.8 mL and 0.25 mL of COD reagents solutions A and B, respectively, at 150°C for 2 h in a COD digester (DRB-200, Hach, China) and then cooled to room temperature. The blank sample (DI water, 2 mL) was also digested similarly. The change in the colour of digested control and coloured wastewater sample was compared using a portable data-logging colourimeter (DR/850, Hach, China) and the values are expressed as the COD (mg/L). The biological oxygen demand (BOD, mg/L) of coloured wastewater was determined by BOD_5 method[39]. The dissolved oxygen (DO) level in coloured wastewater at time t = 0 and after incubation of 5 days at 20°C was compared using a DO test kit (HQ30d, Hach China). The BOD value was calculated using Eq. 3, where, DO_0 and DO_5 were the DO levels in the coloured wastewater at t = 0 and after incubation period of 5 days. BC was the DO content in the blank sample (DI water) before and after incubation.

$$\text{BOD (mg/L)} = \frac{((DO_0 - DO_5) - BC) * 100}{DO_0} \qquad \ldots(3)$$

Results and Discussion

Characterization of coloured wastewater

In case of textile educational institutes, the coloured wastewater produced is primarily associated with colouration experiments of textile materials (fibers, yarns, and fabrics), where dyeing of textile materials in fabric form is the most preferred due to its ease of processing as compared to that of fibers and yarns. This study hence focuses the coloured wastewater of dyeing experiments of different natural and synthetic fiber based textile fabrics, and its characterization and remediation. The dyeing shades chosen were low to high, which accommodated the lowest and highest concentrations of various parameters of coloured wastewater, since, the concentration of chemicals and auxilliaries in the dyebath is governed by the depth of dyeing shade. In case of dyeing of cotton with reactive and direct dyes, dyeing shades were 0.5 and 5%, whereas it was 10% for sulfur black dye. The lowest dyeing shade for protein and synthetic fabrics was 0.5% and the highest dyeing shade was 3%, which usually produces very dark shades. While performing the dyeing experiments, water was used generously that enabled the quantification of maximum volume of coloured wastewater that may be generated. The volume of wastewater due to the miscellaneous factors such as leakages, running taps, washing of hands, etc. leading to increased total volume of coloured wastewater and reduction in characteristic parameters of coloured wastewater due to dilution was not considered.

The coloured wastewater resulted from pre-dyeing steps (preparation of stock dye solution, setting-up of dyebath, and rinsing of fabric samples) and post-dyeing steps (residual stock dye solution, unspent dyebath liquor, rinsing, washing, reduction clearing, and cleaning of dyebath and glassware) were all together accounted in the total volume of colored wastewater produced. Hence, the total volume of coloured wastewater, herein, represented the maximum possible extent of dilution and mimicked the coloured wastewater, which generates during the regular dyeing experiments. The total volume of coloured wastewater produced in case of cotton dyeing was between 11 L (direct dyes) and 23 L (vat dyes), and for rest of the dyes it was intermediate. In all other dyeing experiments, volume of coloured wastewater was ranged between 6.5 and 13 L (Fig. 2a). The volume (2.5-3.5 L) of coloured wastewater produced in preparatory steps of dyeing was similar in all the dyeing experiments, except dyeing of cotton with vat and sulfur dyes, where it was ranged between 6.5 and 7.5 L. The relatively higher consumption of water in preparatory steps of vat and sulfur dyes was owed to rinsing of fabric with reduced-alkaline solution of $Na_2S_2O_4$ and NaOH, and distinct dye solution preparation process under reduced-alkaline condition. These typical steps eventually needed more water for cleaning of glassware contaminated with high alkalinity and reducing agent. In case of dyeing of cotton, the total volume of coloured wastewater for different class of dyes was in order of vat > sulfur > reactive > direct. Amongst different class of dyes, the lowest volume of coloured wastewater was observed for dyeing of wool with acid dyes (7.8 ±1.4 L).

Once the total volume of coloured wastewater was estimated, it was homogenized overnight prior to characterizing for pH, TDS, turbidity, color concentration, BOD and COD values (Fig. 2). It is well known that protein and synthetic fabrics are dyed in acidic pH and during dyeing of cotton the pH of dyebath is of low to high alkalinity. The pH of coloured wastewater from acidic to highly alkaline was influenced by the factors such as pH of dyeing bath, pH of after-treatment, and degree of dilution. pH of coloured wastewater of acid, basic, and MC dyes was acidic (3.6-4.7 ±0.2). Though the dyeing of polyester with disperse dyes was performed in acidic condition (pH = 4.5-5.5), the coloured wastewater of polyester dyeing had pH of 7.2 ±0.5, which was caused by the alkaline-reduction clearing after-treatment. Colored wastewater of direct dyes had pH of 7.7 ±0.3, whereas in case of vat dyes it was 11.3 ±0.1. Coloured wastewater of reactive and sulfur dyes had pH between 9 and 11. Use of high dosages of alkali such as NaOH and/or Na_2CO_3 in dyeing of cotton with vat, reactive, and sulfur dyes and washing step after dyeing, caused the high alkalinity in respective coloured wastewaters produced.

It was observed that colored wastewater of cotton dyeing with reactive dyes had the highest TDS value amongst all the classof dyes. In case of reactive dyes, consumption of large amount of NaCl and Na_2CO_3 for exhaustion and fixation, respectively, use of non-ionic detergent for washing (removal of physically adhered and hydrolyzed reactive dye molecules from the fabric surface) and neutralizing step during washing caused the highest TDS ranging between 4280 and 5438 mg/L. TDS for colored wastewater of vat and sulfur dyes was between 1324 and 1507 mg/L, while it was significantly less in case of direct dyes, 167-393 mg/L. TDS value for colored wastewater of protein and synthetic fabrics ranged between 91-612 mg/L, which owed to the low dosages of dye bath additives during dyeing for exhaustion.

The turbidity of coloured wastewater of water-insoluble dyes was highest for wastewater for cotton dyeing with sulfur black dye and value of turbidity was as high as 32 NTU. The high turbidity value was preferably associated with relatively high dyeing shade of 10% and well known low exhaustion property of sulfur dyes. Fig. 3 depicts the residual dyebath liquor of polyester dyeing using HT and carrierdyeing method for dyeing shades of 0.5 to 3%. It was observed that in carrierdyeing method the dyebath liquor had higher residual color as compared to HT-dyeing method, and eventually higher turbidity. Coloured wastewater of polyester dyeing using carrier

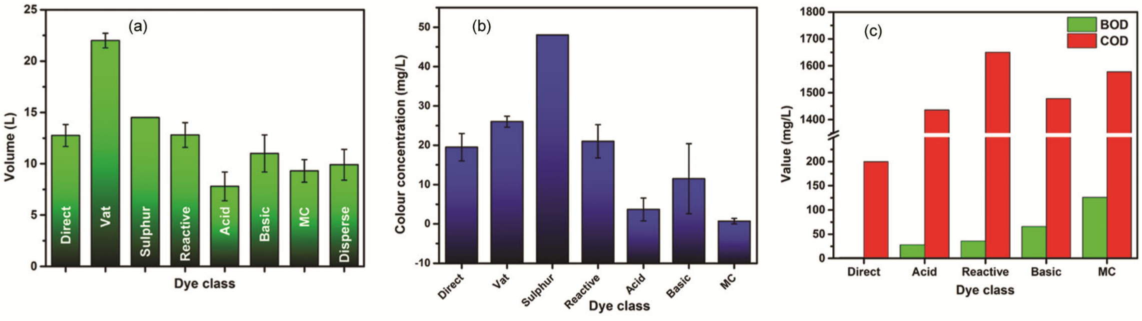

Fig. 2 — Characteristics of generated coloured wastewater; (a) volume, (b) colour concentration, and (c) BOD and COD

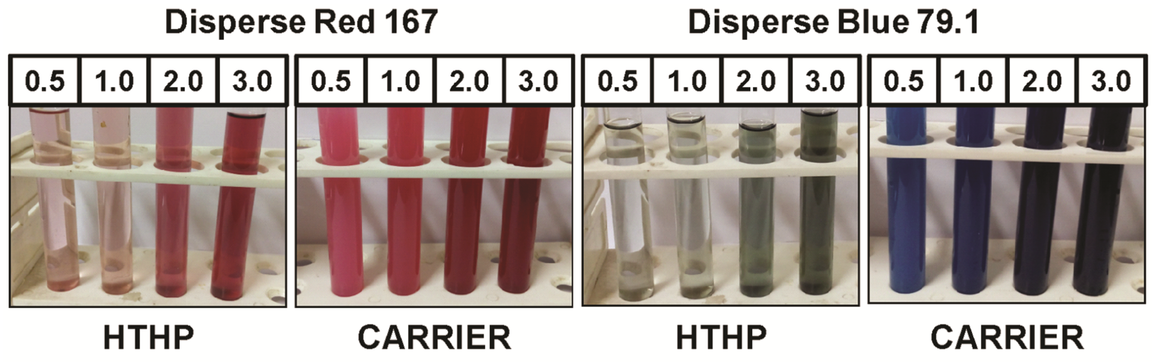

Fig. 3 — Residual dyebath of polyester dyeing with disperse dye (dyeing shades, 0.5-3%)

dyeing method had turbidity of 4-31 NTU and that of HT-dyeing method was 6-11 NTU. It anticipated that high turbidity in carrier dyeing method was a mixed effect of low dyeing temperature (100°C as compared to 130°C of HT-dyeing method) and insufficient dye-fiber-carrier interactions resulting in poor diffusion of dye molecules within the fibers from the dyebath[42].

Colour concentration in the wastewater (Fig. 2b) and residual dye bath were determined to estimate the exhaustion level and the values of exhaustion percentage is presented in Table 2. Colour concentration in the wastewater was highest for cotton dyeing with sulfur dyes (i.e. 48 mg/L) and it owed to a very high dyeing shade of 10%, and it is well established that with increasing dyeing shade exhaustion level of dyes on to the fibers decreases. Extremely high exhaustion of MC dyes on wool samples (up to 98% at dyeing shade of 0.5 and 3%) contributed the lowest colour concentration (1.5 ±0.7 mg/L) in the wastewater. Similarly, during dyeing of wool with acid and MC and of silk with acid dyes exhaustion values more than 90% were observed at 0.5% dyeing shade. Although dyeing of nylon with acid dyes had a high exhaustion level up to 98% at 0.5% dyeing shade, it decreased to 58% when dyeing shade was 3%. In case of basic dyes, the exhaustion values followed the trend, acrylic > silk > wool irrespective of dyeing shade. Dyeing of polyester with disperse dyes using HT-dyeing method showed better exhaustion as compared to carrier dyeing method. In general, at a given dyeing shade, in addition to dyeing method (polyester dyeing), exhaustion of dyes on the fibers was governed by the chemical structure of dye and fiber which influenced the dye-fiber interactions, and hence extent of migration of dye molecules from dye bath to fiber leading to high or low exhaustion values[43]. Further, especially for dyeing of cotton, addition of electrolyte (salt) promotes the physical adsorption of dye molecules on the fiber and lead to better exhaustion by diminishing the zeta-potential between cotton and dye molecules[44].

Table 2 — Exhaustion range of dyes for different class of dyes on different fabric samples

Fabric	Dye	Exhaustion (%)			
		Dyeing shade (%)			
		0.5	3	5	10
Cotton	Direct	58-65	-	15-47	-
Cotton	Reactive	40-92	-	25-73	-
Cotton	Vat^a	57-78	55-74	-	-
Cotton	Sulfur^a	-	-	-	72
Wool	Acid	91-98	86-98	-	-
Silk	Acid	96-98	89-98	-	-
Nylon	Acid	97-98	58-74	-	-
Wool	Basic	46-63	20-42	-	-
Silk	Basic	38-98	36-87	-	-
Acrylic	Basic	66-88	52-66	-	-
Wool	MC	96-98	96-98	-	-
PET	Disperse^b	Excellent	Fair	-	-
PET	Disperse^c	Moderate	Poor	-	-

^{a}measured in solubilized form (reduced-alkaline condition), ^{b}HT-dyeing method, ^{c}Carrier dyeing method

BOD values of the coloured wastewater generated in case of water soluble dyes was determined by BOD 5-days method and COD values were determined by closed reflux colorimetric method. It was observed that irrespective of the class of dye, BOD values were significantly lesser than COD values. The determined BOD values were ranged between 25 mg/L (acid dye) and 150 mg/L (MC dye). The significantly lesser BOD values of coloured wastewater for all the class of dyes than their COD values, indicated the high stability of dyes and other chemical impurities present in the coloured wastewater against biological degradation. This implied to their ability to persist in the natural environment for longer period. Presence of other additives and their concentration along with the dyes had the significant impact on the COD values of the coloured wastewater. The contribution of acetic acid towards BOD of coloured wastewater of cotton and polyester was negligible, since, acetic acid was neutralized due to presence of NaOH and/or Na_2CO_3 in coloured wastewater. In case of dyeing of wool, silk and nylon, and acrylic with basic, and MC dyes, use of acetic acid and low extent of dilution governed relatively higher BOD values of the coloured wastewater as compared to that of cotton dyeing with direct and reactive dyes. BOD values of coloured wastewater of acid dyes were comparable to that of reactive dyes and at much higher level that of direct dyes. COD values determined for coloured wastewater of MC and reactive dyes were comparable, which possibly linked to the distinct chemical structure of the dye molecules. The highest COD value (1650 mg/L) for reactive dyes was also certainly associated with the use of non-ionic detergent during the washing step on post-dyeing. Since, the non-ionic detergents are mostly non-biodegradable hence would have caused more COD. Overall, presence of different chemicals in the dyebath like acid, alkali, buffer salts, levelling agent, and non-ionic detergent, and their respective concentrations eventually influenced the BOD and COD values of the coloured wastewater. Moreover, establishing the correlation between the BOD and COD values with composition of coloured wastewater **(I)** was a challenge because all the dyes had different chemical structure and chemical composition of dyeing auxilliaries was unknown.

Colour removal study

The composition of simulated coloured wastewater (**I**) and (**II**) used for colour removal study is presented in Table 1. Although the residual colour in generated wastewater was less than 50 mg/L, the simulated coloured wastewater (**I**) and (**II**) were prepared with excess dye concentrations, i.e. 200 and 1200 mg/L, respectively. The purpose was to accommodate all the possible experimental conditions in dyeing laboratories such as a general tendency of preparing the dye solutions in excess, spillage of dye solutions, errors in setting-up dyebath, and repeat experiments. Further, dyebath additives such as acid, alkali, and salt, textile-auxiliaries, and non-ionic detergent used in respective class of dye were also the component of simulated coloured wastewater (**I**) and (**II**), which intended to assess their effects on colour removal efficiency. Primarily, the effect of coagulant dose and type (inorganic, hydrolyzed metal salt - $FeCl_3$ and polymeric coagulant - RAMF), and pH of coagulation bath on colour removal efficiency and nature of resulting flocs were studied. Other parameters of CF experiments for colour removal study like mixing time and speed, settling time, and temperature of coagulation bath were same. The color removal efficiency for both the coagulants at coagulant doses (0.25, 0.5, and 1 g/L) and pH of coagulation bath (6, 8, and 10) in case of simulated coloured wastewater (**I**) and (**II**) is summarized in Table 3.

Simulated colored wastewater (I)

It has already been established that during CF the mixing speed and the settling time have significant influence on the colour removal efficiency[45]. Hence, in this study, to avoid any variation in colour removal efficiency *via* CF, parameters i.e. mixing speed, mixing time, and settling time were kept persistent. It was observed that during the CF, irrespective of pH of the coagulation bath, coagulant dose and coagulant type, the settling time of 60 min for reactive and basic dyes, and 30 min for rest of the dyes resulted in settling of about 60-70% of flocks produced. Superiority of RAMF over $FeCl_3$ was witnessed w.r.t colour removal efficiency. For RAMF, the highest colour removal efficiency was observed for direct dyes (>98%, irrespective of pH and coagulant dose) and it was least for MC dyes (7.1%, pH 10, and dose, 1g/L). The better colour removal efficiency of RAMF as compared to $FeCl_3$ was associated with its polymeric and pre-neutralized nature. The pre-neutralized nature of coagulant is advantageous as the pH of coagulation bath had insignificant effect on the floc-formation ability and stability of produced flocs[46]. In case of $FeCl_3$, the least colour removal efficiency was observed for reactive dyes (22.6%, pH 10, and dose, 1g/L) and highest for direct dyes (97.4%, pH 10, and dose, 0.25 g/L). However, it was difficult to draw a general relationship for colour removal efficiency of both the coagulants w.r.t increasing coagulant dose and pH of coagulation bath. For both the coagulants, colour removal efficiency was a mixed effect of pH of coagulation bath and coagulant dose. For example, at 0.25 g/L dose of $FeCl_3$, with increasing pH, the colour removal efficiency for basic dyes was initially increased then decreased, whereas in case of direct and reactive dyes it was increased linearly with increasing pH of coagulation bath. Similarly, for the polymeric coagulant also the colour removal efficiency was

Table 3 — Colour removal efficiency for simulated coloured wastewater (**I**)

Dye class	Coagulant dose (g/L)	Colour removal efficiency (%)					
		$FeCl_3$			RAMF		
		pH			pH		
		6	8	10	6	8	10
Direct	0.25	92.5	96.8	97.4	98.2	98.9	99.1
	0.5	94.6	93.2	83.6	98.9	98.9	98.8
	1.0	67.7	68.5	76.6	99.1	99.1	99.1
Reactive	0.25	3.4	4.1	15.8	63.9	58.8	43.8
	0.5	4.8	2.7	19.9	80.9	80.4	71.6
	1.0	12.3	3.4	22.6	89.2	78.6	70.9
Acid	0.25	54.6	50.9	50.0	92.1	88.4	81.8
	0.5	57.3	57.4	56.5	98.4	98.1	97.9
	1.0	64.9	60.9	62.8	97.1	96.7	96.3
Cationic	0.25	78.6	79.3	76.9	90.4	90	90.1
	0.5	76.4	76.31	78.6	90.2	90.5	90.4
	1.0	77.9	76.59	77.8	90.3	90.5	90.1
MC	0.25	56.6	66.7	65.0	95.6	94.1	83.7
	0.5	67.2	71.9	75.1	88.5	81.3	41.8
	1.0	88.9	88.1	86.7	13.5	10.3	7.1
Vat	0.25	65.5	60.5	58.4	70.4	75.9	79.6
	0.5	72.7	69.4	65.5	80.2	84.4	87.6
	1.0	75.4	73.28	68.4	81.3	83.5	84.1
sulfur	0.25	76.6	64.8	71.9	68.5	97.5	98.4
	0.5	91.9	86.6	89.1	66.3	57.6	48.8
	1.0	93.2	88.5	90.7	65.2	50.3	41.7

decreased or increased with increasing pH at a given coagulant dose. For wastewater of disperse dyes, the removal efficiency was assessed visually due to their water-insolubility. The highest colour removal efficiency for disperse dyes using both the coagulants reflected at coagulant dose 0.5 g/L and pH of 10, where highly compact flocs were produced in case of RAMF (Fig. 4).

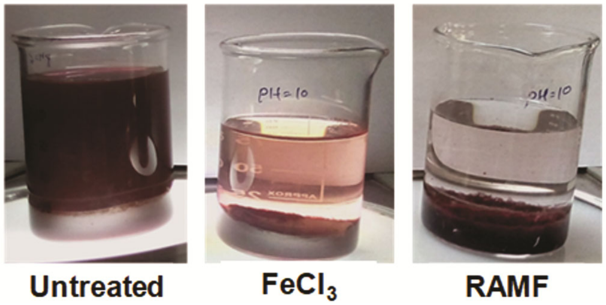

Fig. 4 — CF treatment of simulated coloured wastewater of disperse dyes

To summarize, for both the coagulants, the colour removal efficiency was increased, decreased or initially increased and then decreased with increasing pH and/or coagulant dose. Certainly, different dye-coagulant interactions at different pH and different coagulant dose influenced the colour removal efficiency. It has been established that depending upon the pH of coagulation bath and chemical nature of dye, the dye-coagulant interactions are governed by different destabilization mechanisms such as charge-neutralization, adsorption, and bridge formation, which modify the colour removal efficiency[47]. The colour removal efficiency at a particular pH is a result of neutralization of anionic charges present on dye molecules; hence it governs the floc formation during mixing and settling. Conversely, at a particular pH, increasing coagulant dose after a critical level, promotes the charge generation on flocs, which causes the enlargement of flocs through destabilization (occurring due to mutual-repulsions), and eventually reduces the removal efficiency[48]. Further, it has been speculated that at a particular pH and coagulant dose, the chemical-structure, ionic-nature, and solubility of the dye affect the optimum colour removal efficiency[26]. The relatively low colour removal efficiency for coloured wastewater of reactive dyes in case of $FeCl_3$ implies to presence of non-ionic detergent in the coloured wastewater which minimizes the aggregation of dye molecules, and hinders the formation of compact flocs[49].

Simulated colored wastewater (II)

The colour removal study for simulated coloured wastewater **(II)** was also performed in a manner like coloured wastewater **(I)** and the characteristics of coloured wastewater **(II)** prior and post-CF are presented in Table 4 and Fig. 5. After CF treatment, the turbid and dark blue coloured wastewater **(II)** was transformed into a clear and yellow colour. The colour removal efficiency of RAMF was significantly higher than that of $FeCl_3$, irrespective of coagulant dose and pH of coagulation bath. For $FeCl_3$, colour removal efficiency was initially increased and then decreased with increasing pH from 6 to 10; whereas, for RAMF it continuously declined with increasing pH from 6 to 10. For both the coagulants, increasing coagulant dose increased the colour removal efficiency at a given pH level. At a given pH and coagulant dose up to 1 g/L, the colour removal efficiency for RAMF was much higher than that of $FeCl_3$. The maximum colour removal efficiency for RAMF was 85% (pH, 6 and dose, 1 g/L) and 44% for $FeCl_3$ (pH, 8 and dose, 1 g/L). To further investigate the effect of RAMF dose on colour removal efficiency, its concentration was increased up to 1.5 g/L. It was observed that colour removal efficiency was increased slightly on increasing the RAMF dose from 1 to 1.5 g/L, and a plateau was observed after RAMF dose of 1.25 g/L. Hence, the highest dose of polymeric coagulant was restricted to 1.5 g/L. It was observed that, the maximum colour removal efficiency for **(II)** was relatively lower as compared to **(I)**. The little decrease in maximum colour removal efficiency for **(II)** was possibly the cause of different dye-coagulant interactions and inter-competition of dyes from different class of dyes. It was also observed that on post-CF, the values of absorbance, turbidity, and pH for **(II)** were significantly reduced. Following the colour removal using RAMF (1.25 g/L), COD of the supernatant was estimated, and it was found to be little higher as compared to that of **(II)** before CF treatment, which was possibly associated with excess dose of polymeric coagulant in coagulation bath, i.e. 1.25 g/L.

Table 4 — Characteristics of simulated colour wastewater (II)

Characteristics	Initial	After CF (RAMF, 1.25 g/L and pH = 6)
Colour	Dark blue	Light yellow
Appearance	Turbid	Clear
Turbidity (NTU)	27.28	6.8
pH	12.48	6.5
Colour removal efficiency using RAMF (%)	-	87
COD (mg/L)	1480	1526

Recommendations

The characteristics of coloured wastewater generated in dyeing experiments and results of colour removal study *via* CF, are the basis for proposing the recommendations to reduce the volume and contamination level in coloured wastewater of academic institutions. The reduction in the volume and contamination level in coloured wastewater will further improve the colour removal efficiency by using coagulants or any other employed method. Overall, the recommendations summarized as follows are easily accessible and need to be implemented and practiced for reduction and effective remediation of the coloured wastewater.

Abiding to standard procedures while performing the dyeing or any other experiments is the most important aspect is to reduce the wastewater volume and its characteristics. The information like chemical structure of dye, dyeing recipe, dyeing method, exhaustion values of dyes, storage conditions of dyes and dyeing-auxiliaries are often provided by the suppliers, and those need to be well documented and utilized. Dyes which possess high extent of exhaustion on to the fibers (exhaustion values for a dye-fiber combinations at different dyeing shades are usually reported by the suppliers or that may be searched from the literatures) will contribute lesser residual dye in the coloured wastewater, hence such dyes should be used for performing the coloration

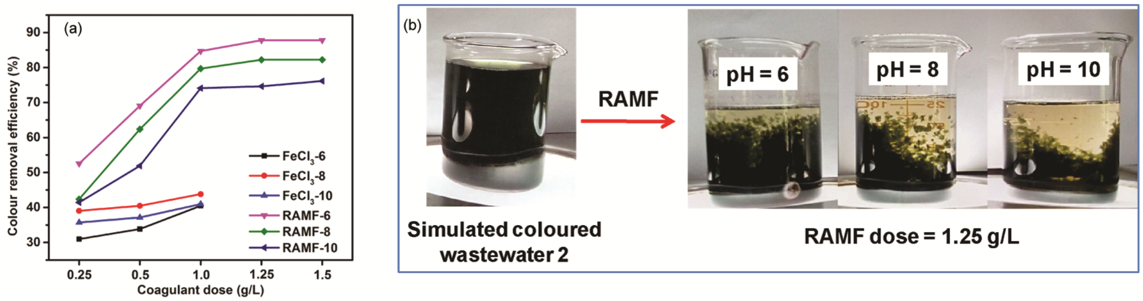

Fig. 5 — CF treatment of simulated coloured wastewater (II); (a) color removal efficiency and (b) color removal using RAMF

experiments. Since, dark dyeing shades require more time for exhaustion, hence, dyeing shades more than 3% should be avoided as the experimentation time is limited in the academic curriculum, which will reduce the concentration of dyes and chemicals in the generated coloured wastewater. The concentration of dyeing chemicals and auxiliaries during dyeing should always be used according to the values specified or suggested in the datasheets provided by the suppliers, to eliminate their excess consumption and minimize their concentration in the coloured wastewater. For example, TDS removal from the coloured wastewater is challenging and needs the advanced technology like reverse osmosis. Hence, if the concentration of exhausting agent like sodium salt or Glauber's' salt during dyeing is used as suggested by the supplier, it will reduce the TDS load. Use of dyeing auxilliaries whose composition is generally unknown in the specified concentration is mandatory to avoid the complexities during coloured wastewater treatment. These two cases can be easily correlated with the CF treatment of coloured wastewater of reactive dyes, where very low level of colour removal efficiency was observed for $FeCl_3$ due to presence of non-ionic detergent in the wastewater. Moreover, it would be better to replace the commercial dyeing auxiliaries with pure chemicals having similar functions. For instance, ethylene and propylene glycol, and glycerin may be effectively used as the wetting agent instead of commercial ones. Hence, such alternate chemicals need to be recognized and should be used during dyeing as emulsifier, wetting, dispersing, levelling, and retarding agent, as applicable. Further, this strategy will help in predicting the characteristics of coloured wastewater.

The strategy of segregation is often reported in the literatures focusing on textile coloured wastewater treatment. This strategy can be specifically recommended for segregating the residual dyebath from the washing wastewater of reactive dyeing where the presence of non-ionic detergent deteriorated the colour removal efficiency *via* CF for $FeCl_3$ as observed in this study. The segregation of coloured wastewater will eliminate the dilution and facilitate the efficient treatment of coloured wastewater of low volumes. Precisely, if the coloured wastewater is diluted then the dye molecules and other impurities are dispersed over a large volume and to capture those impurities during the coloured wastewater treatment *via* CF or any other method is relatively challenging, and more inputs of coagulants, energy, and time are required.

Dyeing experiments, involving the use of environmental deteriorating chemicals should only be the demonstrative once. For example, polyester dyeing using carriers and dyeing of cotton with azoic colours. Since, carriers are generally aromatic hydrocarbons and ethers, phenolic and chlorinated aromatic compounds, most of the carriers are found to be toxic for humans and aquatic organisms. Similarly, the toxic NOx fumes generated during the dissolution of base and as many of the azoic colour (naphthol and base combinations) have been identified as carcinogen, hence, such naphthol and base combination should be avoided. Further, the demonstrative nature azoic colour dyeing of cotton will contribute reduced volume of coloured wastewater and NOx fumes in the environment. Experiments on bleaching of cotton and other fibers using chlorine based bleaching agents should also be avoided as these compounds produce problem of adsorbable organic halides when discharged in the environment. Interestingly, as many reactive dyes are prone to oxidative degradation in presence of oxidizing agents like hydrogen peroxide, the wastewater of hydrogen peroxide bleaching may be used to decolorize the coloured wastewater of reactive dyes prior or later to colour removal *via* CF or any other method.

Miscellaneous factors such as leakages in taps and water-pipes, and wastewater generation due to unnecessarily running of water-taps need to be controlled. Preparation of stock dye solution in minimum volumes as much as possible and its complete consumption will also avoid the surplus colour concentration and other impurities in the wastewater. Creating the awareness among the students about the harsh effects of coloured wastewater on the environment, aquatic life and human health is highly necessary, so that students will take extra precautions to reduce the coloured wastewater volume and contamination level while performing the experiments. The strategy of right first time and every time should be emphasized to the students to reduce the coloured wastewater volumes generated due to reprocessing (repeat experiments) of textile materials. Workshops may also be conducted for elaborating the different strategies to reduce the coloured wastewater volumes and characteristics. Polymeric coagulants, as they are effective in low dosage and perform well over a wide range of pH are beneficial as compared to those based on hydrolyzed metallic-salts. Further, polymeric coagulants do not contribute metal ions in the wastewater hence may be preferred for performing the colour removal experiments *via* CF. Although CF process is highly effective for removal of many of the dyes form the coloured wastewater, however, sludge volume and its safe disposal are still a concern. Hence, sludge produced during the CF should be collected, dried, stored in air-tight containers, and disposed-off at the hazard disposal-sites.

Conclusion

In this study, the characterization and remediation of coloured wastewater produced in dyeing experiments of the textile curriculum is emphasized. The coloured wastewater obtained after dyeing of natural and synthetic fabric pieces using standard dyeing procedures was characterized, quantitatively and qualitatively. Experiments on dyeing of cotton with vat dyes produced highest volume of coloured wastewater. The dyeing conditions and after treatments influenced the pH of coloured wastewater. Coloured wastewater of sulfur dye had the highest colour concentration and that of reactive dyes demonstrated highest TDS and COD values. The residual colour concentration in the wastewater generated during dyeing was less than 50 mg/L. The characteristic values of coloured wastewater influenced the composition of simulated coloured wastewater **(I)** and **(II)** containing dyes, dyeing chemicals, and dyeing auxiliaries. The excess concentration of dyes and chemicals in simulated coloured wastewater accommodated the miscellaneous factors such as spillages of dye solutions, preparation of excess of stock dye solution, and repeat experiments. The color removal study was performed using $FeCl_3$ and a polymeric coagulant. The colour removal efficiency of polymeric coagulant was superior to $FeCl_3$ at lower coagulant dose and it was not influenced much by the pH of coagulation bath. For coloured wastewater of direct dyes, colour removal efficiency was more than 90% for both the coagulants. For $FeCl_3$, lowest colour removal efficiency was observed for the coloured wastewater of reactive dyes and for RAMF it was lowest in case of wastewater of MC dyes. The colour removal efficiency was influenced by the pH of coagulation bath, coagulant dose and type.

The suggests recommendations are the preventive measures to reduce the volume and characteristics of colored wastewater, which will efficiently improve the color removal efficiency *via* CF. Certainly, the recommendations are specific to general in nature, and their adapted will positively reduce the volume of coloured wastewater to be discharged from the academic or any other dyeing laboratory. Moreover, the recommendations will facilitate the generation of coloured wastewater with relatively lower amount of dyes and chemicals, thus directing towards the sustainability. Succinctly, the results of this study may be served as the stepping bridge for the development of wastewater treatment system at textile educational institutes where the cumulative volume of coloured wastewater is large and should not be neglected.

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Supplementary Information

Supplementary information is available on the website http://nopr.niscpr.res.in/handle/123456789.

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