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Chemical or electrochemical techniques, followed by ion exchange, for recycle of textile dye wastewater

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Abstract

This paper examines the use of chemical or electrocoagulation treatment process followed by ion-exchange process of the textile dye effluent. The dye effluent was treated using polymeric coagulant (cationic dye-fixing agent) or electrocoagulation (iron and aluminum electrode) process under various conditions such as various current densities and effect of pH. Efficiencies of COD reduction, colour removal and power consumption were studied for each process. The chemical or electrochemical treatment are indented primarily to remove colour and COD of wastewater while ion exchange is used to further improve the removal efficiency of the colour, COD, Fe concentration, conductivity, alkalinity and total dissolved solids (TDS). From the results chemical coagulation, maximum COD reduction of about 81.3% was obtained at 300 mg/l of coagulant whereas in electrocoagulation process, maximum COD removal of about 92.31% (0.25 A/dm²) was achieved with energy consumption of about 19.29 kWh/kg of COD and 80% (1 A/dm²) COD removal was obtained with energy consumption of about 130.095 kWh/kg of COD at iron and aluminum electrodes, respectively. All the experimental results, throughout the present study, have indicated that chemical or electrocoagulation treatment followed by ion-exchange methods were very effective and were capable of elevating quality of the treated wastewater effluent to the reuse standard of the textile industry.

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Keywords: Textile dye effluent; Chemical treatment; Electrocoagulation; Ion-exchange process

1. Introduction

Many techniques have been used for treatment of waste wastewater, such as adsorption, biological treatment, oxidation, coagulation and/or flocculation, of such coagulation is one of the most commonly used techniques. Inorganic coagulants such as lime and salts of iron, magnesium and aluminum have been used over many years, but with changes in dyes and with the dye consents proposed by water authorities, these no longer given completely satisfactory treatment [1]. Not all dyes are effectively removed by inorganic coagulants; thus alum is unsatisfactory for the removal of soluble dyes such as reactive and direct dyes [2]. Organic polymers have therefore been developed for colour removal treatment and, in general, they offer the same advantages with the production of less sludge and significantly improved colour removal [1]. For the removal of colour

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from dye house effluent, no such effective and simple inorganic option exists for many soluble dyes [3]. Traditional methods for dealing with textile wastewater consist of various combinations of biological, physical and chemical treatment methods [4]. Common biological treatment processes are often ineffective in removing dyes, which are highly structured polymers with low biodegradability [5]. Various physical-chemical techniques are also available for the treatment of aqueous streams to eliminate dyes; chemical coagulation followed by sedimentation [6] and adsorption are the widely used ones [7], but other advanced techniques are often applied, e.g. UV [8,9], ozonation [10], ultrasonic decomposition, or combined oxidation processes [11–13]. Electrocoagulation is a process consisting of creating metallic hydroxide flocs within the wastewater by electrodissolution of soluble anodes, usually made of iron or aluminum. This method has been practiced for most of the 20th century with limited success. Recently, however, there has been renewed interest in the use of electrocoagulation owing to the increase in environmental restrictions on effluent wastewater. In the past decade, this technology has been increasingly used in developed countries for the treatment of industrial wastewaters [14-16]. Electro-

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coagulation has been proposed for the treatment of various effluents such as wastewater containing food and protein wastes [17], textile wastewater [18], aqueous suspensions containing kaolinite, bentonite, and ultra fine particles [19,20], fluoridecontaining water [21], restaurant wastewater [22,23], textile dye solution [24-25] and smelter wastewater containing has full arsenic [26]. Several researchers have studied the feasibility of electrochemical degradation of textile dyes using various electrode materials for wastewater treatment. Electrochemical degradation of different dye compounds was studied using titanium based DSA electrodes [27,28], platinum electrode [29], diamond and metal alloy electrodes [30] and boron doped diamond electrodes [31,32]. One available treatment technology widely used in recent years is Fenton oxidation. This advanced chemical oxidation technology is based on the production of hydroxyl radicals, •OH, which have an extremely high oxidation potential ($^{\bullet}OH/H_2O = +2.73 \text{ V}$). The Fenton oxidation process has been employed successfully to treat different industrial wastewaters [33–38]. These methods are indented to treat the textile wastewater to a level that meets the discharge standards required by the government. However, due to dwindling supply and increasing demand of water in the textile industries, a better alternative is to attempt to further improve the water quality of treated wastewater for reuse standard. Meanwhile, high treatment costs of these methods have stimulated, in recent years, the search for more cost effective treatment methods and reuse standards. In the present work, an attempt has been made to study the treatment efficiency of chemical (polymeric chemical coagulation) or electrocoagulation process for the removal of colour and COD of the effluent, followed by ion-exchange process for further lowering the colour, COD, Fe concentration, conductivity, alkalinity and total dissolved solids (TDS).

2. Mechanism of polymeric coagulant

Textile dye-fixing agents are used as a polymeric coagulant. Direct and reactive dyes contain RSO₃⁻ group and dye-fixing agent are RN⁺Cl⁻. When these two solutions are dissolved in water, they ionize in to long cations and small anions. On the other hand, when a dye is dissolved in water, it ionizes into long coloured anions and small sodium ions. When theses two solutions are mixed the long ions of opposite ionic charge combine with one another to form an insoluble complex [39].

$$\underset{(Dye)}{\text{RSO}_3\text{Na}} \leftrightarrow \text{RSO}_3^- + \text{Na}^+ \tag{1}$$

$$\begin{array}{c} R_4 N - Cl \\ (Polymeric \ coagulant) \end{array} \leftrightarrow R_4 N^+ + Cl^- \end{array} \tag{2}$$

$$R_4N^+ + RSO_3^- \rightarrow \underset{(Insoluble complex)}{RSO_3 - NR_4}$$
(3)

2.1. Mechanism of electrocoagulation

Four main mechanisms have been suggested to describe the process by which contaminants are removed from wastewa-

ter using this system. These consist of surface complexation, electrostatic attraction, chemical modification, and precipitation.

The chemistry of surface complexation is not well understood and is specific to each effluent, but is thought to occur in the following manner [40]:

$$Dye-H + (HO)OFe \rightarrow Dye-OFe + H_2O$$
 (4)

$$Dye-H + (HO)OAl \rightarrow Dye-OAl + H_2O$$
 (5)

In addition, for forming all possible surface complexes, it may be possible to remove remaining dyestuff by simple electrostatic attraction, hydrous iron oxide. In combination with the various surface complexes, contains areas of apparent positive and negative charge. The attraction of opposite charges is sufficient to remove some dissolved species from the effluent stream. Chemical modification may occur on passage of effluent through the electrochemical cell as well as during subsequent degassing. The chemistry C=C and N=N (common in dyes) allows "catalytic hydrogenation" or reduction to occur in the presence of hydrogen gas and a catalyst. The presence of metal ions and hydrogen gas in this reaction suggests that the above could occur, but further research is required to confirm the extent of this mechanism and the conditions, which favor it. Precipitation is a mechanism by which soluble species with opposite charges react with one another to form insoluble solids. This reaction often determines the success of heavy metal removal from contaminated water. In the case of dyestuffs, however, it is believed to have limited applicability [40].

3. Material and methods

All the reagents used were of Analytical Reagent grade. Polymeric coagulant (dye-fixing agent- Sandofix-WRN) and dye effluent (Procion Black 5B-Reactive dye) were collected from Textile industry. The cross-linked polystyrene bead resins, which are commercially known as Amberlite IR 120 and Amberlite IRA 400, were used.

3.1. Analytical measurements

The analytical methods of the COD of all samples were determined by the dichromate reflux method [41]. In electrochemical process the chloride ions cause interference, because they can be oxidized to Cl_2 by dichromate under the analysis conditions, the samples were treated with Ag_2SO_4 to form a precipitate of AgCl that was separated by centrifugation. The oxidized products and the reduction in dye concentration were measured by using a UV–Visible Spectrophotometer (Systronics 118), the colour removal was calculated by following formulae:

$$\text{Color removal}(\%) = 100 \frac{[\text{ABS}_0^{\text{M}} - \text{ABS}^{\text{M}}]}{\text{ABS}_0^{\text{M}}}$$
(6)

where ABS^M is the average of absorbance values as it is maximum absorbency visible wavelength. ABS_0^M the value before electrolysis, ABS^M the value after electrolysis.



3.2. Electrochemical cell

The electrolysis cell (Fig. 1) used in the present study consists of a glass beaker of 500 ml capacity closed with a PVC lid having provision to fit a cathode and an anode. Commercially available mild steel and aluminum were used as anodes while stainless steel was used as cathode. It is connected to 2 A and 15 V dc regulated power supply.

3.2.1. Ion-exchange experimental set up

The ion-exchange column consisted of glass column of length 60 cm and diameter 2.5 cm. It was plugged with cotton at the bottom for free flow of the treated wastewater and to hold the resin intact. The columns consisted 20 ml of cation-exchange resin (Amberlite IR 120) and other one consisted of 40 ml of anion-exchange resin (Amberlite IRA 400). The constant flow rate (5 ml/min) through the column was maintained with the help of a peristaltic pump. The flow cell is shown in Fig. 2.

3.3. Experimental procedure

The present experimental study involved two major processes such as chemical treatment or electrocoagulation (iron and aluminum electrode) process followed by ion-exchange process. These two treatments can be operated independently in batchwise processes. Two hundred millilitres of industrial effluents (Procion Black 5B-Reactive dye) of known concentration were taken for each experiment. In this first process the effluent was



Fig. 2. Ion-exchange experimental set up: (A) treated wastewater, (B) cationexchange resin column, (C) anion-exchange resin column, (D) reusable water and (E) peristaltic pump.

chemically treated using appropriate amounts of Sandofix-WRN (cationic dye-fixing agent) under various conditions whereas in the second process, electrocoagulation was carried out under galvanostatic condition. Samples were collected at regular intervals of time for estimation of COD, colour, and power consumption. After completion of chemical treatment or electrocoagulation, the wastewater was then subjected to ion-exchange process. The ion-exchange experiments were conducted in a flow cell process.

3.3.1. Chemical treatment

In chemical treatment, textile dye-fixing agent was used as a polymeric coagulant. Four hundred millilitres of wastewater sample was taken and coagulated using Sandofix-WRN (cationic dye-fixing agent) under various pH ranges from 2 to 12 and amount of coagulant required was optimized.

3.3.2. Electrocoagulation

Electrocoagulation is the modification of electrofloatation where the coagulation agent is produced in situ at the respective electrodes (The most commonly used electrodes are iron and aluminum.). In this method, the pollutant is transformed into amorphous precipitates or adsorbed on hydroxides formed at the anodes. This method is also called as electroflocculation. Four hundred millilitres of wastewater sample was taken and coagulated. In this process, mild steel/aluminum were used as anode and stainless steel was used as cathode under various current densities $(0.25-1 \text{ A/dm}^2)$.

3.3.3. Ion-exchange process

After the chemical or electrocoagulation treatment, 1000 ml of treated wastewater subjected to ion-exchange columns under constant flow rate (5 ml/min). Before the ion-exchange process, the pH was adjusted to about 7 and the wastewater was filtered using glass wool to remove the suspended solids. The filtration significantly helped to avoid fouling of ion-exchange resins. The samples were taken periodically for measurements of COD,



Fig. 3. Effect of COD reduction and colour removal on polymeric coagulant (Sandofix-WRN).

colour, conductivity, S.S, total hardness, alkalinity, Fe and other ion concentrations.

4. Result and discussion

4.1. Polymeric coagulants

The effect of coagulant dosage on the percentage of COD reduction and colour removal for dye effluent are presented in Fig. 3. These results indicate that a significant amount of dye can be removed. It was also observed that removal efficiencies of both COD and colour increases with increase of coagulant dosage. The maximum COD reductions of 81.3% and colour removal of about 98.2% were obtained at 300 mg/l of coagulant. It is evident from the results that minimum dosage amount of coagulant (300 mg/l) is required to treat the above dye wastewater. It is also observed that the COD began to appear in the electrolyte when dosage amount exceeds the optimum level. This could be due to the presence of the residual coagulant remains in the treated water. Also the pH of the electrolyte solution has been varied in order to check its influence on colour removal and COD reduction. Experiments were conducted under acid, alkaline and neutral conditions and the observed results are given in Fig. 4. It can be ascertained from Fig. 4 that the percentage of COD reduction has increased from 56% (pH 7) to 81.3% (pH 10) when the electrolyte pH increased to alkaline condition from neutral. On contrary, in acidic condition precipitation was not seen. Increase in the pH of electrolyte favors the precipitation reaction. Consequently precipitation of dye has been increased. The reaction was favorable at neutral (pH 7) and more acute in alkaline conditions particularly at pH 10.



Fig. 4. Effect of pH on COD removal: (\$) 300 mg/l.



Fig. 5. Effect of current density on COD (iron electrode) removal. (\blacklozenge) 0.25 A/dm², (\blacksquare) 0.5 A/dm², (\bigstar) 0.75 A/dm², (\bigstar) 1 A/dm².

4.2. Electrocoagulation

The electrocoagulation has been carried out for two different anodes, namely iron and aluminum. The optimum conditions for effective removal of COD, colour and minimum energy consumption have been studied at different current densities in batch reactor. The supply of current to the electrocoagulation system determines the amount of Al³⁺ or Fe²⁺ ions released from the respective electrodes. For aluminum, the electrochemical equivalent mass is 335.6 mg/Ah. For iron, the value is 1041 mg/Ah. Experiments were conducted at four current densities keeping other parameters constant. The COD reduction increased with current density (Figs. 5 and 6). This may be explained that the release of ions, i.e. the release of metal ions increased with current density, which eventually increases the reduction of COD. The efficiencies of COD removal at different reaction times in various conditions are shown in Figs. 5 and 6. These results show that 100% COD reduction was achieved while iron electrode was used. On the other hand, it was 80% with aluminum electrode. Figs. 7 and 8 show colour removal efficiencies at different reaction time under various current densities. The maximum colour removal efficiencies were in the order of 97.06 and 94.27% for



Fig. 6. Effect of current density on COD (aluminum electrode) removal (\blacklozenge) 0.25 A/dm², (\blacksquare) 0.5 A/dm², (\bigstar) 0.75 A/dm², (\bigstar) 1 A/dm².



Fig. 7. Effect of current density on colour removal (iron electrode) (\blacklozenge) 0.25 A/dm², (\blacksquare) 0.5 A/dm², (\bigstar) 0.75 A/dm², (\bigstar) 1 A/dm².



Fig. 8. Effect of current density on colour removal (aluminum electrode) (\blacklozenge) 0.25 A/dm², (\blacksquare) 0.5 A/dm², (\bigstar) 0.75 A/dm², (\bigstar) 1 A/dm².

iron and aluminum electrodes. Though the COD removal was 100% while using mild steel electrode, the residual colour indicated incomplete removal of soluble iron. But COD reduction was more than that of aluminum electrode due to more soluble in iron electrode during the electrolysis. The power consumption of the both electrodes was shown in Figs. 9 and 10. On the other hand, power consumption has increased with increasing



Fig. 9. The variation of power consumption with time for the different current densities applied (iron electrode) (\blacklozenge) 0.25 A/dm², (\blacksquare) 0.5 A/dm², (\land) 0.75 A/dm², (\checkmark) 1 A/dm².



Fig. 10. The variation of power consumption with time for the different current densities applied (aluminum electrode) (\blacklozenge) 0.25 A/dm², (\blacksquare) 0.5 A/dm², (\land) 0.75 A/dm², (\checkmark) 1 A/dm².

applied current density. The 100% COD removal was achieved within 6 h with a maximum energy consumption of 78.5 kWh/kg of COD at 1 A/dm². However, at 0.25 A/dm², the maximum COD removal of t 92.31% was achieved with lower energy consumption of about 19.29 kWh/kg of COD in iron electrode and 130.095 kWh/kg of COD at 1 A/dm² with 80% of COD removal in the aluminum electrode for maximum current density. Therefore, an optimum point must be carefully determined giving a faster removal rate and energy consumption quite low. Hence, the optimal current densities of 0.25 and 1 A/dm² were chosen for iron and aluminum electrodes, respectively. From the results, it is understood that, the process involving iron electrode, completely removes the dye with low electrical consumption in a short time.

4.3. Ion-exchange process

In the present study, chemically/electrochemically treated wastewater was subjected to ion-exchange process. The conductivity of the wastewater after chemical treatment and electrocoagulation treatment was observed as 4910 and 5200 µmho/cm, respectively, against the reuse standard of 100 µmho/cm. Such a high conductivity of wastewater indicates that it still contains a significant amount of inorganic salts and other ions. To remove these ions and other impurities, both crosslinked divinylbenzene-polystyrene based cationic (Amberlite IR 120) and anionic (Amberlite IRA 400) ion-exchange resins were used in the flow cell experiment. A cation-exchange resin captures positively charged ions and an anion-exchange resin negatively charged ions. In fresh resin, the cations attached to the cationic resin are H⁺, those attached to the anionic resin OH⁻. Treated wastewater, which was passed through the cationexchange resin resulted in exchange of the cations (displacing of H⁺) and subsequently passed through the anion-exchange resin absorbed anions, displacing the OH⁻ ions, As a result, finally ion free water was obtained. The optimum ratio of resins were studied at room temperature using 100 ml of treated wastewater (5200 µmho/cm) and amount of resin (Amberlite IR 120 (H⁺): Amberlite IRA 400 (OH⁻) in ml) 1:1, 1:2, 1:3, 2:1 and 3:1 in

Table 1Water quality change after various treatment steps

Particulars	Industrial effluent	Polymeric chemical coagulation (300 mg/l)	Electrocoagulation treatment (iron) $C.D = 0.5 \text{ A/dm}^2$	Electrocoagulation treatment (aluminum) $C.D = 1 \text{ A/dm}^2$	Ion-exchange process	Reuse standard
COD removal (%)	_	81.3	92.31	80	Nil	_
Conductivity (µmho/cm)	5200	4910	5320	5260	48	1
Colour removal (%)	_	98.2	96.93	87.93	Nil	10
NTU	10.6	0.8	0.9	1.3	0.2	-
Total hardness (mg/l)	36.8	28	36	39	Nil	10
Alkalinity (mg/l)	420	280	260	310	12	50
Fe concentration (mg/l)	-	-	_	_	Nil	0.1
TDS (mg/l)	3800	3680	3720	3790	90	50
S.S	130	84	72	86	0	0

Initial COD: 1040 ppm, λmax: 640 nm.



Fig. 11. The resin ratio of Amberlite (H⁺)/Amberlite (OH⁻): (\blacklozenge) 1:1, (\blacksquare) 1:2, (\land) 1:3, (X) 2:1 and (*) 3:1.

500 ml beaker. The solution was stirred using a magnetic stirrer for 10 min. Samples were collected every minute for measurements of conductivity. An optimal ratio of 1:2 the cationic and anionic resin was observed and is shown in Fig. 11. At the other ratios, the treated wastewater has a rather high conductivity and also has a low or high pH. It is of interest to note that the optimal ratio 1:2 of cationic to anionic exchange resins is much in line with that was recommended by Jiang [42]. An experimental study was carried out to determine the break-even point of ion-exchange resin columns. It is very much important to study the operational capacity of the resin and the process. Fig. 12 demonstrates the break-even point of ion-exchange resins. The experimental data indicate that 20 ml of cation-exchange resin and 40 ml of anion-exchange resins are needed to treat 400 ml



Fig. 12. Break-even point.

Table 2

Inorganic concentrations (mg/l) in the treated wastewater and deionized water

Particulars	Cl ⁻	SO_4^{2-}	NO ³⁻
Treated wastewater before ion exchange	2080	380	56
Deionized water	2.6	0.8	0.1

of treated wastewater containing 5200 μ mho/cm effectively and the treated water can be reused in the other processes. HCl (5% solution) and NaOH (4% solution) were used for the regeneration process [43]. Water quality changes after various treatment steps were presented in Table 1. Also ion exchange treated wastewater was compared with deionized water quality. Inorganic in the treated wastewater before and after ion exchange were measured by spectrophotometer, NOVA 60 and are listed in Table 2. Three major inorganic ions such as Cl⁻, NO₃⁻ and SO₄²⁻ were measured in the wastewater before and after ionexchange process. The inorganic ion concentrations measured in the treated wastewater after ion exchanges are nearly equal to those in the deionized water.

5. Conclusion

The treatment systems consisted of the chemical or electrocoagulation followed by ion-exchange process. Polymeric chemical coagulant (Sandofix-WRN) and electrochemical treatment methods were employed in the present study to investigate the effectiveness of treatment of industrial dye effluents (Procion Black 5B-Reactive dye). In the case of chemical coagulation, maximum COD reduction of about 81.3% was obtained at 300 mg/l of coagulant whereas in electrocoagulation process, maximum COD removal of about 92.31% (0.25 A/dm²) was achieved with energy consumption of about 19.29 kWh/kg of COD and 80% (1 A/dm²) COD removal was obtained with energy consumption of about 130.095 kWh/kg of COD at iron and aluminum electrodes, respectively. Increase in the current density increases the power consumption. Hence, the optimization of energy consumption of the treatment methods studied, the maximum COD removal 92.31% (0.25 A/dm²) with minimum energy consumption (19.29 kWh/kg of COD) could be achieved at electrocoagulation using iron electrode. To further elevate the water quality to the reuse standard, an ion exchange using cationic and anionic exchange resin was found necessary. Experimental results indicated that 20 ml of cation-exchange resin and 40 ml of anion-exchange resins were needed to treat 400 ml of treated wastewater containing 5200 μ mho/cm effectively. The water quality of treated wastewater was observed to be consistently excellent, comparable to that of deionized water.

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