

Electrochemical coagulation for textile effluent decolorization

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Abstract

The three most commonly used dyestuffs in textile industry are reactive, acid, and dispersed dyes. One dye from each group, C.I. Reactive Blue-19, C.I. Acid Red-266, and C.I. Disperse Yellow-218 was chosen to study the feasibility of coagulation for color removal. The dyes used in these experiments were chosen to represent the two major structural features: anthraquinone and azo dyes. Reactive Blue is an anthraquinone-based dye, and Acid Red and Disperse Yellow represent azo-based dyes. As there is no standard method to measure the color intensity, a Hach spectrophotometer was used to measure the absorbance before and after the dye solution was treated. Removal efficiencies on these dyes were obtained by measuring absorbance of a sample at 592 nm for Reactive Blue, 498 nm for Acid Red, and 428 nm for Disperse Yellow. Aluminum and ferric coagulants were produced in a parallel-plate electrochemical reactor by anodic dissolution. Removal efficiencies of more than 98%, in terms of absorbance, were observed in laboratory conditions. Removal was found highly dependent upon NaCl concentration, applied voltage, current density, and pH. The NaCl in the solution effectively reduced the power consumption and promoted the coagulant generation by depassivating the Al–water and Fe–water electrochemical systems. The processes were determined to be highly NaCl dependent. A mechanism was proposed for the corresponding liquid phase chemistry.

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1. Introduction

Textile manufacturing begins with the production or harvest of raw fiber. After the raw natural or synthetic fibers are shipped from the farm or the chemical plant, they pass through four main stages of processing: yarn production, fabric production, finishing, and fabrication. For most uses, these fabrics must undergo further processing, which includes bleaching, printing, dyeing, mechanical finishing, preshrinking, and shaping. Many different textures can also be obtained through the application of resins and sizings and the use of high temperature and pressure.

Dyes used by the textile industry are largely synthetic and are derived from coal tar and petroleum-based intermediates. The primary classification of dyes is based on the fibers to

which they can be applied, and the chemical nature of each dye determines the fibers for which the dye has affinity. Acid dyes are water-soluble anionic compounds applied to nylon, wool, silk, and some modified acrylic textiles in an acidic medium. They have one or more sulfonic or carboxylic acid groups in their molecular structure. The dye-fiber affinity is the result of ionic bonds between the sulfonic acid part of the dye and the basic amino groups in wool, silk, and nylon fibers.

Reactive dyes are water-soluble, anionic dyes that require relatively simple dyeing methods. They are mainly used for dyeing cellulosic fibers, such as cotton and rayon, but are also used for silk, wool, nylon, and leather. Reactive dyes have largely replaced direct, azoic, and vat dyes and are the largest dye class in the United States. The dyes form covalent bonds with the fiber and become part of the fiber. To exhaust the dyes, large amounts of salt are generally essential, and significant amounts of dye can remain unfixed at end of the process.

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Disperse dyes have a very low water solubility, so they are applied as a dispersion of finely ground powders in the dye bath. The dyes dissolve at low concentration in the aqueous dyeing bath but transfer into the synthetic fiber polymer because of their higher solubility in the substrate. High temperatures and pressures are consistently used for application. Disperse dyes are used for oleophilic fibers and polyester and other synthetics that do not accept water-soluble dyes.

In general, wastewater treatment methodologies in the textile industry are mature and well developed. The industry and its regulators have focused on improving the efficiency of existing treatment processes for some time. The textile industry is concerned that color residues in dyeing wastewater might not be amenable to treatment with existing treatment systems. Technologies to remove color from textile effluents continue to be an active area of research and development, pursued actively in the United States and abroad.

2. Literature review

Decomposition of chromophores in organic dyes can be achieved by injecting a strong oxidizing agent, such as ozone (O_3) and hydrogen peroxide (H_2O_2), to the industrial effluents [1–3]. Very high color removal and significant amount of chemical oxygen demand (COD) reduction can be achieved with ozone application. The process also increases the aerobic biodegradability of the dyes in the wastewater. However, a large dose of ozone is usually required for decolorization due to the significant amount of auxiliary chemicals, such as acetic acid and silicone oil in the dye bath. Little color removal is achieved by H_2O_2 alone or even with ferrous ions as catalyst, known as Fenton's reagent. Current research has been focused on combining H_2O_2 with UV or ozone to generate hydroxyl radicals (HO^\bullet) to oxidize various organic chemicals [4,5]. These processes are known in the literature as the advanced oxidation processes (AOP). The oxidation of organic dyes with AOP usually produces oxygenated organic compounds and low molecular weight acids that are more biodegradable.

Treatment with strong reducing agents, such as sodium hydrosulfite, sodium borohydride, and sodium dithionite, has been shown to significantly reduce color in textile effluent [6]. These compounds chemically break the nitrogen–nitrogen double bond of azo dyes and produce lower molecular weight aromatic amines. A small number of aromatic amines are classified as being carcinogenic or potentially carcinogenic to humans. Those azo dyes that can release harmful aromatic amines upon reductive cleavage of azo group will soon be banned in European market.

Adsorption processes have been applied for concentrating organic compounds from industrial effluents for years. The most widely used adsorbent is granular activated carbon (GAC). GAC has been proven to be effective for removal of color from textile and pulp and paper effluents. Due to the high capital, regeneration, and disposal costs, full scale GAC

adsorption systems for effluent decolorization have yet to be accepted by the industry. Cucurbituril, a cyclic hexamer with an internal hydrophobic cavity, has been studied for removal of reactive dyes [7]. An adsorption capacity of 1 g/g makes this material a candidate for further development. However, high pH and salt concentrations may have an adverse effect on the adsorption.

Aerobic treatment of industrial effluents typically removes most of the biochemical oxygen demand (BOD) and 60–80% of COD in wastewater [2,8,9]. However, these treatments are ineffective for color removal from textile industry wastewater because most of the synthetic dyes are non-biodegradable. In anaerobic processes, the azo bonds can be used as electron acceptors in the electron transport pathways. The products of the azo bond cleavage are lower molecular weight aromatic amines. Although they are colorless, they cannot be further degraded under anaerobic conditions. Therefore, various combined processes, such as anaerobic/aerobic [8], chemical/biological [10], and electrochemical/biological [11], processes have been under intensive study to completely mineralize organic dyes. All the pretreatments show potential for increasing the biodegradability of textile effluents for further biological treatment. However, the presence of salt, sulfur compounds, heavy metals, and other toxic compounds often inhibits the anaerobic microbial population and increases chemical and power consumption in these pretreatment processes.

Among electrochemical treatments, electrochemical oxidation and electrochemical coagulation are more effective than others in decolorizing textile effluents. Although some electrochemical processes involve the direct oxidation of pollutants at anode surfaces, most of the others involve production of active species to react with target pollutants. The rate of electrochemical oxidation depends on the type of electrolyte, electrolyte concentration, reaction temperature, and current density [12]. Another promising electrochemical process is by coagulation [13]. Aluminum and iron sheets have been used as consumable anodes to generate coagulants that adsorb and remove organic dyes. An aluminum coagulant removes colorant by simple adsorption without involving chemical reactions while an iron-based coagulant provides ferrous ions (Fe^{2+}) to further degrade the dyes [14]. Likewise, the iron process seems more effective for removal of reactive dyes while the aluminum process is superior for removal of disperse dyes [15].

Most of the ongoing development programs address an approach of extending the capacity of existing processes for dye decomposition or removal. A thorough cost estimation has been done on a wastewater treatment system for a flow rate of 100,000 gallons per day [16]. Table 1 details the result of that study. Operating costs of ozone treatment are higher than that of electrochemical treatment for the same level of color removal. Chemical reduction is only able to remove a portion of the color but at a competitive cost. A GAC system can remove both color and COD only at an extremely high operating cost. The results indicate that electrochemical

Table 1
Color and COD removal efficiencies and operating costs of available technologies

Technology	Color removal (%)	COD removal	Operating cost (US\$/year)	Capital cost (US\$/year)
Electrochemical oxidation	97.5	95.1%	US\$ 1,066,000	ND
Ozone (chemical oxidation)	97.6	55.9%	US\$ 2,030,000	US\$ 700,000
ColorClear (chemical reduction)	76.8	Chemical interference	US\$ 566,000	US\$ 23,700
GAC (activated carbon)	86.1	70.9%	US\$ 6,813,000	US\$ 243,000
Fenton's reagent (chemical oxidation)	25.4	ND	ND	ND
Ferric sulfate (chemical coagulation)	0.0	ND	ND	ND

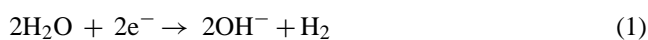
ND, not determined.

treatment is the most efficient process in terms of operating costs for the complete removal of both color and COD. An electrochemical process that removes color merely by applying low voltage direct current to a waste effluent could have a significant impact on control strategies.

3. Process chemistry

When wastewater is treated with direct current, a number of physicochemical processes take place. These are oxidation and reduction of impurities in wastewater, dissolution of metallic anodes, discharge, and coagulation of colloidal particles, electrophoresis, electroflotation of particles, precipitation of metallic ions on the cathodes, split salts, concentration of acids and alkalis, and water desalination.

The main pathway for removing colorant from aqueous solutions is coagulation. At large cathodic potentials, local hydroxyl ions (OH^-) can be generated due to water and oxygen reduction:



With cast iron sheets as consumable electrodes, ferrous (Fe^{2+}) and ferric (Fe^{3+}) ions are produced on the anodes.

Anode:



Cathode:



With aluminum sheets as consumable electrodes, Al^{3+} ions are produced on the anodes.

Anode:



Cathode (same as Eq. (5)):



During the process, an increase in pH is expected. As a result of the high pH, precipitates of $\text{Fe}(\text{OH})_2$, $\text{Fe}(\text{OH})_3$, and $\text{Al}(\text{OH})_3$ are formed and remain in the solution. The gelatinous suspension removes colorants by complexation and electrostatic attraction followed by coagulation.

4. Experimental

The study of freshly generated coagulant for color removal was conducted in a bench-scale electrochemical system, as shown in Fig. 1. The system consists of a dc power supply, a power control and measurement units, an electrochemical reactor, a reservoir, a feed pump, a flow control valve, a flow measuring unit, and a circulation pump. The reactor is made of PVC plastic with an active volume of 88 cm^3 . The anode is made of either aluminum or cast iron sheets and the cathode is made of graphite. The electrodes are situated approximately 1 cm apart from each other and submerged in the solution. The cathode and the anode each have a surface area of 88 cm^2 . The system was operated in a simple batch recirculation mode with a circulation pump to prevent the sedimentation of the coagulant.

Azo and anthraquinone colorants are the two major classes of synthetic dyes and pigments. Together they represent about 90% of all organic colorants. Based on their applications, organic dyes can be subclassified as acid, azoic, basic, direct, disperse, reactive, mordant, sulfur, and vat dyes. Reactive dyes have largely replaced direct, azoic, and vat dyes and are the largest dye class in the United States. Fig. 2 shows the anthraquinone structure of Reactive Blue-19 and Fig. 3 shows the azo structure of Acid Red-266. Disperse Yellow-218 has an unknown azo-based chemical structure. The dyes used in this study were chosen to represent the variety of organic dyes used in textile industry. A UV-vis range spectrophotometer was used to determine the wavelength of maximum absorbance (λ_{max}) for each dye. Color intensity was determined by measuring the absorbance of a sample at 592 nm for Reactive Blue-19, 498 nm for Acid Red-266, and 428 nm for Disperse Yellow-218. The data were used to calculate removal efficiency (x). The percent removal is written as

$$x (\%) = \frac{A_0 - A_t}{A_0} \times 100\% \quad (7)$$

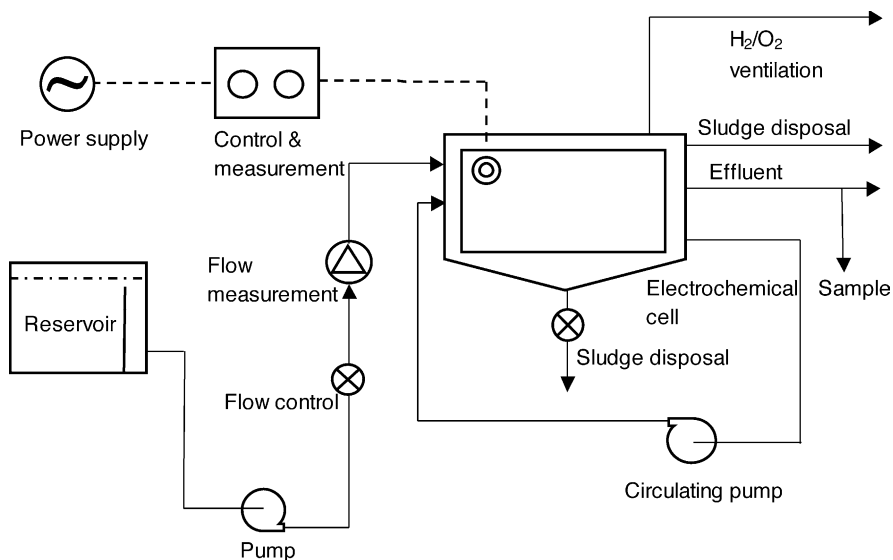


Fig. 1. Schematic of the electrochemical coagulation system.

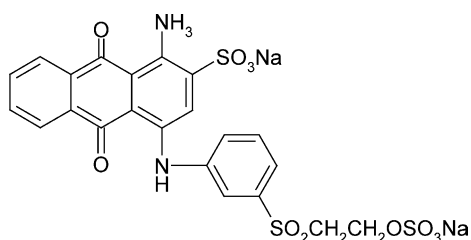


Fig. 2. Chemical structure of Reactive Blue-19.

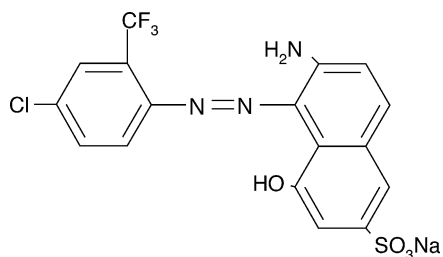


Fig. 3. Chemical structure of Acid Red-266.

5. Results and discussion

This study was divided into an Fe-coagulation process and an Al-coagulation process. For the Fe-coagulation process, iron sheet was used as consumable anode to generate ferric and/or ferrous ions. These ions precipitate out as hydroxides (Fe-coagulant) with OH^- anions that are generated on the cathode through water electrolysis. Aluminum sheet was used as a consumable anode to generate the Al-coagulant in the Al-coagulation process. Sodium chloride (NaCl) was added to provide extra electrical conductivity in the solution and to promote the production of coagulant. Parameters, such as voltage and NaCl concentration, were studied for their effect on color removal. Current, color intensity, and system pH

were monitored during the experiment. The results are given in this section.

5.1. Fe-coagulation process

Due to the generation of OH^- on cathode surfaces, the pH in the system is expected to increase with treatment time. When the pH increases to a certain level, coagulant will begin to accumulate in the solution. A portion of the colorant will be decomposed by the formation of Fe^{2+} and/or Fe^{3+} from the anode. The majority of color was removed by adsorption and complexation, however. Fig. 4 shows the characteristics of the Fe-coagulation process for organic dye removal. Two grams of Reactive Blue-19 along with 2 g of sodium chloride were used to prepare 1 L of synthetic dyebath for the test. A graphite sheet was used as the cathode to pair up with the iron anode. Initially, the dyebath had an absorbance of 1.6 absorbance units (AU) at 592 nm, which is the λ_{max} of Reactive Blue-19. The solution was pumped into the electrochemical reactor where 10 V dc was applied across the 1 cm

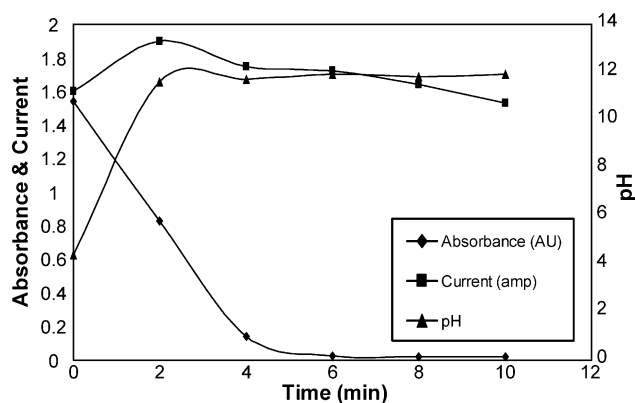


Fig. 4. Characteristics of Fe-coagulation process for color removal.

gap. With 2 g/L NaCl in the solution, the applied voltage resulted in a current of 1.6 A through the reactor. The current fluctuated a bit and stabilized at around 1.6 A throughout the 10-min treatment. During the experiment, the pH of the solution increased from 4.2 to 11 in 2 min and remained at 11 throughout the treatment. The solution turned dark brown in the first minute and a significant amount of coagulant was observed after 4 min. Samples were withdrawn from the reactor every 2 min. After a 5-min centrifugation, a measure of color intensity was taken from the spectrophotometer. The freshly generated Fe-coagulant removed 50% of the color in 2 min and more than 95% by 6 min. The sludge on the bottom of test tubes turned from dark blue-brown to light brown in 1 week of storage. It is believed that the dye molecules were first adsorbed on the surface of the Fe-coagulant and were broken down through time.

Fig. 5 shows the effect of applied voltage on color removal. With 10 V dc and 1 g/L NaCl, it took 2 min to accumulate enough coagulant to start reducing the color intensity. Ultimately, the system was able to remove the color to below the detection limit of the spectrophotometer. With 5 V applied to the reactor, the system was unable to generate coagulant for color reduction. It was found that a minimum current was carried through the reactor at that voltage. In order to increase the current density in the reactor, NaCl concentration in the synthetic dyebath was increased to 2 g/L. Fig. 6 shows the color removal with the extra NaCl in the solution. A vigorous reaction was observed in the reactor when 10 V dc was applied to the solution containing 2 g/L of NaCl. Color removal started immediately and continued until all the color was removed. However, the excessive amount of coagulant generated by the process ended up with the sludge deposited on the bottom of the reactor. Centrifugation is an effective method to separate the sludge from the solution. For the process to be applied to an industrial scale, a sludge separation mechanism needs to be considered and examined thoroughly.

An electrochemical reactor can be operated under constant voltage or constant current. Theoretically, reaction rate is proportional to current density. Applied voltage and NaCl concentration both contribute to the current density in the

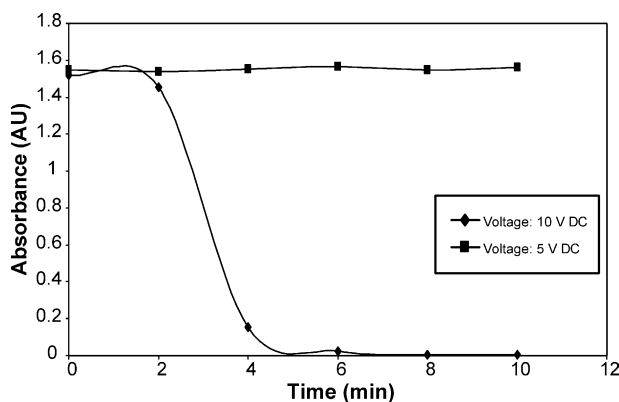


Fig. 5. Effect of applied voltage on color removal in Fe-coagulation process.

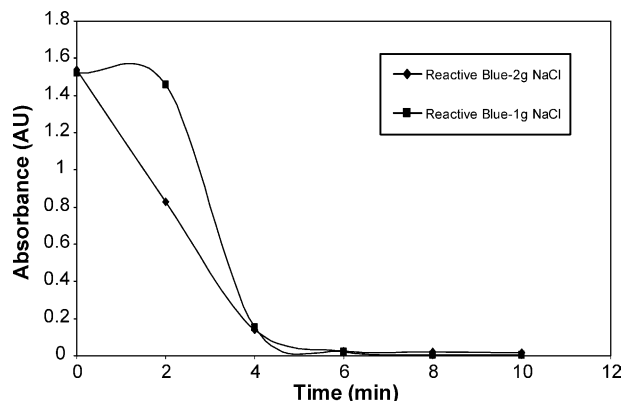


Fig. 6. Effect of salt concentration on color removal in Fe-coagulation process.

reactor. Fig. 7 shows the current carried through the reactor under different combinations of NaCl concentration and applied voltage. At a concentration of 2 g/L, 10 V dc was able to draw a steady current of 1.6 A across the reactor throughout the 10-min treatment time. Under the same concentration, only 0.5 A was carried through the reactor with a voltage of 5 V. With 1 g/L NaCl in the solution 10 V dc will initially draw a current of 1.6 A through the reactor. The reaction was vigorous and a significant amount of coagulant was generated. During the operation chloride (Cl^-) was consumed to produce chlorine gas (Cl_2) on the surface of the anode. Some Cl_2 evolved from the surface as tiny bubbles while the majority of Cl_2 dissolved and equilibrated with hypochlorous acid (HClO), trichloride ions (Cl_3^-), and hypochlorite (ClO^-) in the solution. After 2 min of operation, the solution began to lose its electrical conductivity and showed a reduction of current through the reactor. Fig. 8 shows the corresponding color removal under these conditions. With high salt (NaCl) concentration and high voltage, the system started to remove color at the moment power was applied and completely removed the color in 6 min. It took 2 min to initiate the low salt process. By the end of the treatment, the low salt process reached the same level of color removal in the same

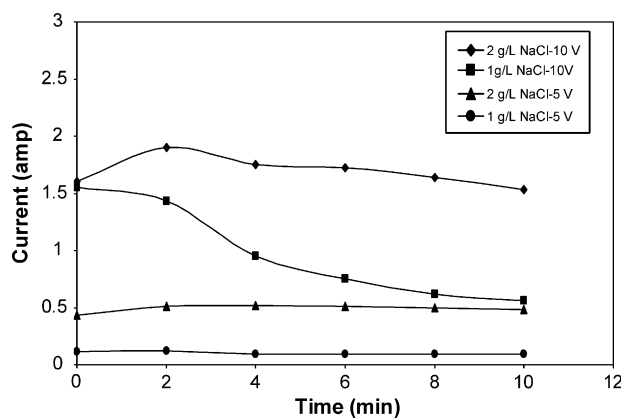


Fig. 7. Current as a function of salt concentration and applied voltage in Fe-coagulation process.

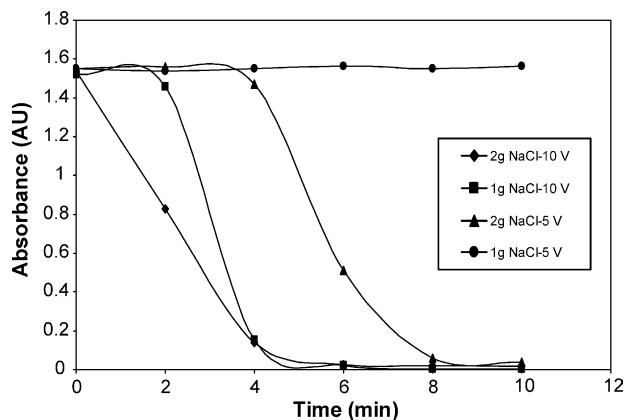


Fig. 8. Effect of current on color removal in Fe-coagulation process.

treatment time. As can be expected, the process efficiency, in terms of color removal and treatment time, is closely related to the current through the reactor. At a low salt concentration and low voltage, low current was carried through the reactor and no color removed in a 10-min treatment.

In viewing the success of the electrochemical coagulation for reactive dye removal, it must be determined how widely this technology is applicable to other textile dyes. Acid Red-266 and Disperse Yellow-218 were chosen to test the process. Different from Reactive Blue, both Acid Red-266 and Disperse Yellow-218 are azo dyes. Usually azo dyes are more accessible to chemical reduction and reactive dyes are more easily broken down by chemical oxidation. Due to low solubility, disperse dyes rarely participate in aqueous phase chemical reactions. Because electrochemical treatment provides oxidation, reduction, adsorption, coagulation, and flotation in one reactor, one would expect that the treatment might offer an universal process for decolorizing a matrix of textile dyes. Solutions of all three dyes were prepared at a concentration of 2 g/L with a same concentration of NaCl to provide the needed conductivity. Initial absorbance of these solutions was between 1.5 and 1.8 AU at each λ_{\max} . These solutions were treated in the electrochemical reactor under 10 V dc for 10 min. Fig. 9 shows the removal efficiencies of the electrochemical coagulation on the three dyes. It took 6 min to complete the treatment for all three dyes. Residual color was always observed in the Disperse Yellow dyebath. Still, 90% removal is achievable. It is believed that the mechanism for disperse dye removal is adsorption/complexation followed by flotation. The reactor is designed to optimize the generation of coagulant. Therefore, a modification is needed to improve flotation thus disperse dye removal.

5.2. Al-coagulation process

In terms of chemical coagulation, aluminum ions are in most cases more effective than ferric or ferrous ions. It became apparent that freshly generated Al-coagulant must be evaluated for color removal. An aluminum sheet was used to replace cast iron as the consumable anode. The same pro-

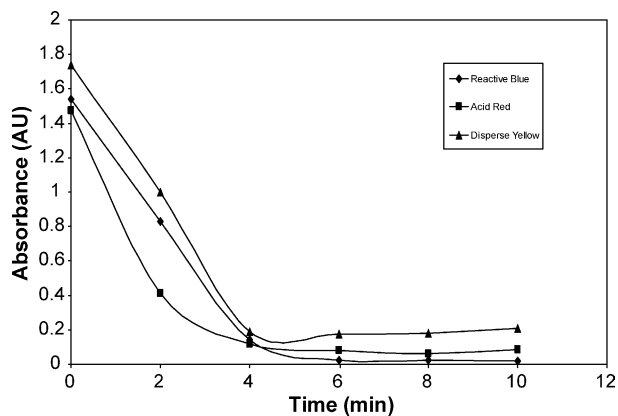


Fig. 9. Application of Fe-coagulation process on reactive, acid, and disperse dyes.

cedure from the previous section was followed to yield the results for comparison. Again, Reactive Blue-19 was chosen for a thorough evaluation. Fig. 10 shows the effect of applied voltage on color removal. Five-volt dc caused minimum current through the reactor, which resulted in no color removal. With 1 g/L NaCl in the solution 10 V dc created significant current, resulting in enough Al-coagulant for color removal. Color removal reached a maximum of 75% but the color was restored at the end of the 6-min treatment time. Fig. 11 shows the same trend with 10 V dc applied to the reactor. At a NaCl concentration of 2 g/L, the applied voltage caused a vigorous reaction in the reactor thus a significant color reduction. The color was completely removed in 6 min but the solution regained its color at the end of the 10-min treatment. The synthetic dyebath was prepared with an initial pH of 4.2. During the treatment, the pH quickly passed through 7 and increased to 11. Due to amphotism, aluminum hydroxide dissolves at high and low pH and has a lowest solubility at pH 6.2. When the pH reaches 6, most of the aluminum hydroxide precipitate out as Al-coagulant and adsorb the colorant. When the pH reaches 9, some of the Al-coagulant will dissolve and restore a portion of the adsorbed colorant into the solution. The restored color had an observable turbidity that did not

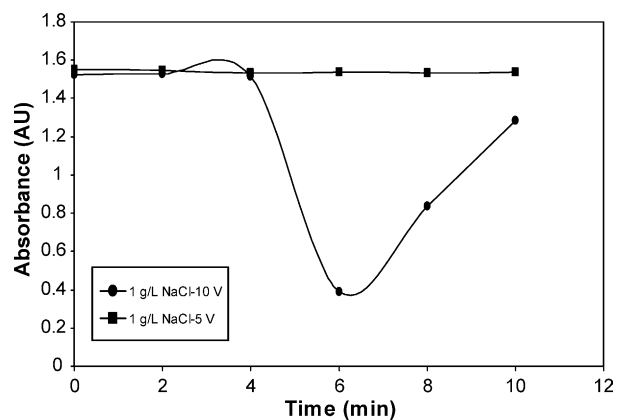


Fig. 10. Effect of applied voltage on color removal in Al-coagulation process.

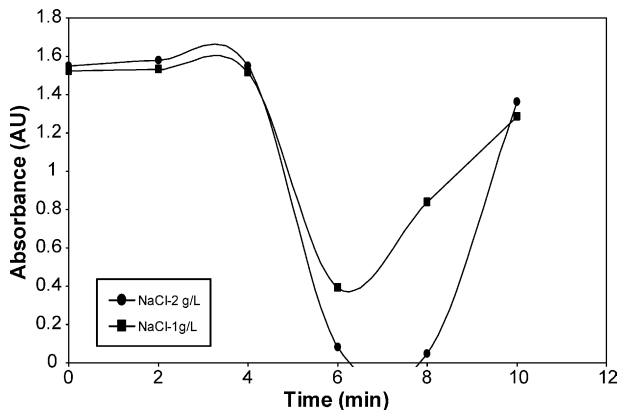


Fig. 11. Effect of salt concentration on color removal in Al-coagulation process.

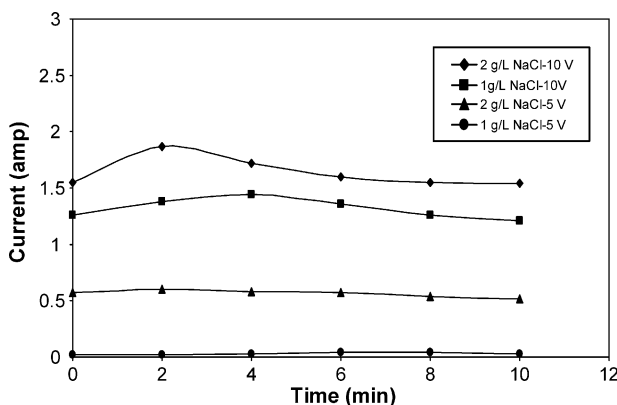


Fig. 12. Current as a function of salt concentration and applied voltage in Al-coagulation process.

settle after 5 min of centrifugation. It is believed that the turbidity is caused by the gelatin-like aluminum hydroxide that dissolves in the high pH aqueous solution.

Fig. 12 shows the stable currents in the reactor at different combinations of salt content and applied voltage, and Fig. 13

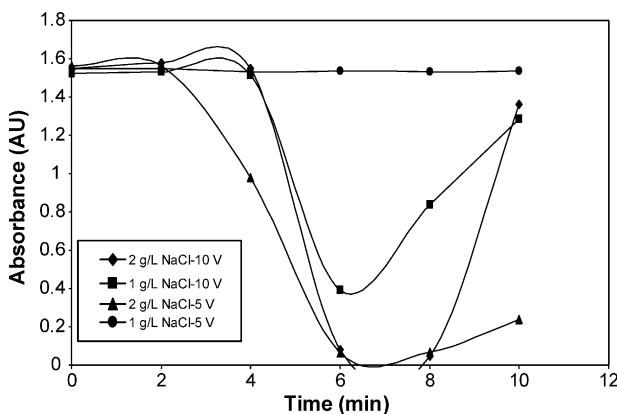


Fig. 13. Effect of current on color removal in Al-coagulation process.

shows the corresponding color removal for each treatment. It can be seen from Fig. 13 that high salt content and low applied voltage produces optimum conditions for color removal. High salt content favors the color removal and the low current density delays the system from color restoration through Al-coagulant dissolution. After treatment, acids can be added to the effluent to bring the pH from 11 down to around 6 or 7. This will stabilize the Al-coagulant and thus prevent the treated solution from color restoration. NaCl is commonly used in dyebath to promote dye exhaustion. The residual NaCl can be used in electrochemical treatment of spent dyebath for color removal as well. During the operation, NaCl is oxidized to chlorine gas on the anode surfaces. The chlorine will dissolve in water to form HClO and ClO⁻. These are strong oxidizing agents and can be used to further oxidize the dye molecules that are adsorbed on the coagulant. These species are widely used disinfectants. Discharge of these chemicals into a wastewater treatment plant may disrupt its biological processes.

A set of experiments was conducted to explore the applicability of Al-coagulant for decolorization of different dyebath. Solutions of Reactive Blue-19, Acid Red-266, and Disperse Yellow-218 were prepared with the procedure stated in the previous section. Two grams of NaCl were added to 1 L of the solution to provide the essential electrical conductivity. At 5 V dc, a significant amount of Al-coagulant quickly accumulated in the reactor followed by color reduction. Fig. 14 shows the removal of each dye under this treatment. Due to low water solubility, disperse dyes are difficult to break down by chemical oxidation or reduction. Al-coagulation removed color to a level that was beyond the detection limit of the spectrophotometer. Because of the low solubility of the disperse dyes, carriers are used to stabilize the dispersing system and keep the dye powders in an emulsified solution. The addition of polyvalent aluminum ions will neutralize the colloids and destabilize the dispersing system thus allowing the colorant to attach to the Al-coagulant and be removed from the solution.

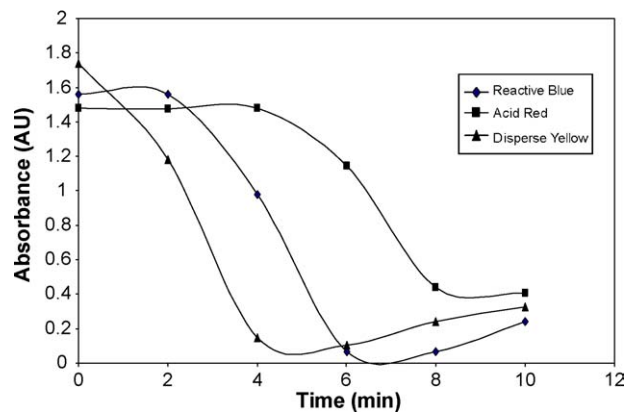


Fig. 14. Application of Al-coagulation process on reactive, acid, and disperse dyes.

6. Conclusions

The Fe-coagulation process is effective for color removal from textile effluents. The associated sludge must be dealt with. The by-product of the process, NaClO, is a strong oxidizing agent that can be used to further decompose the dye structure. Sodium chloride is essential in this process because the existence of NaCl reduces power consumption, depassivates the electrochemical system, and forms NaClO for dye decomposition. The decomposition of adsorbed dye molecules might be caused by the existence of ferrous ions or NaClO. The Al-coagulation process is effective for removing reactive, acid, and disperse dyes from a synthetic dyebath, especially disperse dyes. The removal is due to the demulsification by aluminum ions and adsorption of dye molecules on the freshly generated coagulant. The solution restore its color after an excessive treatment, because the formation of OH⁻ on the cathode increases the solution pH, ultimately dissolving the coagulant and restoring dye molecules to the solution. Due to amphotism, aluminum hydroxide dissolves at high and low pH and has a lowest solubility at pH 6.2. To optimize the Al-coagulation process for colorant removal, pH control or adjustment is essential.

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