



Reuse and Recovery of Water from Industrial Textile Dyeing Effluent Using High-Performance Electrodes Continuous Flow Electrocoagulation Reactor

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ABSTRACT

The dye effluents released from the textile and printing industries contain strong colorants, inorganic salts, and other toxic compounds. The conventional coagulation technique of dye effluent treatment is plagued with issues of low removal rate of color, generation of large quantities of sludge, and toxic end-products. Recently electrocoagulation technique gained immense attention due to its high efficiency. This technique involves the dissolution of the sacrificial anodes to provide an active metal hydroxide as a strong coagulant that destabilizes the pollutants and removes them by precipitation or flocculation. This study is about the efficiency of the electrocoagulation process using titanium coated - aluminum and mild steel electrodes to treat industrial dye wastewater. Effects of parameters such as current density & initial dye concentration were investigated. It was observed that, for the same current density, electrode consumption was higher with TiO_2/Al electrode than with mild steel electrode, resulting in more color removal efficiency (CRE) using TiO_2/Al electrode. The settling rate of the flocs was higher in the reactor having TiO_2/Al electrode at the 100 mL with current density ($2.5 \text{ mL}\cdot\text{min}^{-1}$ to $5.3 \text{ mL}\cdot\text{min}^{-1}$), while in the reactor with mild steel electrode, the settling rate was very less. The results showed that dye removal was 95.11% and 92.1% for mild steel and titanium-coated electrodes, respectively. It was observed that 50 % of Aluminum was removed from the treated effluent after the final stage of filtration. Based on the multicriteria analysis to identify the optimum operational parameters to be applied at the field level, it was observed that maximum CRE may be obtained with TiO_2/Al electrode and the applied current of 1 Amps with a flow rate of $100 \text{ mL}\cdot\text{min}^{-1}$. It can be concluded that electrocoagulation is a highly efficient and the fastest method to treat dye effluents from industries.

INTRODUCTION

In recent days, synthetic dyes have been widely used in leather, textile, paper, food, cosmetics, and pharma industries. Wastewater, which is the outcome of dyeing and washing processes from textile industries, is of special concern due to the wide range of chemicals used in the corresponding process and its related toxic effects. It is estimated that about 15% of the dyestuff is left unused and is discharged as waste.

Due to the expansion of industrial activities and increased population growth, environmental degradation is intensified, as visually seen by the deterioration of land and water ecosystems. Nature, which was once tamed by anthropogenic activities, has started rebounding its action by posing a great challenge in the treatment of water and wastewater to researchers, engineers, and scientists. It is reported that 2,500 textile weaving factories and 4,135 textile finishing factories are functioning in India. Out of

60% of total dye production for various applications, 10-15% of unspent dyes are let out into water bodies which makes the water polluted with a concentration range of 10-200 ppm. Though conventional methods like reverse osmosis, ion exchange, membrane filtration, adsorption, electrocoagulation, and chemical precipitation are widely used, adsorption is an attractive, efficient, and cost-effective method with less maintenance cost that is commonly applied to remove Total Dissolved Solids (TDS), organic, inorganic and heavy metals.

In the physical treatment method, the dyestuffs in wastewater are removed by using naturally occurring forces, such as gravity, electrical attraction, and van der Waal forces, as well as by the use of physical barriers. Physical treatment methods include adsorption, membrane filtration, and coagulation. On the other hand, chemical treatment processes include, oxidative process, ozonation, chemical coagulation, etc. In the chemical coagulation process, coagulants with

charges opposite to those of the suspended solids are added to wastewater to neutralize the negative charges on dispersed non-settable solids such as organic substances. When the charge is neutralized, the small suspended particles stick together to form flocs that settle down over time. The major disadvantage of the chemical coagulation method is that it transfers toxic compounds into the sample, which has to be treated subsequently, and it involves high costs and is time-consuming. Because of these reasons, another alternate method that overlays these disadvantages has to be developed. The conventional methods of wastewater treatment demand more area, consume more time, cause sludge disposal problems, energy-intensive and demand-based.

Electrochemical Methods

Electrochemical methods gained attention during the second part of the 20th century. Though the investment and current consumption were high at the time of the introduction of the electrochemical method, after extensive research on electrochemical methods, the cost of electrochemical methods is comparable with other wastewater treatment techniques (Vik 1984, Stuart 1946, Bonilla 1947). In specific applications, the electrochemical methods are more efficient than conventional methods of treatment. The main disadvantage of using the chemical coagulation method is, due to the addition of chemicals like alum and ferric chloride, additional anions get into the effluents by which the TDS increases in concentration more than that of the electrochemical method where the pure metal electrodes (Al, Fe, Ti, etc.) are used as sacrificial anodes. Electrocoagulation is a water treatment technique recently discovered that is used to remove the total suspended solids (TSS), heavy metals (Mn, Cu, Zn, Ni, Al, Fe, Co.), emulsified oils, bacteria, and other contaminants from the wastewater (Heidmann et al. 2010, Abdelwahab et al. 2009). Electrocoagulation consists of a pair of metal sheets called electrodes. In this process, the electro-dissolution of sacrificial anodes, usually made of aluminum or iron, to the wastewater leads to the formation of hydrolysis products (hydroxo-metal species) that are effective in the destabilization of pollutants. The hydroxo-metal species forms flocs that trap the pollutants in the wastewater (Zongo et al. 2009, Balasubramaniam et al. 2009, Azarian et al. 2007).

Electrocoagulation

In electrocoagulation, the coagulant/metal ions/destabilizing agents are generated in situ through electrolytic redox reactions at the anode and cathode in the electrochemical reactor that neutralize the charged colloids in the solution.

The sacrificial anodes get corroded due to the oxidation process. The generated electrons move towards the cathode that dissociates water into hydrogen gas and hydroxide ions. The metal ions generated from the anode get hydrolyzed in the solution to form various monomeric hydroxide species based on the pH of the solution. In the case of Al electrodes, in the alkaline environment (at pH>9), the final pH of the treated effluent is reduced because of the formation of aluminate ($\text{Al}(\text{OH})_4^-$) which consumes alkalinity. When the metal ions with high charge are released from anodes, polyvalent poly-hydroxide complexes (Al-O-Al-OH)/gelatinous charged hydroxy cationic complexes with high adsorption capacity are formed that will adsorb the colloids/pollutants. In the case of Fe electrodes, the $\text{Fe}(\text{OH})_n$ species in the gelatinous form remove the colloids/pollutants either through electrostatic force of attraction or through complexation (in which the colloids/pollutants act as a Ligand to bind the hydroxyl ion. In an acidic environment and the presence of oxygen, Ferric ions are formed that lead to the generation of amorphous $\text{Fe}(\text{OH})_3$ species with large surface area, which are beneficial in the adsorption of organic and inorganic pollutants. At higher pH, the higher forms of hydroxide species $\text{Fe}(\text{OH})_4^-$ and $\text{Fe}(\text{OH})_6^-$ are formed. During the electrocoagulation process, simultaneous generation of O_2 at the anode and H_2 at the cathode are released due to the decomposition of water. In addition to that, highly reactive OH^\cdot and HO_2^\cdot radicals are formed (Morena et al. 2007, Picard et al. 2000)

Electrocoagulation is a cost-effective and efficient technology that has been widely applied in the area of textile dye removal, treating wastewater containing organic compounds like fats, oils, detergents, heavy meals, microorganisms, other anions and cations, etc.

The type of electrochemical reactions and the type of actions released into the solution depends on the type of electrode materials used. In the case of Al electrodes, the metal ions are released in the form of Al^{3+} , and Fe gets dissolved in the form of Fe (II) and Fe (III). The performance of Fe (III) ions is better in the removal of pollutants because of the higher positive charge and lower solubility of hydroxides (Linares et al. 2009, Krishna et al. 2010, Katal et al. 2011). Based on the type of pollutants to be removed and the size of the cations, the performance of Al and Fe electrodes differ. The size of Fe^{3+} ions (10-30 micrometer) is bigger than Al^{3+} ions (0.01-1 micrometer). The bigger the size of the metal ions, the higher the removal of colloids/pollutants. From past works, it was observed that the Fe electrodes are more efficient in odor removal. In most of the studies, Fe, Stainless steel, Al, Cu, and Ni electrodes have been used as the sacrificial electrodes (Alil et al. 2012, Marconato et al. 1998, Secula et al. 2012).

In this study, the TiO_2/Al electrodes are used as an anode and cathode for the *in situ* generation of Ti^{4+} metal ions, which have a tetravalent charge with better dye removal efficiency than Al^{3+} or Fe^{3+} ions. The Ti^{4+} ions will be released till the coating/ thermally deposited Ti^{4+} gets dissolved into the solution. Then, after, Al^{3+} ions may start getting dissolved. This type of nano-composite electrode takes advantage of both metal ions in removing the dye from the effluent. The main reactions occurring in the reactor are: when the potential is applied to the metallic anode, the metal ions (Ti^{4+} , Al^{3+}) get dissolved into the solution, while an equivalent number of electrons pass through the circuit and dissociate water into H_2 (g) and OH^- ions at the cathode. The hydrogen gas is released at the cathode, while oxygen is released at the anode. In the presence of supporting electrolyte and with slow stirring, the electro-driven coagulants and hydroxyl ions come into contact to form both monomeric, polymeric metal hydroxides and poly-hydroxy metallic flocs like $\text{Al}_{13}(\text{OH})_{34}^{5+}$, $\text{Al}_6(\text{OH})_{15}^{3+}$, $\text{Al}_8(\text{OH})_{20}^{4+}$, $\text{Al}_{13}\text{O}_4(\text{OH})_{24}^{7+}$, $\text{Al}_7(\text{OH})_{17}^{4+}$, which finally get transformed to $\text{Al}(\text{OH})_3$ based on precipitation kinetics, that adsorb/entrap the dye/ pollutants from the waste stream. Due to the formation of OH^- (aq) and consumption of protons at low initial pH, Al ions get dissolved from the cathode to form aluminate ($\text{Al}(\text{OH})_4^-$) which increases the pH of the effluent in the reactor. Capacitive Deionization is one of the efficient

electro-adsorption techniques for removing salt from aqueous solutions. When the electrodes are charged, the oppositely charged ions get adsorbed into the electrical double layer region on the surface of the respective electrodes (anions to anode and cations to cathode), thereby the concentration of anions and cations contributing to the increase in TDS gets removed. Then, during depolarization, due to a reduction in the cell voltage, the adsorbed ions get released into a waste stream (Mahvi et al. 2009, Canizares et al. 2009, Ahmed et al., 2016).

The generated hydroxyl species and generated metal ions affect the conductivity of the solution. When the initial pH of the wastewater to be treated is in the acidic medium, during electrocoagulation, due to the interaction between metal ions and other anions present in the wastewater as well as the formation of hydroxyl species near the anode. Also, the evolution of H_2 gas at the cathode increases the pH of the solution. But when the initial pH of the wastewater is around 2, then there will not be a significant increase in the pH of the solution (Abhinesh Kumar et al. 2016). At $\text{pH} > 4$, the alkalinity of the medium gradually rises.

The current intensity greatly influences the voltage between the anode and the cathode. The economics and amount of in-situ generation of metal ions from the anode depend on the power consumption/current density (Danis et

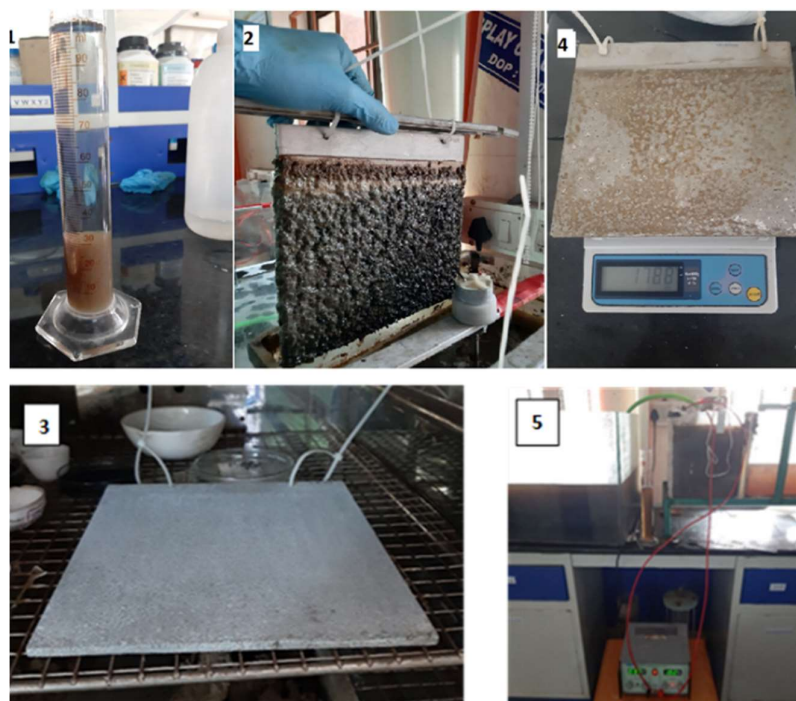


Fig. 1. Settling of sludge 2. Anode scrap 3. Weight measurement of the electrode 4. Weight measurement of cathode 5. Continuous flow reactor.

al. 2017, MohdKhairu et al. 2016). The actual dissolution of the metal ions from the anode for the applied current is compared with the calculation using Faraday's law. In an acidic or neutral medium, the dissolution from Al and Fe electrodes is primary, whereas in the alkaline medium, the actual rate of dissolution of Fe is less than the theoretical value (calculated through Faraday's law). The applied current greatly affects the size of the reactor and also the efficiency. At higher currents, the reactor size may be small. But when a high current (more than optimum) is applied, the performance is poor since most of the electrical energy is converted into heat energy, which raises the temperature of the treated effluent (Ming et al. 2012, Honore et al. 2017, Chafi et al. 2011).

MATERIALS AND METHODS

Continuous Flow Studies

The electrocoagulation reaction tank was initially filled with the dye effluent to be treated. The electrodes were labeled and kept inside the reaction tank. A DC power supply meter is used to pass current onto the electrodes. The TDS of the solution was kept at 2000-2500 mg.L⁻¹ concentration.

Initially, 1 Amp (25 A.m⁻²) of current was applied to the electrodes to achieve the formation of the floc. Later, the

experiment was run by varying the current and the inflow rate of the dye effluent from the storage tank to the reaction tank. Once the flocs were formed, the settling rate of the flocs was measured. When the dense flocs are formed, the continuous flow is maintained from the inlet tank. The quality of the effluent from the reaction tank, settling tank, and filter tank for every one-hour interval. The collected samples were first filtered using filter paper, and the filtrate was used for further analysis (Fig. 1 & Fig. 2).

The samples were tested for pH, EC, TDS, COD, absorbance, and settling rate. The flow rate and current were changed for every 4th or 5th hour. Before varying the flow rate and current, the sludge deposited on the electrode was scrapped and kept in the oven for drying. The electrode plates were then washed and dried before placing it in the reaction tank. This was repeated every time the flow rate and current were changed. The experiment was run with different flow rates, currents, and electrolyte concentrations to find the optimum experimental condition to attain maximum color removal efficiency.

RESULTS AND DISCUSSION

The samples collected from the reaction tank, settling tank, and filter tank were analyzed for pH, EC, TDS, Colour removal efficiency (CRE), and settling rate of the flocs.

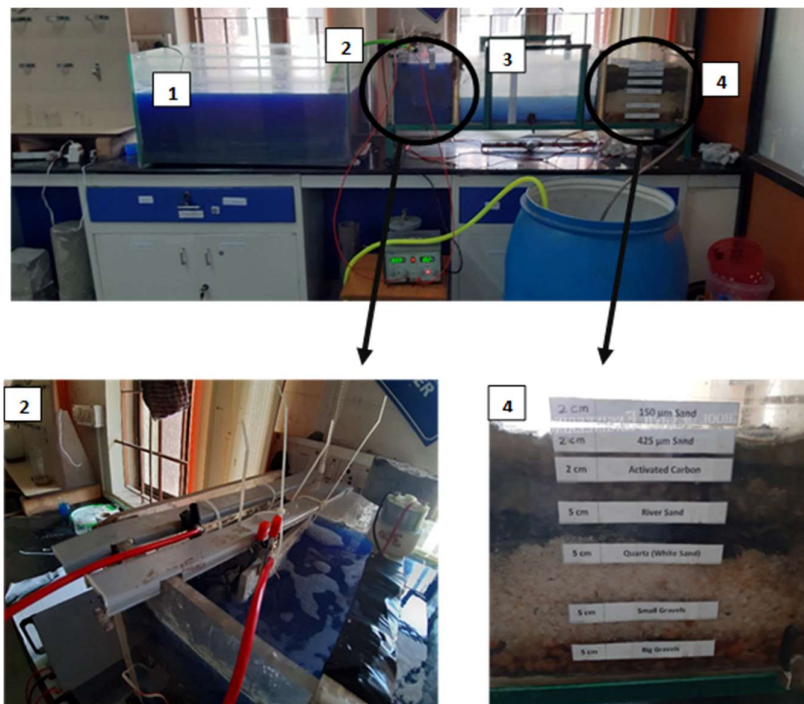


Fig. 2: Continuous Flow Reactor, Settling Tank, and Filtration.

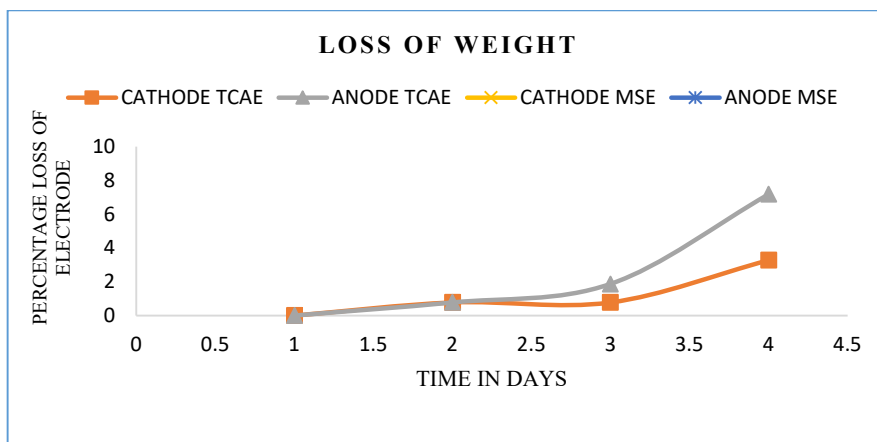


Fig. 3: Loss in weight of anode and cathode after treatment.

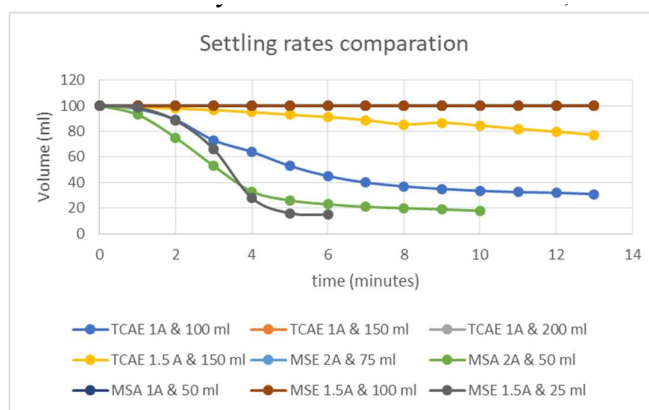


Fig. 4: Comparison of Settling rate with Ti and Mild Steel electrodes.

Electrode Consumption

The weight of the electrodes was measured before starting the experiment. Based on the difference in weight of the electrodes of different materials, the consumption of the electrode was determined. Based on the results (Fig. 3), it was observed that, for the same applied current, the consumption of the mild steel electrode was less when compared to the titanium-coated aluminum electrode. The less consumption of the electrode led to the decreased color removal efficiency in mild steel electrodes (Fig. 3).

Settling Rates

In the case of TCAE, when the flow rate was maintained at 100 mL with a current density was 45 A.m^{-2} , the settling rate of the flocs was high within 13 min (2.5 mL.min^{-1} to 5.3 mL.min^{-1}). When the flow rate was increased to 150 mL.min^{-1} with the same current density, the flocs were apart, and no settling was observed. Similar is the case at

the flow rate of 200 mL.min^{-1} . However, when the current density was increased to 67 A.m^{-2} , the settling was observed, but at a slower rate (1 mL.min^{-1} to 1.75 mL.min^{-1}). In the case of MSE, when the flow rate varied from 50 to 100 mL, there was no settling observed with the varying current from 45 A.m^{-2} to 89 A.m^{-2} . This is due to the lack of formation of hydroxide flocs because of the slow oxidation of anode which, in turn, less metal ions are being released to form the flocs. However, when the flow is reduced to 25 mL.min^{-1} , the flocs are formed due to the release of metal ions that can interact with hydroxyl ions to form the flocs. This has induced the fast settling of sludge at a rate varying from 2.5 mL.min^{-1} to 14.2 mL.min^{-1} . After this, the compression settling occurred. Also, at the flow rate of 50 mL.min^{-1} , with the current density of 89 A.m^{-2} , the settling rate was almost constant (7 mL.min^{-1} to 7.2 mL.min^{-1}).

The graphical plot represents the settling volume of the sludge at every minute when the reactor was operated at different current densities and flow rates. Mild steel

electrodes operated at $100 \text{ mL}\cdot\text{min}^{-1}$ flow rate with 1.5 Amp potential showed higher settling rates. However, the necessity to apply high potential for fewer flow rates makes mild steel undesirable over the titanium-coated aluminum electrode (Fig. 4).

Color Removal Efficiency (CRE)

The color removal efficiency (CRE) of the electrodes used was determined by the absorbance value measured at 325.5nm and 520nm wavelengths using a UV-spectrophotometer. The absorbance of the filter tank samples is also influenced by the packing materials that are used in the filter tank as also they contribute to the removal of the pollutants from the effluent. In the case of mild steel electrodes, a higher removal

efficiency of about 95.11% was observed with $50 \text{ mL}\cdot\text{min}^{-1}$. The flow rate of the effluent and 1.5 Amps of potential applied. In the case of titanium-coated Aluminum Electrodes (TCAE), better removal of about 92.1% was observed with $150 \text{ mL}\cdot\text{min}^{-1}$ and 1.5 Amps potential being applied (Fig. 5).

The absorbance of the samples collected from the filter tank, settling tank, and reaction tank indicates that the current applied to the electrodes plays a major role in the removal of the pollutants present in the effluent sample. It is observed that both electrodes showed better results when high potential was applied with minimum flow rates. The increase in flow rates has also influenced the removal efficiency, as the active flocs that are formed during the reaction time were being utilized over time.

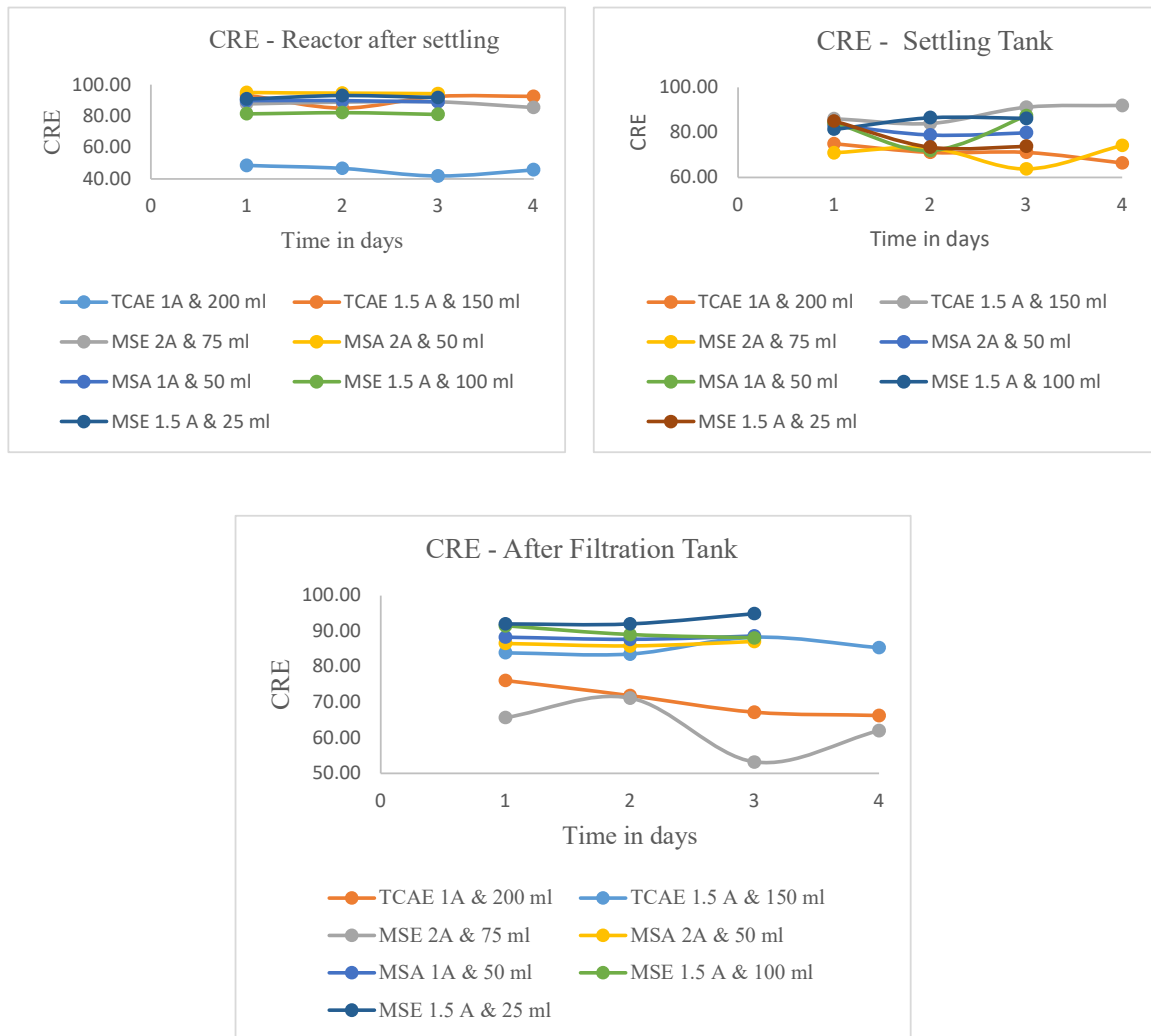


Fig. 5: Comparison of settling rate with Ti and mild steel electrodes at different flow rates.

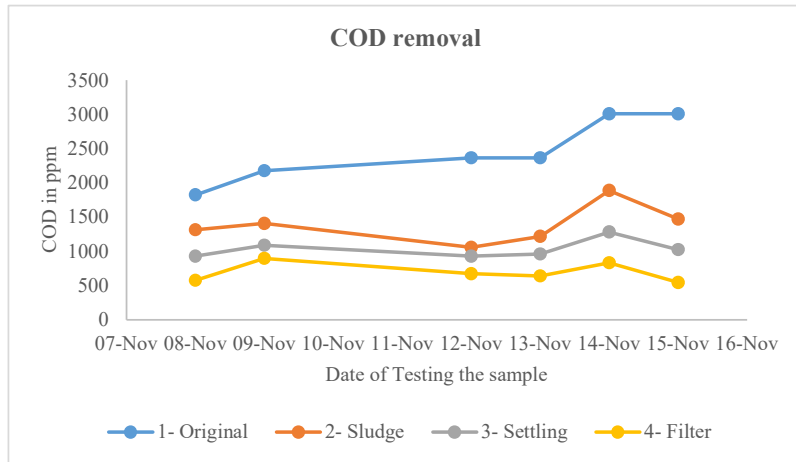


Fig. 6: Comparison of EC with Ti and mild steel electrodes at different flow rates in the reactor.

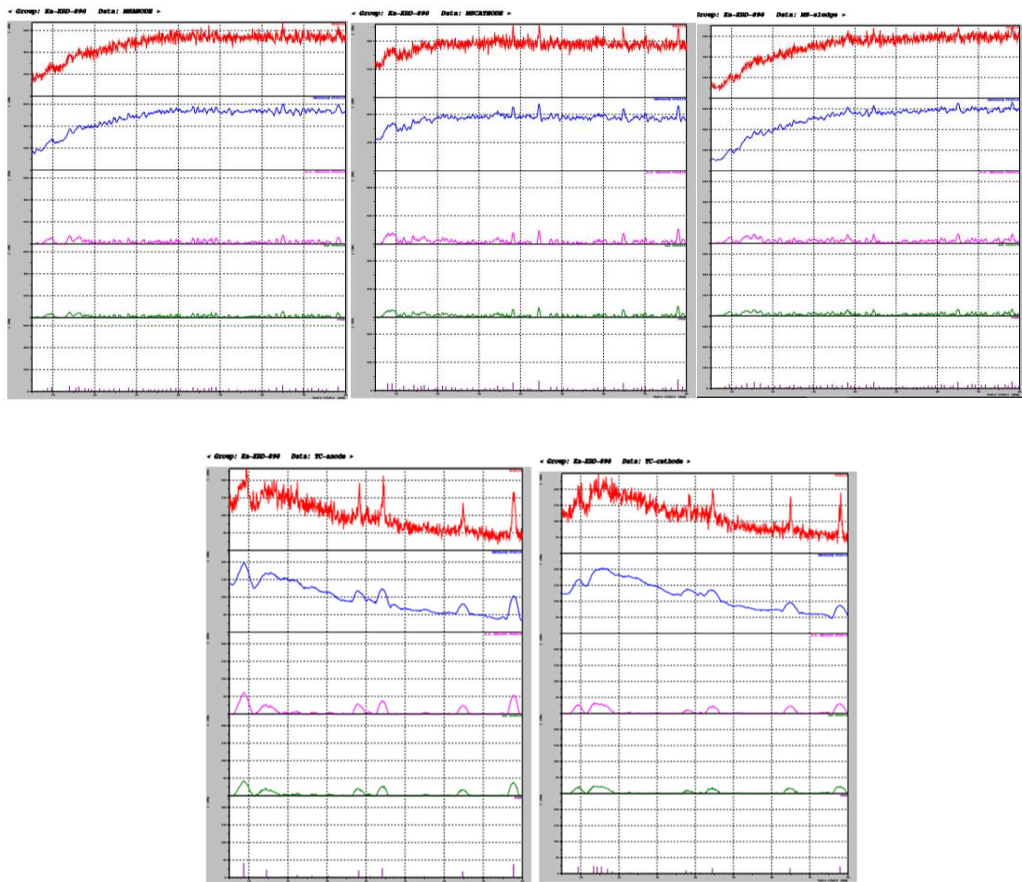


Fig. 7: XRD analysis of anodes and cathodes of Titanium coated Aluminum Electrodes (TCAE) and mild steel electrodes.

Chemical Oxygen Demand (COD)

Reduced levels of Chemical Oxygen Demand is an indicator of better treatment of the wastewater by the method adopted. In comparison with both the electrodes, the COD levels are greatly reduced in the case of the TCAE than the MSE (Fig. 6).

XRD

In the case of TCAE (scrap and sludge), the XRD pattern of Titanium shows a high intensity, strong diffraction peak at $2\theta=26,1^\circ$, which corresponds to an interlayer distance (d) of 3.401 Å. This confirms the presence of Titanium. The strongest peak at $2\theta=77.7^\circ$ with an interlayer distance

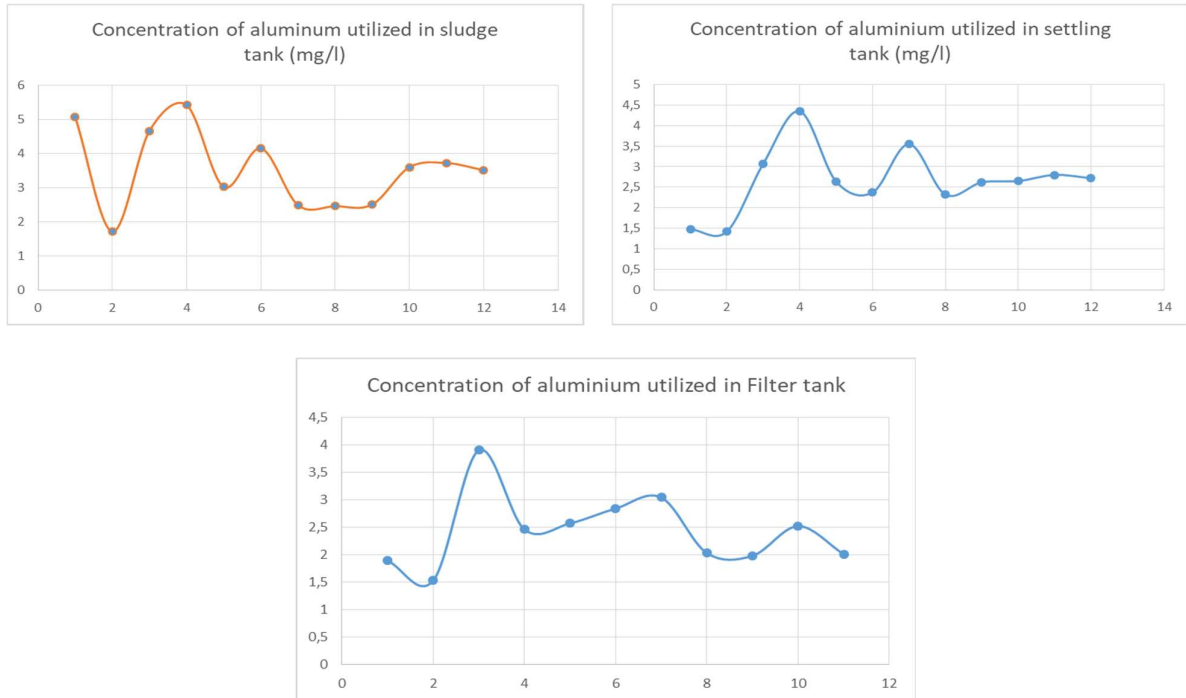


Fig. 8: Concentration of Aluminum in the reactor, settling tank and filtration.

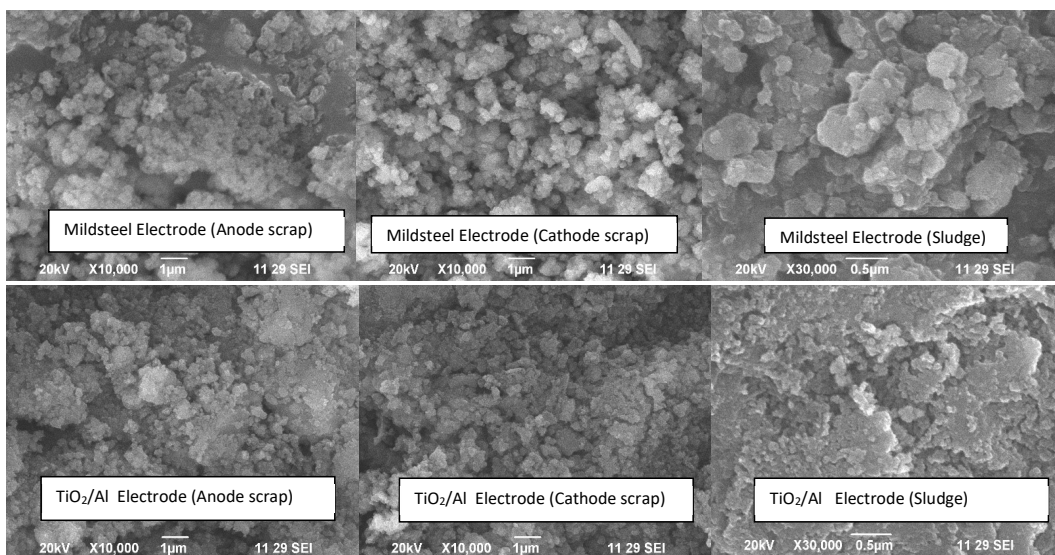


Fig. 9: SEM images of the sludge and the scrap from anode and cathode.

Table 1: Multicriteria analysis for optimization of operational parameters.

	TCAE 1A & 100 mL	TCAE 1A & 150 mL	TCAE 1A & 200 mL	TCAE 1.5A & 150 mL	MSA 2A & 75 mL	MSA 2A & 50 mL	MSA 1A & 50 mL	MSA 1.5A & 100 mL	MSA 1.5A & 25 mL
Lose of weight	8	8	8	8	5	5	5	5	5
Setting rate	14	0	0	5	0	17	0	0	17
% colour removal	16	13	9	16	5	16	14	20	22
pH	7	8	7	8	8	8	8	8	8
EC	10	6	10	2	8	7	6	5	4
TDS	10	6	10	2	8	7	6	5	4
COD	16	16	16	16	10	10	10	10	10
Total	81	57	60	57	44	70	49	53	70

(d) of 1.227 Å confirms the presence of aluminum. The crystalline or grain size of titanium was calculated using the Scherrer formula: $D = \frac{K * \lambda}{\beta * \cos(\theta)}$. Using this formula, the

crystalline size of aluminum and titanium is found to be 5.48 nm and 54.56 nm, respectively. In the case of MSE (scrap and sludge), the XRD diffraction peaks at 42.9, 65.3, 44.5, and 38.3 indicated the presence of Fe (Fig. 7).

Concentration of Aluminum

The above graph of AAS analysis indicates the concentration of aluminum in the filter samples collected from the reaction, settling, and filter tank (Fig. 8).

SEM Images

From the SEM images (Fig. 9), it was observed that the sludge obtained using TiO₂/Al electrodes is flaky, while the sludge obtained using mild steel electrodes is amorphous. The scrap obtained from both anode and cathodes from both types of electrodes are dense flocs, which are amorphous.

Multicriteria Analysis for Optimization of Operational Parameters

The most common way to obtain the best possible result is using a multicriteria analysis; in this method, the values are represented in percentages of any parameter. The comparison study run by using TCAE and MSE was mainly focused on analyzing the optimum experimental condition in terms of flow rate, current applied, electrolyte concentration, and reaction time.

From the multicriteria table analysis (Table 1), it is found that the TCAE operated at 1 Amp current with a 100 mL flowrate of effluent gave better results. From this study, it is found that the TCAE is efficient even at low potential

applied with maximum flow rate. Whereas in the case of MSE electrodes, it required high potential to be applied with minimum flow rates for better results. Thus, among the two electrodes used TCAE was more desirable to be used for industry-scale treatment methods.

CONCLUSIONS

The continuous flow treatment of industrial textile dyeing effluent from a small-scale textile dyeing unit was carried out using a prototype model in the laboratory in which titanium-coated aluminum plates and mild Steel plates were used as anodes and cathodes. The influence of different operational parameters, sludge settling studies, characterization of the sludge, and the water quality parameters were studied. From the experimental investigations, it was observed that the range of CRE varies from 92 to 96 % at the varying flow rate from 25 to 200 mL.min⁻¹. About 70% removal of COD was noticed in the treated effluent in addition to the dye removal. The characterization studies confirmed the removal of dye and organic content. The crystalline size of the sludge/flocs formed using the treatment using TiO₂/Al electrodes in the reactor and characterized through XRD was 10 times larger than the flocs formed using mild steel electrodes, leading to the faster settling rate and high CRE. The multicriteria analysis used to identify the optimum operational parameters for the efficient removal of dye also confirmed the same. It is concluded that TiO₂/Al can be effectively used in small-scale textile dyeing units with high CRE, COD, and less settling time and current density.

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