Research Article

Remediation of COD and color from textile wastewater using dual stage electrocoagulation process

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Abstract
The study has been done to investigate the effects of the current density on COD removal, color removal and electrode consumption on simulated textile wastewater using malachite green dye in the dual stage electrocoagulation process with settler. The results indicated that double stage electrocoagulation with settler is efficient at current density 165 A/m^2 and able to attain 92.60% COD removal and over 93.11% color removal. The most suitable current density for process is 135 A/m^2 in term of COD and color removal efficiency giving removal of 90.93 and 92.18%, respectively. analysis of variance (ANOVA) reveals that COD removal consistently changes for different COD concentrations among first and second stage electrocoagulation as well as settler. However, Tukey's test shows significant ($P < 0.001$) removal of COD. In case of color, ANOVA reveals significantly different color concentration among first and second stage electrocoagulation as well as settler. Moreover, Tukey's test also shows significant ($P < 0.001$) removal of color. In case of electrode consumption, *t* test revealed significant electrode consumption. Correlating the removal of COD concentration (mg/L) and electrode weight loss (mg) by Pearson correlation analysis for 1st reactor and 2nd reactor, it revealed a significant and positive correlation between COD removal and electrode weight loss ($r = 0.98$, $P < 0.001$) indicating that as removal in COD concentration increases, electrode weight loss increases.

Keywords Electrocoagulation · COD · Color · Current density · Wastewater

1 Introduction

Water is one of the most essential components for the life of living beings on earth. Among many water consuming industries; textile is the one which releases wastewater having high concentrations of BOD, COD, heavy metals, pH and toxic colored compounds etc. into the ecosystem, which causes one of the most remarkable sources of visual pollution and perturbation in the aquatic life as well as public health [14, 20]. The color and polluting agent not only lead to undesired water properties but also disturb the light transmission, photosynthesis under water and thus water body [17, 19]. Therefore, it is essential to treat textile industry effluent in feasible, viable and economical manner.

Many technologies like adsorption, coagulation-flocculation and membrane based technologies, biological methods, advanced oxidation/reduction methods etc. [5, 11, 12] have been reported to treat textile industry effluents. Adsorption onto activated carbon is associated with high cost, difficult regeneration and high waste disposal cost while other current processes are not economically feasible on a large scale [16, 18, 28]. Recently, due to addition of electrochemical process to these technologies, the removal of organic pollutants especially organic dyes from various effluents are common [10, 21]. Electrochemical techniques were traditionally used for synthesis

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of various compounds or for metal recovery but in recent times, other processes have been come into existence including their significant role in different textile industries processes. Oxidative processes have been reported to be effective in chemical degradation such as chlorine, but it produces some very toxic products such as organic chlorine compounds [23, 25]. Advanced oxidation processes such as ozonation, ultra-violet (UV) and ozone–UV combined oxidation, photo catalysis, Fenton reactive and ultrasonic oxidation are not economically feasible [4, 27, 32]. Electrocoagulation and electro coagulation/flotation processes can be applied to a wide range of both water and wastewater treatment projects and is most effective in removing inorganic contaminants and pathogens. Because of their wide use, they have been used for groundwater and surface water remediation at several sites [33]. These processes are characterized by ease of operation, reduced production of sludge and negligible consumption of chemicals. They have been applied efficiently to various water treatment problems. So, if electrocoagulation could replace normal chemical coagulation processes, very little alteration would be required to make the present treatment plants more efficient and resolve those many troubles caused by chemical coagulation [15].

In recent years, some of the researchers have found that electrocoagulation showed better removal efficiency in COD, color, turbidity and total dissolved solids [2, 3] in dye laden wastewater discharged from textile industries. Electrocoagulation process provides a simple process for the treatment of wastewater without any need of supplementary chemicals, due to which no secondary pollution occurs. It also minimizes the rate of sludge production, which has to be disposed. In most basic terms, electrocoagulation is a process in which electrical current is applied on wastewater for in situ production of coagulant through electrolytic oxidation of a suitable anode material [6]. This coagulant then forms large agglomerates with the various cation species in the impurities in wastewater and either settle to bottom or rise up on the top along with the gases produced [1, 29]. A major advantage of electrocoagulation above commercial techniques is that dosage of the coagulant can be controlled by adjusting the current which makes process of wastewater treatment very easy [7, 8, 22, 30, 35].

The present study will be carried out to investigate the following: (1) applicability of the electrocoagulation procedure for the treatment of synthetic textile wastewater prepared using malachite oxalate green dye in laboratory. (2) The effects of current density on COD removal, color removal, Electrode consumption for the unit COD removal and to study the effect of combination of reactors with settler.

2 Materials and methods

2.1 Preparation of synthetic textile wastewater

2.1.1 Malachite green dye

Wastewater discharged from various processes from different coloring industries including textile are usually polluted by dyes. Malachite green is one of the most commonly used dye. It is mostly used for the dyeing of cotton, silk, paper, leather and also in manufacturing of paints and printing inks, are toxic and must be removed before discharge into water bodies. About 10–15% of all dyes are combined to wastewater in the dyeing process [26].

Malachite green oxalate dye, C.I. Basic Green 4, Classification Number 42,000, chemical formula = $C_{52}H_{54}N_4O_{12}$, molecular weight = 463.50, λ max = 618 nm (measured value) was supplied by Sigma-Aldrich (M) Mumbai, India (Fig. 1).

Synthetic textile wastewater having dye concentration 300 mg/L was used in our experimental work. The dye was then accompanied with the following chemicals (sodium chloride 0.3 g/L, hydrolyzed starch 3 g/L, ammonium sulphate 0.0174 g/L, disodium hydrogen phosphate 0.0174 g/L and detergent drops in order to correctly simulate the actual dye waste released from textile industries (all the chemicals and reagents used were purchased from SD fine chem. Ltd. Mumbai). This mixture was then added

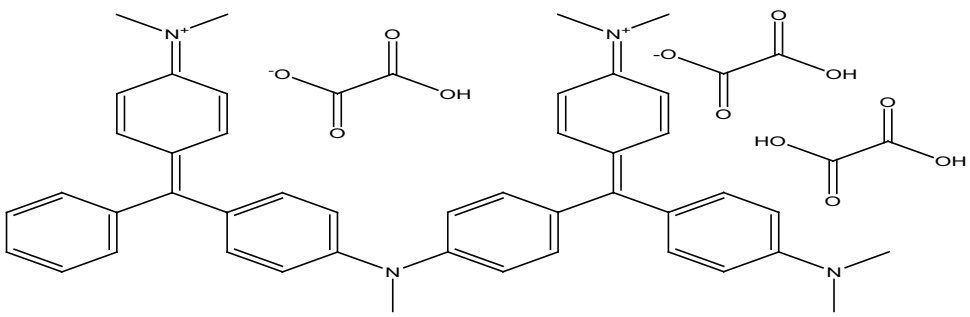

Fig. 1 Chemical structure of malachite basic green-4 dye

to the distilled water and heated at 100 °C for 30 min and cooled to room temperature.

2.2 Reactor design

The experimental system included two EC reactor made up of acrylic sheet. The total volume of both reactors was 5.4 and had 3 L of working volume each. A cassette was also organized so that electrodes were held at their individual positions during the experiment. The dimensions of each Al electrode used were $9.7 \times 8.0 \times 0.2$ cm making their effective surface area as 160 cm^2. The two anodes and three cathodes were set in a monopolar configuration. The overall effective surface area of electrodes in reactor was 800 cm^2. Inter electrode spacing was kept at 2 cm and reactors were connected to a DC power supply unit in series mode. The ammeter and voltmeter installed in power supply helped in recording electric current and voltage values. Two reactors were followed by a settler prepared by acrylic sheet. Settler consisted of 22 parallel plates with each plate inclined at an angle of 60° to the horizontal making total working volume of 7.4 L. Parallel plates were of acrylic sheet and having total effective surface area of 5200 cm^2 to provide improved settling. Flow rate was kept at 200 mL/min for all the experimental runs. HRT was 37 min for settler and 27 min for both the reactors.

2.3 Experimental procedure

Wastewater was flowed into the first reactor from an over head tank through plastic pipe ending up in a ball valve adjusted with pump at required flow rate. The experimental runs at the rate of 200 mL/min were performed continuously in double stage reactor system which was followed by settler. The pH was kept between 5 and 7 in this study.

The runtime of each experimental run was considered to start when DC power supply unit was switched on. Anodes were consumed during the course of the run while hydrogen gas was evolved at the cathode helping the scum material to float at the top. After the steady state was achieved, 10 mL samples were withdrawn from both the reactors and settler after every 20 min. Samples were then analyzed for COD and color removal. Current and voltage were noted down at the time of sample collection for the calculations. The consumption of electrodes was weighted before and after the completion of run.

After each experimental run, electrodes were washed thoroughly and then mechanically scraped so that their clean surface was exposed for the next run. Reactors and settler were also washed cleanly after each run, so that any scum particles were removed. Figure 2 shows the experimental setup.

2.4 Evaluation of removal efficiency

2.4.1 Analytical study

Standard method was used for the analysis of tested physico chemical parameters as described in standard methods for the examination of water and wastewater analyses [31]. Each sample taken from the electrocoagulation cell was analyzed in the laboratory.

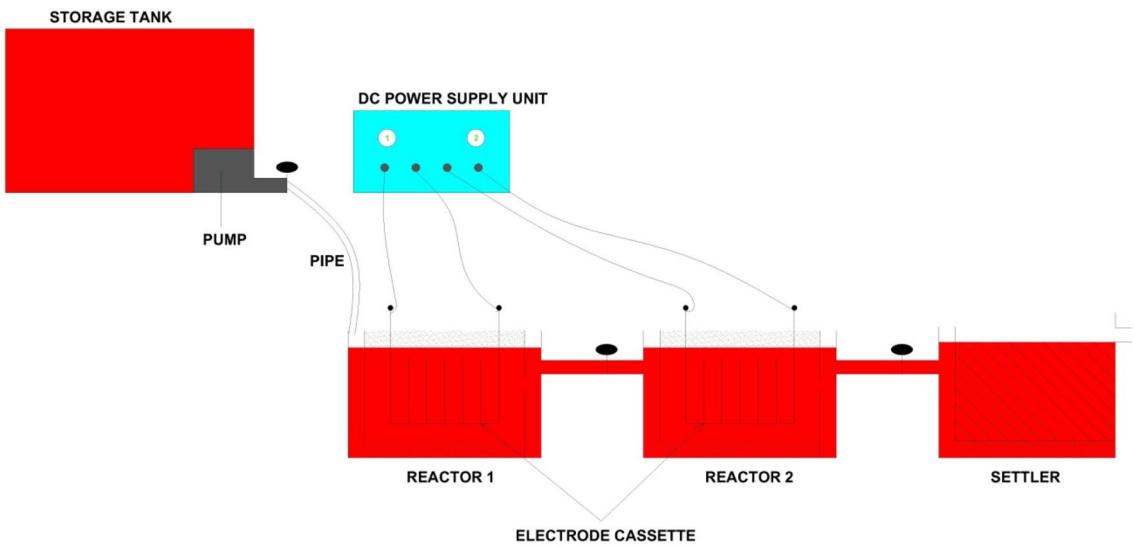

Fig. 2 Experimental setup electrocoagulation process

2.4.1.1 COD The removal efficiency of COD in synthetically polluted water treated by electrocoagulation was calculated as follows:

$$Y\% = \frac{C_0 - C}{C_0} \times 100$$

where; Y% is the COD removal efficiency, C_0 is the COD concentration at initial (mg/L), C is the COD concentration at any time (mg/L).

COD was determined using the COD analyzer (AQUA LYTIC model no. PCH 70361).

2.4.1.2 Color Efficiency of color removal was expressed as percentage of absorbance removal calculated as:

$$Percentage\ color\ removal = \frac{C_i - C_f}{C_i} \times 100$$

where; C_i is the initial concentration of color in wastewater (at 618 nm), C_f is the final concentration of color in treated wastewater.

Color was determined using UV–VIS spectrophotometer (Chemito instruments Pvt. Ltd. Model UV 2000).

2.4.2 Statistical analysis

Data was summarized as mean ± SD. Pre and post groups were compared by paired *t* test. Groups were also compared by one way analysis of variance; ANOVA followed by Tukey's multiple comparison test. Pearson correlation analysis was done to assess association between removal of COD concentration and electrode consumption (weight loss). A 2-tailed *P* value less than 0.05 was considered statistically significant. PRISM version 5.0 statistical software was used for the analysis of data.

3 Result and discussions

3.1 Effect of current density on the removal of COD

The experiments were carried out to understand the effect of electrocoagulation process when current density was varied from 30 to 165 A/m^2 in difference of 15 A/m^2. The samples were collected after 20 min of time interval and their COD was determined. In this study, the primary aim was to comprehend the effect of current density on COD i.e. COD removal profile was obtained for different combination of applied current density. Thus, results of performance of EC process for various combinations of applied current density have been reported as COD removal efficiency. The experiments were continued till the COD of effluent for three consecutive samples were almost same. This state was taken as steady state.

3.1.1 COD result analysis

After first reactor from the Fig. 3 it is clearly evident that COD removal efficiency increased from 37.69 to 75.95% as the current density was increased from 30 to 165 A/m^2. It was found that COD removal efficiency from current density 30 to 90 A/m^2 increased more rapidly i.e. from 37.69 to 71.56% as compared to current density 90 to 120 A/m^2 i.e. from 71.56 to 74.14%. With further increase in current density from 135 to 165 A/m^2, removal efficiency increased from 74.89 to 75.95% i.e. only 1.06% thus can be considered as at steady state. It can also be concluded that as the current density increases there is reduction in results of COD as reported by earlier studies [24]. Maximum COD removal (75.95%) after first reactor was found at current density 165 A/m^2.

After second reactor from the Fig. 4, it is clearly evident that COD removal efficiency continuously increased from 59.03 to 90.18% with the increase in current density from 30 to 165 A/m^2, same trend as was observed in the case of the first reactor. Removal efficiency increased from 59.03 to 85.73% for an increase in current density for 30 to 120 A/m^2 because at a high current density, the level of anodic dissolution of aluminum increases, resulting in formation of greater amount of precipitate and removal of organics. And as we move to current density of 135 to 150 A/m^2 the increment in efficiency goes steady with the increase of 1.35%. Maximum COD removal was obtained at current density 165 A/m^2 (90.18%).

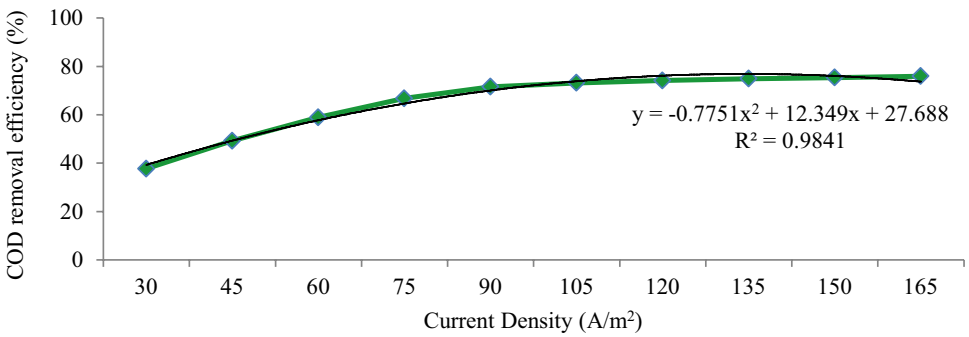

Fig. 3 COD removal efficiency after first reactor for current density 30 to 165 A/m^2

After settler from the Fig. 5, it is clearly evident that COD removal increased constantly with an increase in the current density and reached maximum of 92.60% removal after settler for current density of 165 A/m^2.

Figure 6 represents the combined results of COD removal efficiency after first, second reactor and after settler for current density varied from 30 to 165 A/m^2, although, maximum removal reached 75.95% for first reactor at 165 A/m^2. There was much difference in COD removal efficiency after first reactor and settler from current density 30 to 135 A/m^2 but almost equal or little more increase in COD removal after second reactor and settler. In a similar study [35] it was observed that COD removal was 52.3% at a current density of 100 A/m^2 and 76.1% at a current density of 200 A/m^2 at a constant flow rate of 10 L/h and COD removal was 69.5% at 100 A/m^2 and 49.7% at 200 A/m^2.

The removal of COD concentration from its initial value to three different treatments (after 1st reactor, after 2nd reactor and settler) groups is summarized in Table 1. The initial (pre treatment) (mean ± SD) COD was 514.00 ± 0.00 mg/L, after 20 min of treatments; it remained 175.94 ± 67.41 mg/L after 1st reactor, 111.46 ± 57.36 mg/L after 2nd reactor and 97.88 ± 54.91 mg/L after settler.

From initial, the mean COD removal was cumulatively 65.8, 78.3 and 81.0% at 1st reactor, 2nd reactor and settler respectively as shown in Fig. 7. In other words, the

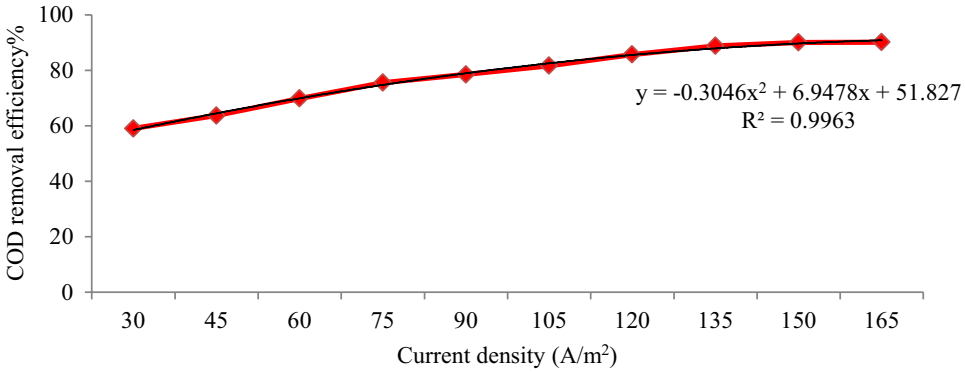

Fig. 4 COD removal efficiency after second reactor for current density 30 to 165 A/m^2

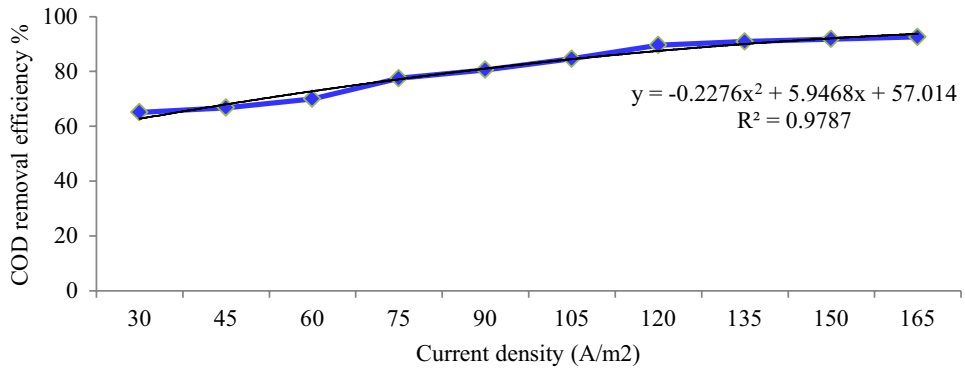

Fig. 5 COD removal efficiency after settler for current density 30 to 165 A/m^2

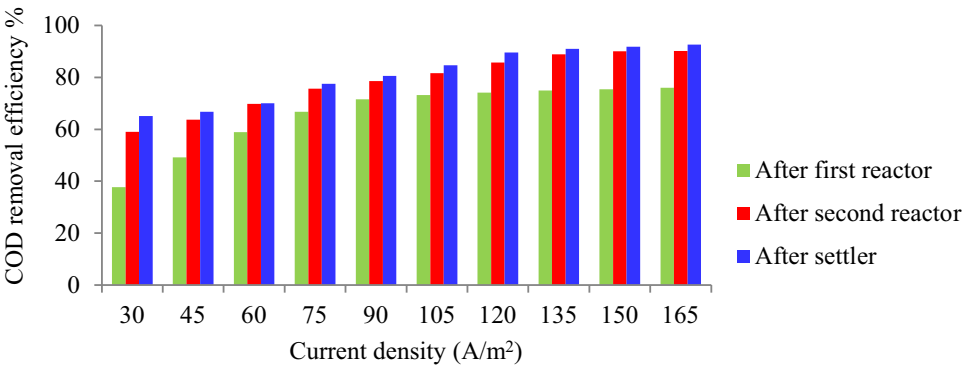

Fig. 6 COD removal efficiency after first, second reactor and after settler for current density 30 to 165 A/m^2

Table 1 Removal of COD concentration (mg/L) at three different processes

Initial	After 1st reactor	After 2nd reactor	After settler
514.00 ± 0.00	175.94 ± 67.41	111.46 ± 57.36	97.88 ± 54.91

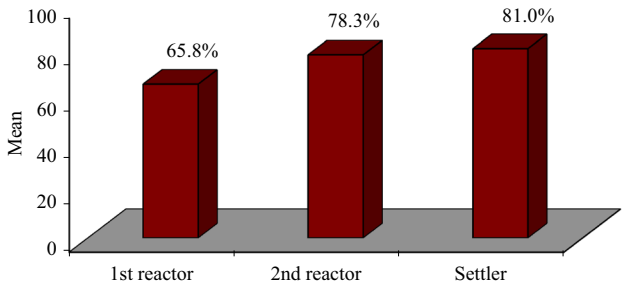

Fig. 7 Cumulative mean removal of COD concentration (%) at three different processes

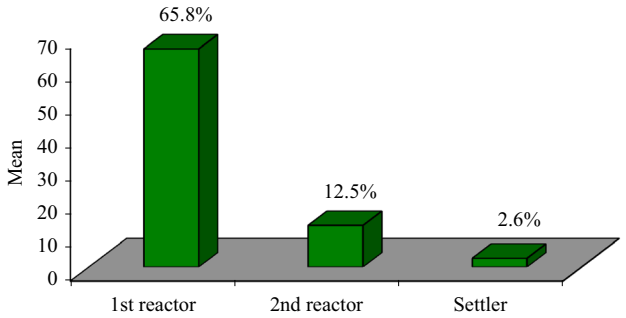

Fig. 8 Net mean removal of COD concentration (%) at three different processes

mean COD removal was 65.8% at 1st reactor, 12.5% at 2nd reactor and 2.6% at settler as shown in Fig. 8.

Comparing the pre (initial) and post (at 1st reactor, at 2nd reactor and settler) treatments COD of four groups together, ANOVA revealed significantly different COD among the groups ($F = 141.30$, $P < 0.001$) as given in Table 2.

Further, after performing ANOVA, Tukey's HSD (honestly significant difference) post hoc test was done to compare the mean COD between the groups as given in Table 3, Tukey's test showed significant ($P < 0.001$) removal of COD at 1st reactor, 2nd reactor and settler as compared to initial.

Table 3 Comparison of COD concentrations between groups by Tukey's test

Comparisons	Mean diff.	q	*P* value	95% CI of diff.
Initial versus 1st reactor	338.10	20.53	< 0.001	275.30–400.90
Initial versus 2nd reactor	402.50	24.44	< 0.001	339.70–465.30
Initial versus settler	416.10	25.27	< 0.001	353.30–478.90

3.2 Effect of current density on color removal

The experiments were carried out to understand the effect of electrocoagulation process under continuous flow regime on various current densities varied from 30 to 165 A/m^2 in difference of 15 A/m^2. The samples were collected at regular time interval of 20 min and tested for % color removal. At various current densities, the results of performance of EC process have been reported as color removal efficiency.

3.2.1 Color removal analysis

Figure 9, clearly shows that as current density increases from 30 to 165 A/m^2, color removal efficiency increased from 52.46 to 81.92%. This was due to the fact that with increasing current density, the amount of Al^{3+} cations released by the anode and therefore of $Al(OH)_3$ particles also increases. From current density 30 to 120 A/m^2, color removal efficiency increases from 52.46 to 76.77% i.e. 24.31%. Further increase in current density from 120 to 165 A/m^2, the rate of color removal efficiency slowed down and almost became same. Zheng et al. [34] also studied electrochemical treatment process for the treatment of wastewater and found a similar trend. At current density of 165 A/m^2, the maximum removal efficiency increases to 81.92%, because at higher current density, the amount of anodic dissolution of aluminum increases, resulting in a superior amount of precipitate and removal of organics.

Table 2 Comparison of COD concentrations among groups using one way analysis of variance

Source of variation (SV)	Sum of square (SS)	Degree of freedom (*DF*)	Mean square (MS)	F ratio	*P* value
Groups	1,150,000	3	383,300	141.30	*P* < 0.001
Residual	97,650	36	2713		
Total	1,247,000	39	386,013		

After second reactor from the Fig. 10, it can be clearly observed that increase in current density from 30 to 165 A/m^2, color removal efficiency after second reactor increases from 61.79 to 90.40%. From current density 30 to 105 A/m^2 removal efficiency rapidly increases from 61.79 to 82.08% i.e. 20.29%. From current density 135 to 165 A/m^2 efficiency gradually increases and is almost same.

After settler it was observed that color removal showed a direct relationship with the current density reaching from 65.51% at 30 A/m^2 to a maximum of 93.11% at 165 A/m^2, as shown in Fig. 11.

Figure 12 represents the combined results of color removal efficiency after first, second reactor and after settler for current density 30 to 165 A/m^2. In the similar study [35] observed that color removal for DR81 dye was 71.5% at a current density of 100 A/m^2 and 90.2% at a current density of 200 A/m^2 and a constant flow rate of 10 L/h. Also color removal was 61.5% at a current density

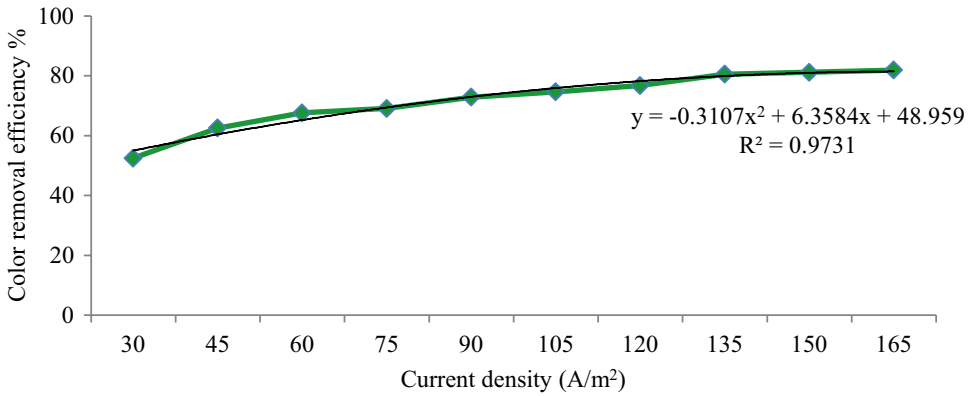

Fig. 9 Color removal efficiency after first reactor for current density 30 to 165 A/m^2

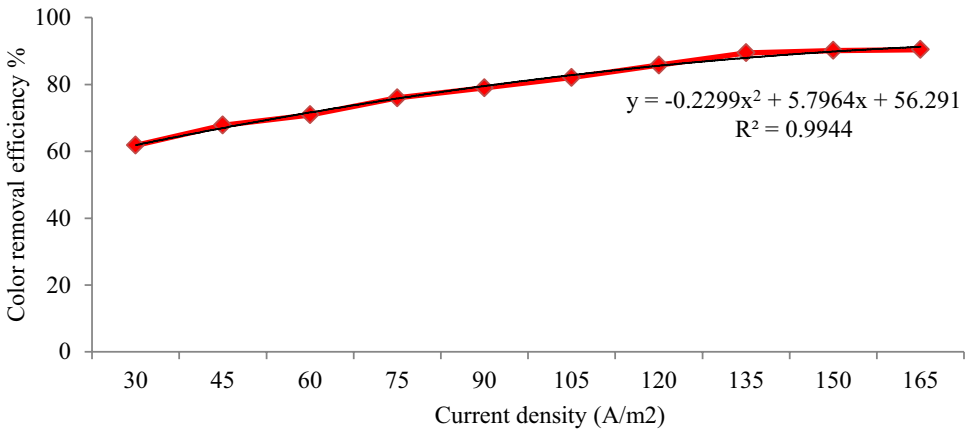

Fig. 10 Color removal efficiency after second reactor for current density 30 to 165 A/m^2

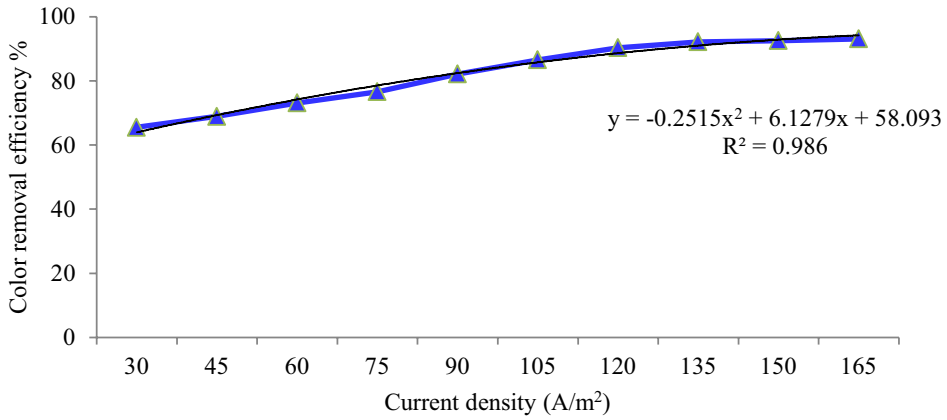

Fig. 11 Color removal efficiency after settler for current density 30 to 165 A/m^2

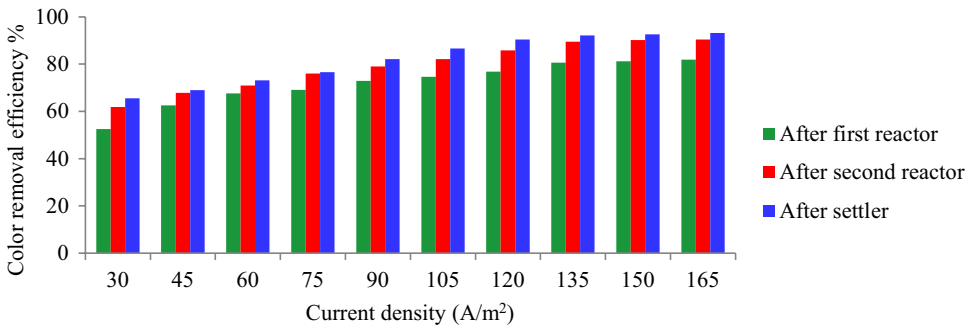

Fig. 12 Color removal efficiency after first, second reactor and after settler for current density 30 to 165 A/m^2

Table 4 Removal of color (mg/L) at three different processes

Initial	At 1st reactor	At 2nd reactor	Settler
300.00 ± 0.00	84.10 ± 28.02	62.04 ± 30.23	53.66 ± 31.29

of 100 A/m^2 and 76.8% at a current density of 200 A/m^2 at a constant flow rate of 28 L/h. As 93.11% efficiency at high current density of 165 A/m^2 is insignificant as [13] predicted that as current density increases energy consumption in process will increase thus cost of the treatment of textile wastewater also increases. So from economic point of view, most suitable current density for process is 135 A/m^2 in term of COD and color removal efficiency giving removal of 90.93 and 92.18% respectively.

The removal of color from its initial to three different treatments (after 1st reactor, after 2nd reactor and settler) groups is summarized in Table 4. The initial (pre treatment) mean ± SD color concentration was 300.00 ± 0.00 mg/L, after 20 min of treatments; it remained 84.10 ± 28.02 mg/L after 1st reactor, 62.04 ± 30.23 mg/L after 2nd reactor and 53.66 ± 31.29 mg/L after settler.

From initial, the mean color concentration removed cumulatively was 72.0, 79.3 and 82.1% at 1st reactor, 2nd reactor and settler respectively (Fig. 13). In other words, the mean color removed was 65.8% at 1st reactor, 7.4% at 2nd reactor and 2.8% at settler as shown in Fig. 14.

Comparing the pre (initial) and post treatments (at 1st reactor, at 2nd reactor and settler) color concentration of four groups together, ANOVA revealed significantly different color concentration among the groups (F = 205.90, $P < 0.001$) as given in Table 5.

Further, after performing ANOVA, Tukey's HSD (honestly significant difference) post hoc test is done to compare the mean color concentration between the groups as given in Table 6, Tukey's test showed significant ($P < 0.001$) removal of color after 1st reactor, 2nd reactor and settler as compared to initial mean concentration.

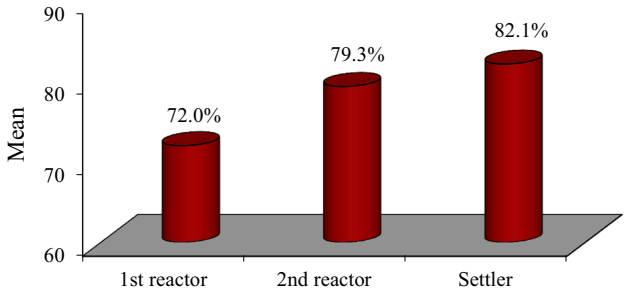

Fig. 13 Cumulative mean removal of color (%) at three different processes

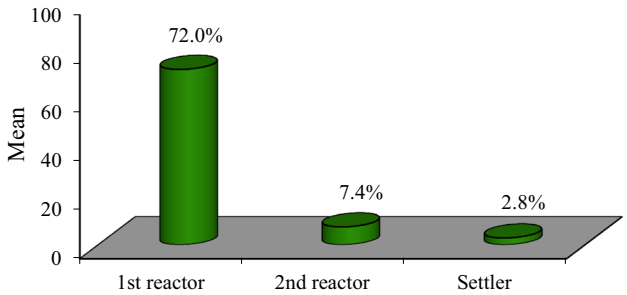

Fig. 14 Net mean removal of color (%) at three different processes

Table 5 Comparison of color concentration among groups using one way analysis of variance

Source of variation (SV)	SS	*DF*	MS	F value	*P* value
Groups	413,500	3	137,800	205.90	< 0.001
Residual	24,100	36	670		
Total	437,600	39	138,470		

Table 6 Comparison of color concentration between groups by Tukey's test

Comparison	Mean diff.	q	*P* value	95% CI of diff.
Initial versus 1st reactor	215.90	26.39	< 0.001	184.70–247.10
Initial versus 2nd reactor	238.00	29.08	< 0.001	206.80–269.20
Initial versus settler	246.30	30.11	< 0.001	215.10–277.50

3.3 Effect of current density on electrode consumption

Electrode consumption (weight loss) during the 1st reactor and 2nd reactor process is summarized in Tables 7 and 8 respectively. The initial (pre treatment) mean weight of electrode at 1st reactor was 44,965.70 ± 490.07 mg and after 20 min of process it was 44,803.63 ± 537.40 mg. Comparing the pre and post electrode weight, *t* test reveled significant electrode consumption (162.07 ± 51.84 mg or 0.36%) at post as compared to pre (44,965.70 ± 490.07 vs. 44,803.63 ± 537.40, t = 9.89, $P < 0.001$). Hakizimana et al. [9] also found the similar trend for electrode consumption as was achieved in the present study.

Similarly, the initial (pre treatment) mean weight of electrode at 2nd reactor was 45,447.59 ± 75.15 mg and after 20 min of process it was 45,419.56 ± 80.64 mg. Comparing the pre and post electrode weight, *t* test reveled significant electrode consumption (28.03 ± 8.92 mg or 0.06%) at post as compared to pre (45,447.59 ± 75.15 vs. 45,419.56 ± 80.64, t = 9.94, $P < 0.001$).

Further, correlating the removal of COD concentration and electrode weight loss of both the processes (1st reactor and 2nd reactor), Pearson correlation analysis revealed a significant and positive correlation between COD removal and electrode weight loss (r = 0.98, $P < 0.001$) indicating as removal of COD concentration increases electrode weight loss increases as shown in Fig. 15.

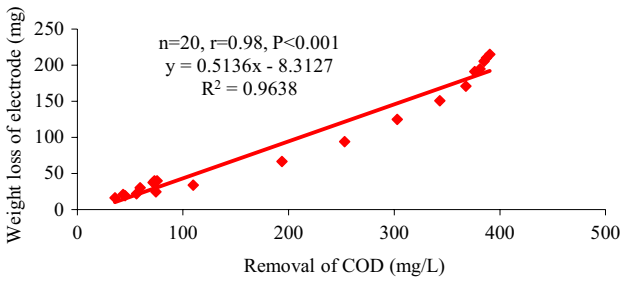

Fig. 15 Correlation between removal of COD concentration (mg/L) and electrode consumption (mg)

4 Conclusions

The result observed in this work shows the application of electrocoagulation process in the treatment of wastewater being discharged from different textile industries. In treating the wastewater for COD and color the efficiency level increases with increase in current density but the rate of increase decline with increasing current density above a certain value. It can be concluded that for highest level of current density the COD and color removal reached a maximum of 92.60 and 93.11% respectively, however more electrode was consumed during the process. For COD removal and color removal, most significant current density in double stage electrocoagulation with settler was 135 A/m^2 in which COD and color removal was 90.93 and 92.18% respectively. In case of electrode consumption it was also observed that the consumption of electrode was less in second reactor as compared to first reactor on increase of current density giving improved efficiency of COD. Statistically, we concluded from the study that in case of COD on comparing the results of four groups which comprises of initial concentration and results after first reactor, second reactor and settler, ANOVA revealed significantly different COD concentrations among the groups. Further, comparing the mean COD between the groups, Tukey's test showed significant ($P < 0.001$) removal of COD at 1st reactor, 2nd reactor and settler as compared to initial. In case of color on comparing the results of four

Table 7 Electrode consumption (mg) at 1st reactor

Initial	Final	Weight loss (initial–final)	t value (*DF* = 9)	*P* value
44,965.70 ± 490.07	44,803.63 ± 537.40	162.07 ± 51.84	9.89	< 0.001

Table 8 Electrode consumption (mg) at 2nd reactor

Initial weight (mg)	Final weight (mg)	Weight loss (initial–final)	t value (*DF* = 9)	*P* value
45,447.59 ± 75.15	45,419.56 ± 80.64	28.03 ± 8.92	9.94	< 0.001

groups together, ANOVA revealed significantly different color concentration among the groups. Further, comparing the mean color concentration between the groups, Tukey's test showed significant ($P < 0.001$) removal of color at 1st reactor, 2nd reactor and settler as compared to initial. In case of electrode consumption, comparing the pre and post electrode weight, *t* test revealed significant electrode consumption. Further correlating the removal of COD concentration (mg/L) and electrode weight loss (mg) by Pearson correlation analysis for both processes (1st reactor and 2nd reactor) a significant and positive correlation between COD removal and electrode weight loss ($r = 0.98$, $P < 0.001$) was observed, indicating that as removal of COD concentration increases, electrode weight loss also increases.

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Compliance with ethical standards

Conflict of interest There is no conflict of interest.

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