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Efficient treatment of textile dyeing effluent by electrocoagulation process using aluminium electrode

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ABSTRACT

The electrocoagulation (EC) treatment of textile dyeing effluent was carried out in a 2 dm^3 electrolytic batch reactor using aluminium as a sacrificial electrode. With the four-plate setup a current density of 110.16 A/m^2, pH 6 and electrode gap 2 cm was found to be optimal. Maximum chemical oxygen demand and colour reduction of 93% and 73% respectively was achieved. The metal ion discharge rate of aluminium was found strongly dependant on initial pH (pH$_i$) of effluent. At lower pH$_i$ (pH 2) anode consumption increases while energy consumption decreased. At the optimum condition, the loss of aluminium electrode was 38.86 mg/dm^3 effluent and energy consumption was 16.09 Wh/dm^3. The settling characteristics of electrocoagulation sludge were also analyzed at different pH. It was observed that the settling characteristic of treated slurry provided better results at pH$_i$ 10. Besides, scum and residues were also characterized at different pH.

Keywords: Textile dyeing effluent; Chemical oxygen demand; Colour removal; Energy consumption; Electrode loss

1. Introduction

India is the second-largest producer of textile and hence it is an important segment of the Indian economy. Further, India has abundant raw materials like cotton, wool, silk and jute in addition to comparative advantage in terms of skilled manpower and cost of production. Recently, the Indian government had permitted 100% foreign direct investment in the textile sector which indicates the focus of government towards Indian textile industries.

The effluent is generated at different stages of fabric preparation that includes the dyeing and wet process. Most of the effluent is generated during wet processes like dyeing, bleaching, scouring, printing, and finishing. Dyeing is an important section of wet process operation. Dyeing industries utilize a large quantity of raw water and at the same time, a huge amount of wastewater is generated from the dyeing and finishing process. The wastewater generated during the dyeing process is commonly known as textile dyeing effluent (TDE). Several textile industries in India use a variety of synthetic dyes for the processing of textiles. TDE is rich in colour along with high chemical oxygen demand (COD). Also, it contains residues of dyes and chemicals. These dyes are organic compounds that are acidic or alkaline in nature. If this wastewater directly discharges in any water receiving body it can damage the aquatic system. Furthermore, the organic compounds which are present in TDE having carcinogenic effects may come in contact. The Central Pollution Control Board (CPCB) of India has fixed standard for the TDE to meet the effluent discharge quality, for the discharge of the wastewater into surface waters (COD < 0.1 kg/m^3, biochemical oxygen demand (BOD) < 0.03 kg/m^3) and sewer

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(COD < 0.3 kg/m³, BOD < 0.1 kg/m³) [1]. Besides, due to increased awareness in environmental issues throughout the world and scarcity of fresh water, proper treatment and disposal of textile wastewater is the need of the hour.

There are several conventional treatment technologies available for the treatment of TDE. Different combinations of physical, chemical and biological methods such as coagulation [2], filtration [3], adsorption [4], membrane separation [5], wet air oxidation [6], thermolysis [7], advanced oxidation process [8], ozonation [9], etc are have been reported in the literature. These treatment technologies have several disadvantages in terms of sludge generation as well as high operating costs. Besides, electrocoagulation is a better option to treat TDE, due to its easy operation, simple to use, energy efficiency and environmental friendliness. It does not require on-site treatment; consequently, there is no increase in the salinity of treated wastewater. Electrocoagulation is a versatile, reliable, effective, and energy-efficient process. It can effectively destabilize the smallest colloidal particle. Table 1 presented a detailed literature review on the studies of textile wastewater.

Basically, electrocoagulation is the generation of metal ions electrically by the supply of external direct current (DC) through the wastewater rather in batch or continuous reactor of different sizes and shapes. Generated metal ions act as a coagulant which forms metal hydroxides with the colloidal particles present in wastewater [15].

There are three steps involved in the EC process along with some side reactions

- Formation of coagulants species on a metal electrode surface

$$2M_{(s)} \rightarrow 2M^{+}_{(aq)} + 2ne^{-} \tag{1}$$

At the same time electrolysis of water takes place at the cathode and anode

$$4H_2O + 4e^{-} \rightarrow 2H_{2(g)} + 4HO^{-} \tag{2}$$

$$4H_2O \rightarrow 2O_2 + 8H^{+} + 8e^{-} \tag{3}$$

- Destabilization of the colloidal particles

$$M^{n+}_{(aq)} + ne^{-} \rightarrow M_{(s)} \tag{4}$$

Furthermore, the metal hydroxide formed at the electrode surface increases the pH of the wastewater which shifts pH towards neutral.

$$2M^{n+} + 2nOH^{-} \rightarrow 2M(OH)_{n(s)} \tag{5}$$

- Aggregation of the destabilized particles

Generated metal ions at the electrode surfaces react with wastewater to form various metal hydroxides and form polymers.

For the case of aluminium, anodic and cathodic reactions are as follow:

Anodic reaction

$$Al_{(s)} \rightarrow Al^{3+}_{(aq)} + 3e^{-} \tag{6}$$

Cathodic reactions

$$3H_2O + 3e^{-} \rightarrow \frac{3}{2}H_{2(g)} + 3OH^{-} \tag{7}$$

$$Al^{3+} + 3H_2O \rightarrow Al(OH)_3 + 3H \tag{8}$$

However, the efficiency of the electrocoagulation process depends on some operational parameters such as current density, pH, electrode gap, electrode material, electrolysis time, temperature and conductivity of the solution [16].

The present work aims to obtain an optimized condition of electrocoagulation process parameters for the treatment of TDE at different conditions. In this perspective, the COD and colour reduction have been determined at varying pH, current density, and electrode gap. Also, the settling characteristics of the electrocoagulation sludge have been presented.

2. Material and methods

2.1. Effluent and its characterization

The TDE used in this work was collected from the nearby textile industry at Pithampur, Madhya Pradesh. To preserve the constant characteristics of TDE, the sample was placed at 3°C in a deep freezer. Aluminium plate was used as a sacrificial electrode. Reactor was made up of Perspex glass. The effluent was analyzed in terms of various parameters such as COD, colour, total solids, total dissolved solids (TDS), total suspended solids (TSS), etc., as per the standard method of analysis [17]. The main characteristic of the original and treated effluent used for this study is presented in Table 2.

2.2. Experimental methods

The lab-scale batch experimental setup used during the EC process is presented in Fig. 1. The characteristic of EC cell is also important which is presented in Table 3. 1.7 dm³ TDE was filled in a 2 dm³ EC reactor. To set the initial pH of effluent, H_2SO_4 (1 M) and NaOH (1 M) were used. Aluminium plates of 2 mm thickness were used for the experiment as a sacrificial electrode. The dimension of each electrode was 8.2 cm × 8 cm. The area of electrodes dipped into the TDE was 65.6 cm². A 1.5 cm gap was provided between the bottom of the reactor and the bottom of the electrodes to maintain easy stirring. The gap between two electrodes in EC reactor was maintained at varying range (1.5–2.5 cm). A magnetic stirrer was used for the mixing of TDE. The electrodes were cleaned with 20% diluted HCl followed by distilled water wash and drying. Thereafter, it was used for further experiments. The current density (36.72–146.88 A/m²) was adjusted by digital DC power supply (0–30 V, 0–5 A). The current density was controlled by using a regulator. At the end of the experiments, the sample was filtered by filter paper (Whatman 42 size, Maidstone,

Table 1
Electrocoagulation used for textile dyeing effluent

Author	Dye	Current density(A/m^2)	Anode – Cathode	% Removal efficiency
Kobya et al. [10]	Industrial textile effluent	6.5	Fe–Fe, Al–Al	85, 77
Golder et al. [11]	Trypan blue	–	Al–Ms, Al–Ms	95, 98
Khandega and Saroha [12]	Acid Red 131	62.5	Al–Al	97
Merzouk et al. [13]	Disperse red	20.8	Al–Al	95
Kobya et al. [14]	Remazol red 3B	15	Fe–Fe	>99

Table 2
Typical composition of TDE before and after treatment by EC at current density = 110.16 A/m^2 and electrode gap = 2 cm

Parameters	Textile dyeing effluent	EC treated TDE at optimum condition
COD	18,600	1,153.8
TDS	23,116	1,012
TSS	19,110	2,613
TS	42,226	3,625
Chloride	224	59
Phosphate	10	7
Total hardness	11,235	1,418
Sulfate	5,264	–
pH	10.1	6.0
Colour	Blackish brown	Light brown (transparent)
Absorbance at wavelength = 475 nm	0.834	0.225
Colour (PCU)	540	145.8

All value in mg/dm^3 except pH and colour.

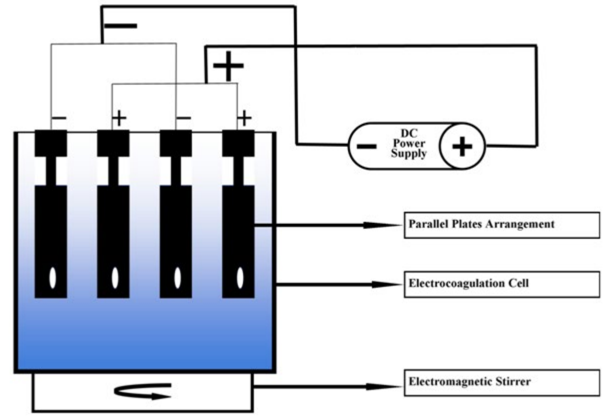

Fig. 1. Schematic diagram of the experimental setup.

Kent, England). The filtered liquid was used for the analysis of parameters (COD and colour). Settling studies were also performed in a 0.5 dm^3 measuring cylinder.

3. Results and discussions

3.1. Effect of pH

Initial pH of the wastewater played an important role during electro-coagulation treatment. It is a well-known fact that the optimum pH of the solution depends upon the type of effluent which is used for treatment [18]. Therefore, the results of experiments performed at varying pH (pH 2–10) at a current density 110.16 A/m^2 and electrode gap of 2 cm are presented in Fig. 2. It can be seen from Fig. 2a that percentage COD reduction increases from pH 2 to pH 6 and after that decreases. The maximum COD reduction of 93.8% was achieved at pH 6 and treatment time 105 min. By further increase in pH, COD reduction decreases. This may be because COD depends on the quality and quantity of hydroxide ions generated at a particular pH. TDE involves several organic compounds having own functional groups and passes negative charge that attached with a positive charge of Al. Furthermore, the quantity of electrode loss, the formation of Al^{2+}, Al^{3+}, $Al(OH)_2$, $Al(OH)_3$, and monomeric and polymeric hydroxide species varied with pH. At pH < 6 > pH, protons in the solution get reduced to H_2 which is responsible for less hydroxide ions production, leading to a lower COD removal efficiency.

The colour reduction is expressed as the percent decrease in the absorbance of the treated TDE sample for untreated samples at λ = 475 nm [19]. Colour reduction followed the pattern of COD reduction. It can be seen from Fig. 2b that the percentage of colour reduction increases from pH 2 to pH 6 and after then decreases. Also, maximum colour reduction 73% was found at pH 6 and treatment time 105 min. The decolourization is due to the settlement of dyes, and other organic components that separate from the effluent during electrocoagulation.

Table 3
Characteristics of EC cell

Electrodes	
Materials (anode and cathode)	Aluminium
Shape	Rectangular plate
Size	8 cm × 8.5 cm
Thickness (mm)	2
Plate arrangement	Parallel
Effective electrode surface area (cm^2)	68 cm^2
Reactor characteristics	
Make	Perspex glass
Reactor type	Batch mode
Dimensions (cm)	12 × 12 × 14
Volume (dm^3)	2.0
Electrode gap (mm)	20
Stirring mechanism	Magnetic bar
Power Supply	Direct current (DC)
Voltage range (V)	0–30
Current range (A)	0–5

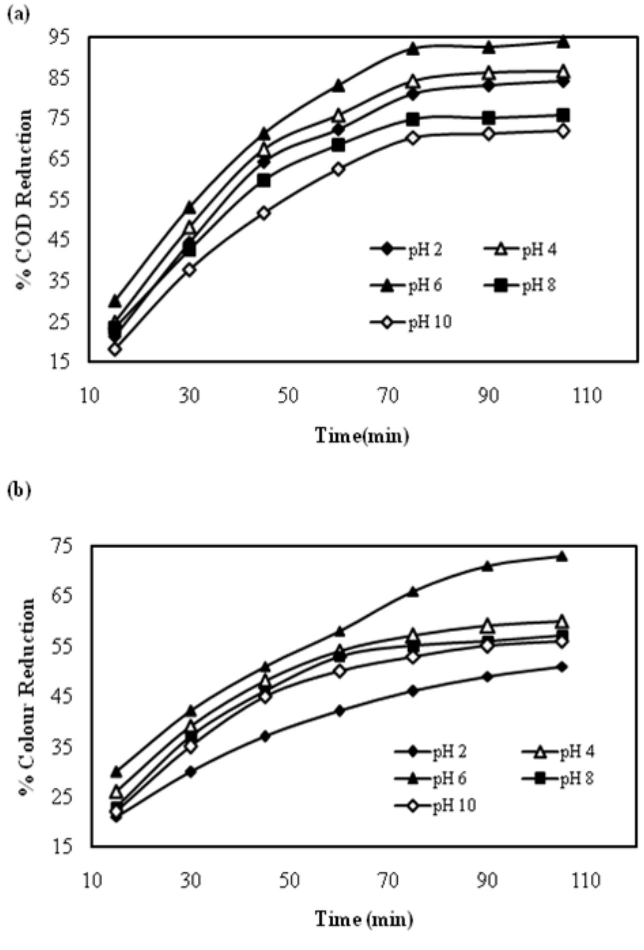

Fig. 2. Effect of pH on: (a) percentage COD reduction and (b) colour reduction, (COD$_i$ = 18,600 mg/dm^3, colour$_i$ = 540 PCU, current density = 110.16 A/m^2, electrode gap = 20 cm).

From the design point of view, electrical energy consumption (during the electrocoagulation treatment) is an important parameter which may be calculated by following Eq. (9),

$$\text{Energy consumption}\left(\text{Wh}/\text{dm}^3\right) = \frac{VIt}{\text{Treated volume}\left(\text{dm}^3\right)} \quad (9)$$

where V is the cell voltage in volt, I is the current in ampere (A), and t is the electrolysis time (h). Therefore, energy consumption during the electrocoagulation of TDE was calculated at varying pH (2–10), current density = 110.16 A/m^2, electrode gap = 2 cm and treatment time = 105 min and presented in Fig. 3a. The energy consumption of 10.9, 13.5, 16.09, 21.2, and 19.6 Wh/dm^3 TDE is noted at pH 2, 4, 6, 8, and 10, respectively. It is observed that energy consumption increases with an increase in pH. This may be attributed to the fact that at particular pH, a large number of flocks adhere to the surface of the aluminium electrode which is acts as an insulator and responsible to cause an increase in voltage [20,21]. The anode consumption at different pH was also calculated. Two anodes and two cathodes were used during the experiments. The values are shown in Fig. 3b at the similar operating condition and at pH 2, 4, 6, 8, and 10. At these pH anodes consumptions are 40.09, 39.06, 38.11, 33.86 and 20.15 mg/dm^3 of TDE treated. Also, the energy used per unit of COD reduction has been also calculated and presented in Fig. 3c. It may be calculated by the Eq. (10)

$$\text{Energy consumption}\left(\text{Wh}/\text{dm}^3\right) = \frac{VIt}{\Delta\text{COD}} \quad (10)$$

It can be seen from Figs. 3a and b that energy used per unit COD reduction has followed a similar pattern of energy consumption at different pH. This may be due to the dependency of amount of organics removal on the metal ions generates during the electrocoagulation process.

3.2. Effect of current density

Current density is a surrogate parameter during electrocoagulation and expressed as an electric current per unit cross-sectional area of electrode [22]. Therefore, experiments have been performed at varying current density (36.72–146.88 A/m^2) at pH 6 and an electrode gap of 2 cm and results are presented in Fig. 4. It can be seen from Figs. 4a and b the percentage COD and colour reduction increases with an increase in current density up to a certain limit thereafter it decreases. The maximum COD and colour reduction of 93% and 73% were achieved at optimum current density 110.16 A/m^2. Further, increase in current density up to 146.88 A/m^2 COD and colour reduction of 91.9% and 65%, respectively, were observed. It is a well-known fact that the amount of metal ions released during the process depends on the current density and the governing relation is expressed by Faraday's law [23].

$$m = \frac{IT_S M}{ZF} \quad (11)$$

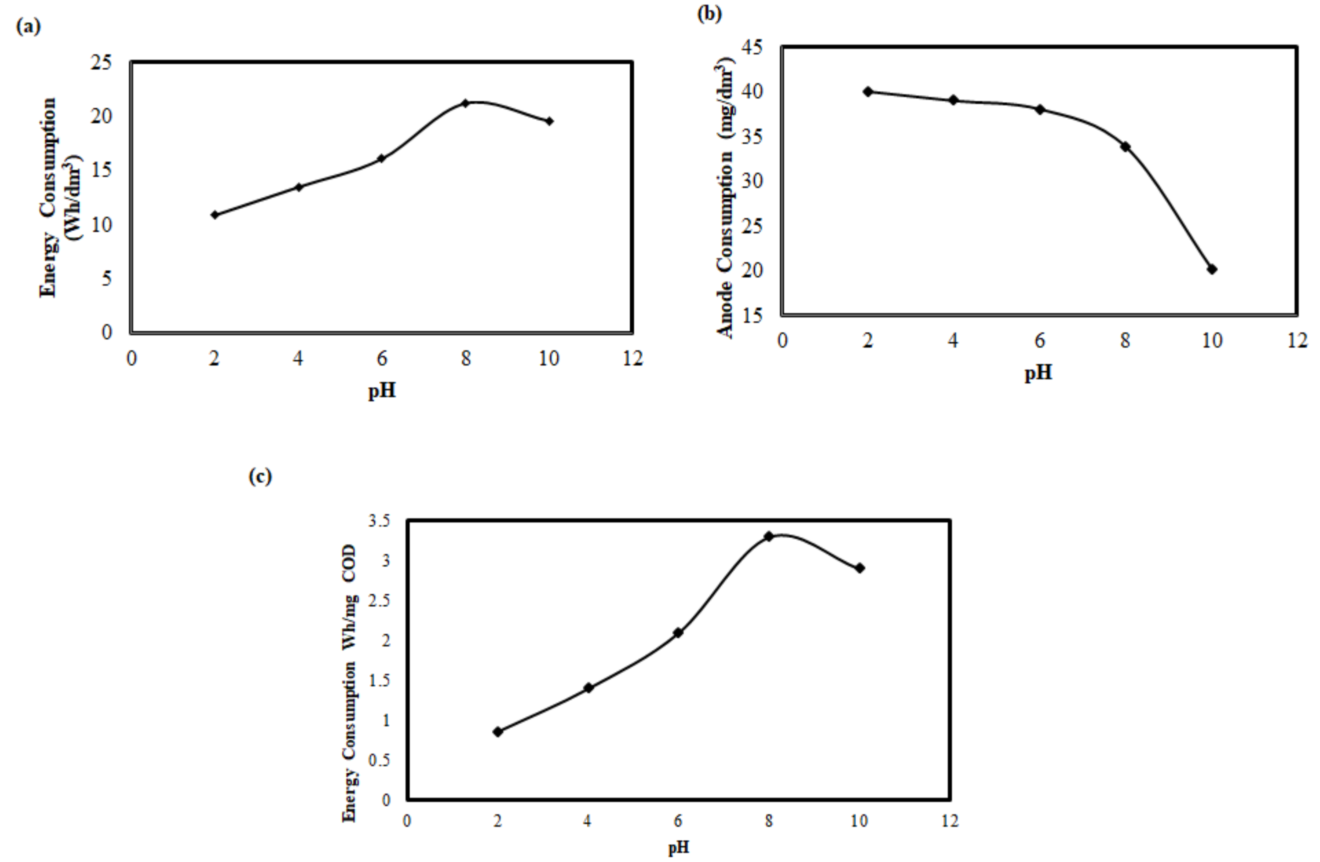

Fig. 3. Effect of pH on: (a) electrode energy consumption, (b) anode consumption, and (c) energy used per unit of COD reduction (COD_i = 18,600 mg/dm³, $colour_i$ = 540 PCU, current density = 110.16 A/m², and electrode gap = 2 cm).

where m = metal ions, F = Faraday constant, Z = valency of the ions m, I = applied current (A), T_S = treatment time, M = molar mass of electrode material. The removal of COD and colour from TDE is strongly depended on the amount of metal ions released during the process which added to the negative ions (i.e. organics have own functional group which possess net negative charge) consequently, heavier flocks generated which is a form of hydroxide and settle down through the gravity. Furthermore, an overdose of metal ions is responsible for a destabilization of the particle [24].

3.3. Effect of electrode gap

The inter-electrode gap during electrocoagulation process plays an important role. To observe the effect of electrode gap on COD and colour removal, experiments were performed at varying electrode gap (1.5–2.5 cm) with pH 6 and current density 110.16 A/m². It can be seen from Figs. 5a and b COD and colour reduction increase with an increase in electrode gap after then decreases. When the electrode gap between the anode and cathode is larger, resistance (R) offered by the cell increases. The concerned relationship is given as [25]:

$$R = \frac{g}{KA} \tag{12}$$

where K is cell-specific conductance and A is electrode surface area. If A is higher, g value between anode and cathode increases R. From Faraday law, the amount of oxidized metal decreases with higher g and consequently, the pollutant removal efficiency decreases. Maximum COD and colour reduction of 93 up to 2.5 cm COD reduction of 89.2 and colour reduction of 69% were observed. However, the minimum electrode gap between the two electrodes is more economical due to the less energy consumption. But it may prevent proper circulation of water.

3.4. Analysis of filtrate

The EC-treated TDE was filtrated by Whatman filter paper (42 size) and then analyzed for different parameters. The results are presented in Table 2. Around 93.8% COD and 73% colour has reduced at current density 110.16 A/m² (3A), (after treatment of TDE at optimized condition COD reduces to a value of 1,153.8 mg/dm³ from an initial value of 18,600 mg/dm³ and colour reduces to 145.8 PCU from 540 PCU). After treatment of TDE other pollutant such as chloride, sulfate, total hardness, and total solid are also reduced up to a certain limit. The treatment at higher current density 146.88 A/m² provided COD 1,506.6 mg/dm³ and colour reduction of 189.6 PCU which indicates overdosing of metal ions can destabilize the settled particles. The optimum result is achieved at pH 6 (i.e. 93.8% COD and 73%

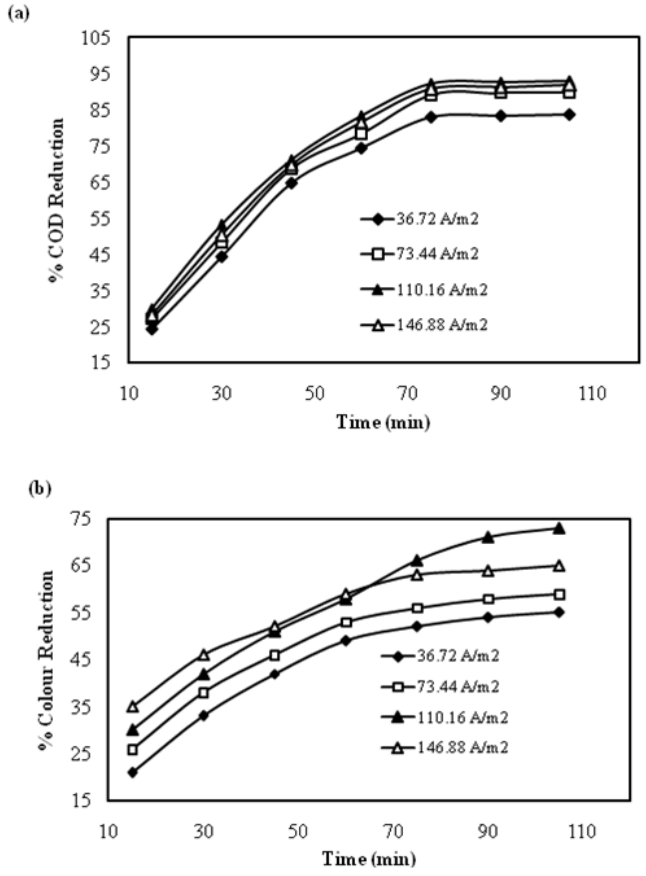

Fig. 4. Effect of current density on: (a) COD, and (b) colour removal of TDE (COD_i = 18,600 mg/dm³, $colour_i$ = 540 PCU, pH 6, and electrode gap = 2 cm).

colour reduction) with current density 110.16 A/m² at an electrode gap of 2 cm. The pH after the treatment of TDE is observed as pH 7. It is clear from the above observation that treated effluent cannot be directly discharged into land or any water receiving body due to the presence of a large amount of COD (1,153.8 mg/dm³) in the EC treated TDE. Hence, post-treatment is needed for this EC-treated TDE. Besides, the number of technologies and physico-chemical methods such as wet oxidation, thermolysis, coagulation, adsorption, and membrane separation are available for further treatment of EC-treated TDE [26]. For better results, TDE should first be treated by EC, which should be followed by membrane separation.

3.5. Settling characteristics

Electrocoagulated slurry was subjected to a sedimentation test. To find out settling characteristics, the electrocoagulation TDE sample was slowly mixed and then taken in a measuring cylinder with having a length of 36 cm and a diameter of 5.5 cm, respectively. The settling characteristics were conducted at varying (pH 4–10). At the pH 2 settling were not found hence it is not incorporated in Fig. 6. Fig. 6 shows the settling characteristics of obtained slurry in terms of the time vs. dimensionless height (H/H_0) as a function of settling time at different pH. Results indicated that at the beginning, the settling rate is slow (for short period) due to the Brownian motion of the particle; which is followed by a steady-state decrease in the height of the solid/liquid interface, commonly known as the zone-settling region [27]. At the end of the experiment, a compressed layer occurs on the bottom of the cylinder called the compression settling zone. The settling rate is found in order of pH 10 > pH 6 > pH

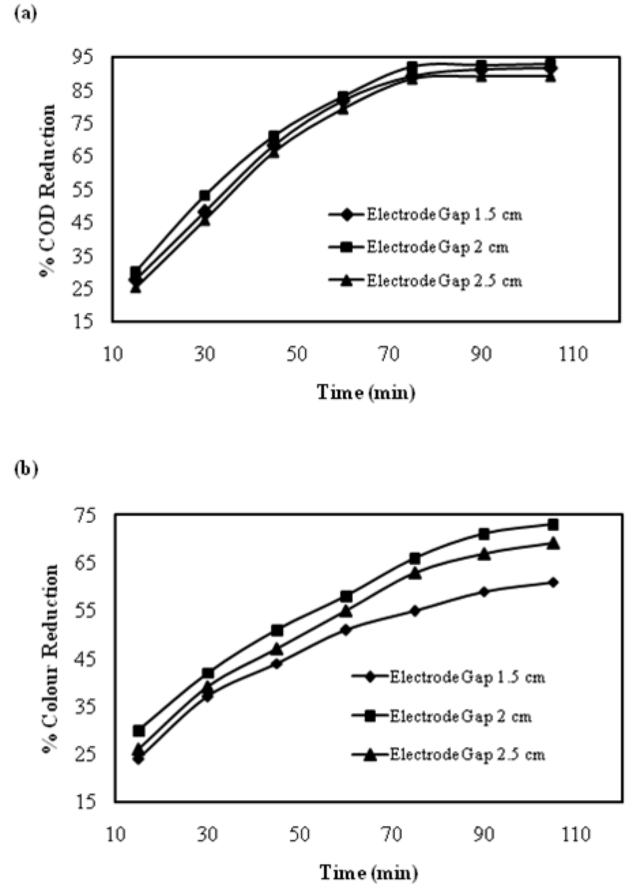

Fig. 5. Effect of electrode gap on: (a) COD reduction and (b) colour reduction. (COD_i = 18,600 mg/dm³, $colour_i$ = 540 PCU, and pH 6 current density = 110.16 A/m²).

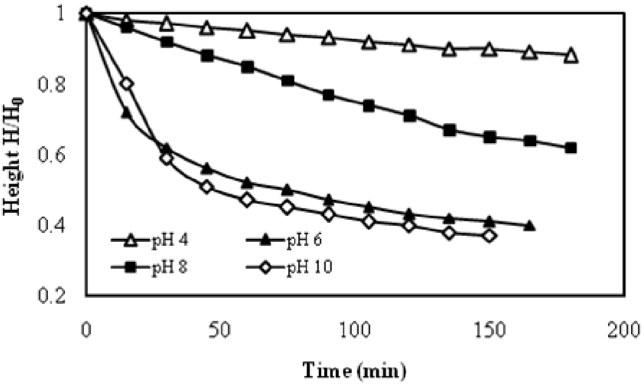

Fig. 6. Effect of pH on settling characteristics of the EC-treated slurry.

8 > pH4. Also, best settling was achieved at basic condition (pH 10) which decreases at the acidic condition (pH 4). This may be due to a basic pH that promotes the formation of heavy flocks that settle down. However, poor organic removal (i.e. COD) limits the EC treatment at pH 10. The method proposed by Richardson et al. [27] is mostly used to design a continuous thickener, which is oriented on batch studies.

3.6. Analysis of residue

To obtain the characteristics of solid residues it was first separated from the treated TDE and then it is dried at 105°C until its weight became constant. The detailed analysis of solid residues was also performed and presented in Table 4. It can be observed that the maximum weight of residue and foam is obtained at pH 6. Drying period lies between 12 to 20 h. Residues of EC treated TDE are hard and difficult to grind. Possibly these residues organics have good heating value [19].

Thermal degradation characteristics of TDE residue obtained after EC treatment were studied by thermogravimetric (TG), differential thermogravimetric (DTG), and differential thermal analysis (DTA) tests in oxidative environment. For the thermal characteristics of TDE, the residue was prepared as a dry solid mass by heating to 116°C. The TGA, DTA, and DTG curves of the catalytic thermolysis residue (CTR) for the medium heating rate of 50 K/min and airflow rate 200 ml/min is shown in Fig. 7. The TG curve for residue indicates that the loss of moisture components takes place (5.31% weight loss) within the temperature range of 28°C–400°C. The rate of weight loss was found to all most constant between 400°C and 900°C. In this temperature range, the sludge does not lose its weight slowly. The maximum rate of weight loss found during this period was 91.4 µg/min. In the third stage of oxidation in the temperature span of 452°C–980°C, only 1.01% weight loss was observed. At temperature 1,010°C, the organics of the residue got oxidized, leaving 93.58% ash fraction.

3.7. Material balance for aluminium

The electrocoagulation process is based on the dissolution of aluminium in the effluent during the treatment. Hence, a residue of the electrocoagulation process has been dried and dissolved under slow heating maintaining 90°C temperature for a complete dissolution of the entire residue. After suitable dilution, the metal content of the residue and the filtrate were determined by AAS. The material balance for aluminium at different pH is given in Table 5. It is demonstrated from Table 5 that the percentage error of aluminium balance varied from 0.49% to 1.03 %, this may be due to the clinging of a small amount of aluminium on the surface of the reactor.

3.8. Economic analysis

The cost analysis was estimated for a plant capacity of 100 m^3/d of wastewater and is presented in Table 6 based on operating cost. The operating cost was calculated using the following Eq. (13) [28]

$$\text{Operating cost (US \$/m}^3) = aC_{\text{energy}} + bC_{\text{electrode}} \tag{13}$$

where C_{energy} is the overall energy consumption (kWh/dm^3) (including power supply and mixing of effluent) and

Table 4
Analysis of residue obtained after EC at different pH

Analysis of residues					
Parameter	At pH 2.0	At pH 4.0	At pH 6.0	At pH 8.0	At pH 10
Weight of residue (kg/m^3)	10.5	25.7	35.36	28.8	32.6
Colour	Blue	Blue	Blue	Blue	Blue
Nature	Bulky mass, difficult to grind	Bulky mass, difficult to grind	Bulky mass, difficult to grind	Bulky mass, difficult to grind	Bulky mass, difficult to grind
Approximated drying period	14	19	16	12	20
% convertible COD	84.1	73.8	93.0	75.6	71.8

Table 5
Material balance for aluminium

pH	Total aluminium in electrocoagulation sludge	Total dissolved aluminium in treated TDE (filtrate)	Total dissolved aluminium in residue	% Error
2	40.09	2.13	37.68	0.69
4	39.06	1.95	36.71	1.02
6	38.11	1.90	35.82	1.02
8	33.86	1.69	31.82	1.03
10	20.16	1.12	18.94	0.49

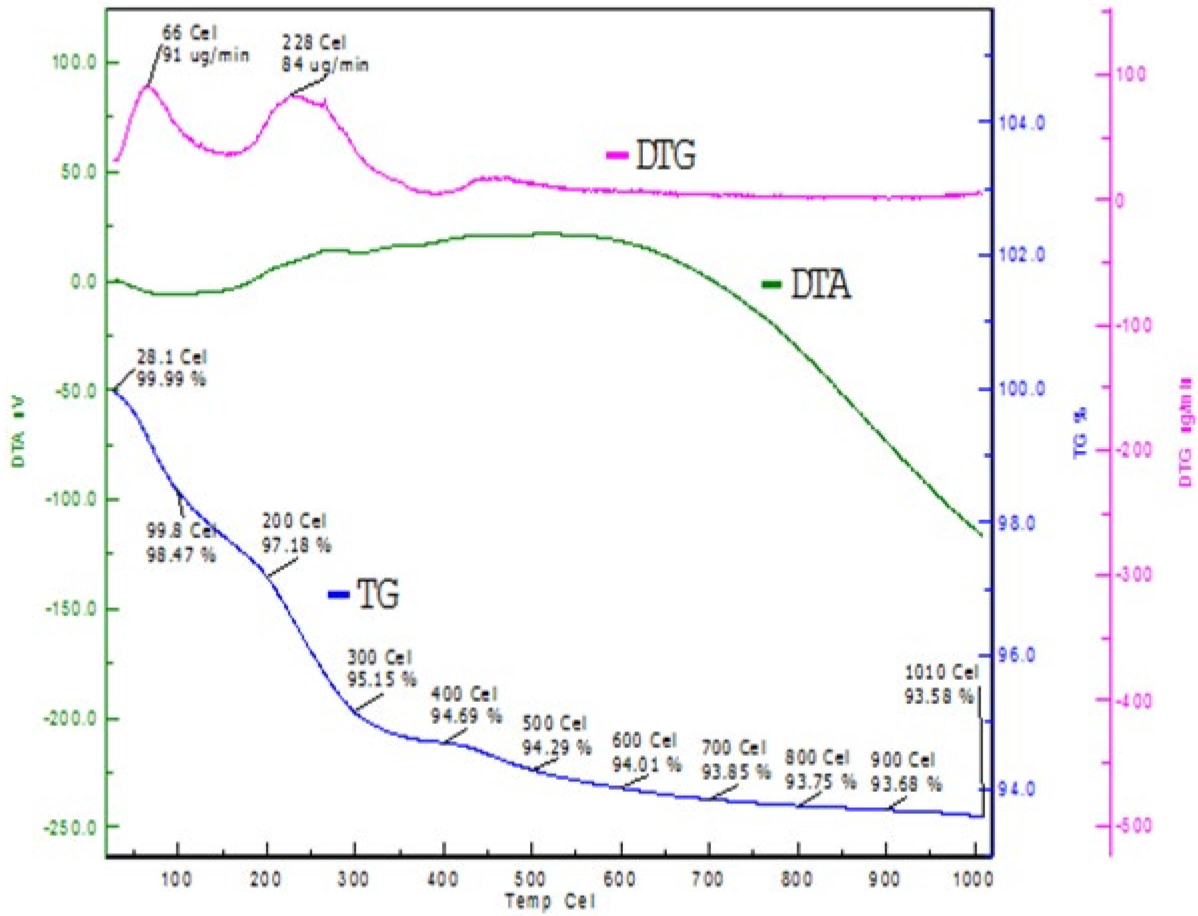

Fig. 7. TG, TGA, and DTA of EC-treated TDE sludge. Air rate, 200 mL/min.

Table 6
Economic data used for calculating the operating cost

Item	Cost ($)
Rectifier installing cost	920
Electrocoagulation tank installing cost	120
Maintenance (m^{-3})	0.04
Electricity (kWh^{-1})	0.15
Labor costs (m^{-3})	0.07
Sludge transportation and disposal cost (kg^{-1})	0.02
Material and chemical cost	
Al electrode (kg^{-1})	3.5

$C_{\text{electrode}}$ is the electrode consumption (kg/m^3) of wastewater treated. Unit prices a, b and c are given for the India market, are as follows: (a) electrical energy price 0.15 US $/kWh and (b) electrode material (Al) price 3.5 US $/kg. It is demonstrated in Fig. 3a, the energy consumption varied from 10.9 to 21.2 kWh/m^3 with pH 2–10. Under optimum conditions such as 110 A/m^2 current density, pH of 6, 120 rpm, 28°C temperature and 2 cm electrode gap, the energy and electrode was 16.05 kWh/m^3, 0.38 kg/m^3, respectively. The operating cost was calculated under the optimum condition and it was found to be 2.1 $/m^3. Also, for better results, advance oxidation process (AOP) may be used where H_2O_2 is required. In the Indian market, the cost of H_2O_2 is around 18.37 $/m^3; however, in this study, AOP has not been used in practice.

4. Conclusion

The EC process is found significant to reduce COD and colour from the TDE in a considerable amount in the optimum operating condition (pH 6, current density = 110.16 A/m^2 and electrode gap = 2 cm). It was found that COD and colour reductions increase with increasing current density up to a certain limit and after then decrease. Maximum COD reduction of 93.8% and colour reduction of 73% were achieved at optimum operating conditions in the cost of the energy consumption 16.055 Wh/L, whereas the electrode weight loss 38 mg/L. The settling characteristics of treated TDE were found best at pH 10. After the treatment process residues obtained may be used as fuel for a different purpose. Even at optimum COD and colour reduction the treated effluent still contains high organic load (12,180 mg/dm^3). Therefore, the post-treatment of the effluent is required. Electrocoagulation followed by membrane separation process may be used for the complete treatment of TDE.

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