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Hybrid dual treatment of real textile wastewater using electrochemical treatment (ECT) and membrane bioreactor (MBR)

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ABSTRACT

Raw real silk textile wastewater was treated using batch electrochemical coagulation (ECC) method. Iron and aluminum electrodes in monopolar parallel arrangement were using a specific electrode area to volume of 24 m^2/m^3 and a cell voltage of 6 V as the optimal operating condition. Chemical oxygen demand removal ranged from 88% to 92% for iron and aluminum electrodes. Settleability and filterability characteristics of ECC-treated wastewater were at ease. UV–visible spectral analysis was carried out to evaluate the efficiency of ECC treatment for color removal from textile wastewater. An electrolysis time of 10–20 min was sufficient to remove maximum apparent color from the solution. The sludge obtained after electrochemical treatment (ECT) was analyzed for its composition by energy-dispersive spectroscopy. The supernatant from ECT after sludge settling was further treated using membrane bioreactor (MBR) for removal of trace pollutants/solids residues. The results proved the suitability of the dual hybrid treatment process for the complete removal of pollutants/contaminants combining ECC and MBR with ~75% clear water reclamation.

Keywords: Silk textile wastewater; Electrochemical coagulation; Fe and Al electrodes; Membrane bioreactor; Filterability

1. Introduction

Wastewater from textile mills varies widely in terms of composition as a result of chemicals and dyes used in different processes. Over 100,000 commercially available dyes are used in dyeing fabric globally with an estimated annual fabric production of over 7×10^5 tons [1]. More than 50% of these dyes are found in the effluent [2] because of their partial adsorption on the fabric fibers [3] resulting in pollution/contamination of the receiving water and land attributes. Textile industry wastewater consists of high organic and inorganic loads, unreacted dyestuff, pH and temperature variations [4]. Dyes are mainly responsible for the recalcitrance of textile effluents. Discharging untreated wastewater containing synthetic dyes to the fragile environment leads to serious complications such as interference with the penetration of sunlight and also causes carcinogenic effect in human beings [5]. Changes of the dyestuff employed in the process cause considerable variation in the wastewater characteristics showing intense color, high chemical oxygen demand (COD), dissolved solids and highly fluctuating pH [3]. Natural degradation of synthetic dyes in the aquatic environment is a very slow process, and by its persistence, it is necessary to eliminate synthetic dyes from wastewater before disposing to water bodies/streams. At present, there is no single-treatment technology effectively applicable for the treatment of a consortium of textile wastewater (TWW) [6].

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Textile industry consists of variety of machineries and processes to produce the required shape and properties of the product. Large volume of effluent is generated in the various processes such as sizing, scouring, bleaching, mercerizing, dyeing, printing and finishing. Auxiliary chemicals, residual dye, salts, acid/alkali and cleaning solvents are the main constituents of effluent. Dyes are considered carcinogenic agents and should be removed from the discharge conduits of textile processing industries [7]. More than 22,700 kg of salts are released per week for 181,600 kg of cotton processing. Typically, the effluent contains ~3,000 mg/L of salt concentration [8]. The salts in the effluent cause soil infertility and damage aquatic life [2]. Conventional chemical and biological treatment techniques have many disadvantages—low efficiency, uncontrolled sludge production and disposal problems [9]. Therefore, a proper complete treatment of such toxic wastewaters is an economical and technical challenge.

Electrochemical coagulation (ECC) as a treatment process involves electrical generation of metal charged ions using suitable electrodes in the wastewater being treated. ECC involves generation of charged in-situ coagulants from metal electrodes. The metal ions so generated at the sacrificial anode migrate and search for negatively charged colloids and suspended solids finally being removed by 'sweep flocculation'. The flocs formed during ECC float at the top of the electrochemical reactor with gases entrapped in it.

ECC treatment is seen applied on treating a variety of wastewater viz., pulp and paper mill wastewater [10], restaurant wastewater [11], poultry slaughter house wastewater [12], etc. ECC is also used for treatment of synthetic and real TWWs stating by few researchers that COD removal depends on the chloride concentration and initial pH [13–15]. ECC is able to deliver high removal efficiencies of color, COD and biochemical oxygen demand (BOD) more efficient than traditional coagulation processes. ECC is inexpensive compared with other treatment methods viz., ultraviolet (UV) and ozone [16]. The residues remaining after electrochemical treatment (ECT) require further refinement in water quality for which membrane bioreactor (MBR) was proposed down line the ECC unit. ECC preceding the MBR unit reduces sludge production and membrane fouling [17,18].

This work was carried out with a purpose of treating TWW combining ECC and MBR. The aim was to remove COD and color from TWW applying ECC using iron (Fe) and aluminum (Al) electrodes. After maximum removal of pollutants/contaminants, the ECC supernatant excluding sludge was transferred to another container having a polyvinylidene fluoride (PVDF) membrane with a pumping mechanism to obtain ~75% clean water reclamation.

2. Material and methods

Textile manufacturing units exhibit both diurnal fluctuations in both flow (Q_{av}) and strength (mg/L) depending on the processing and operations taking place. Raw TWW for use in all the experiments was collected from a local textile manufacturing industry in Mysore, Karnataka. Raw TWW samples were collected from the conduit just before the equalization tank of the existing effluent treatment plant and stored in polyethylene containers. During each sampling ~40 L of raw TWW was collected and used in the ECC experiments.

All the chemicals used in the study were of analytical grade. Iron (cold rolled) and aluminum sheets were used as electrode materials. The electrode materials were cut into rectangular shape with dimensions of 15.5 cm × 9 cm. These electrodes were degreased manually and treated with 15% hydrochloric acid (HCl). The wastewater samples were analyzed for their physicochemical parameters as per standard methods [19]. Raw TWW had various shades of blue color due to the presence of various mixtures of dyes such as Telon Turquoise, Reactive Blue, Turquoise Blue, etc. The characteristics of the raw TWW are shown in Table 1. The raw TWW showed high concentrations of COD, solids and chlorides. The COD to BOD ratio was as high as 5.15–6.299 indicating the investigators of non-amenability of TWW to biological treatment, and henceforth ECC was chosen to be a novel treatment option.

TWW usually contains high fiber depending on the source of the raw material. During processing, most of the thin fiber content ends up into the wastewater stream increasing the total solids (TS) contents. The samples were collected after the grit chamber, and therefore high solids were seen in the raw wastewater. The raw wastewater without any further modification/filtration was used directly in the ECC experiments. The schematic of the experimental setup used in the experiments is shown in Fig. 1. The experiments were carried out in a cubical shape reactor made of Perspex acrylic material with necessary inlet and outlet arrangements with a capacity of 3 L having provision to accommodate a maximum of 14 plate electrodes. The batch reactor was operated under completely mixed conditions using a magnetic stirrer provided at the bottom of the reactor. The electrode spacing was maintained at 1 cm using cut plastic tubes in between the electrode faces using non-conducting wires. The anode and cathode plates were positioned vertically and parallel to each other.

Experimental conditions such as number of electrodes, voltage and electrolysis time (ET) were optimized. Preliminary studies were conducted before actual experimental run to fix the optimum operating conditions for both Fe and Al electrodes. It was decided to choose four electrodes with the corresponding applied cell voltage of 6 V as optimum conditions based on earlier preliminary ECC studies. Varying quantities of coagulant aid polyacrylamide

Table 1
Characterization of raw textile wastewater before use in batch ECC experiments

Parameters	Range
pH	6.30–6.90
COD, mg/L (unfiltered)	1,088–1,600
BOD, mg/L	211–254
COD:BOD	5.15–6.299
Nitrates, mg/L	107–109
Phosphates, mg/L	17.22–19.76
Chlorides, mg/L	900–1,100
Conductivity, m Mho/cm	3.1–3.4
Turbidity, NTU	210–230
Total solids, mg/L	2,600–2,800

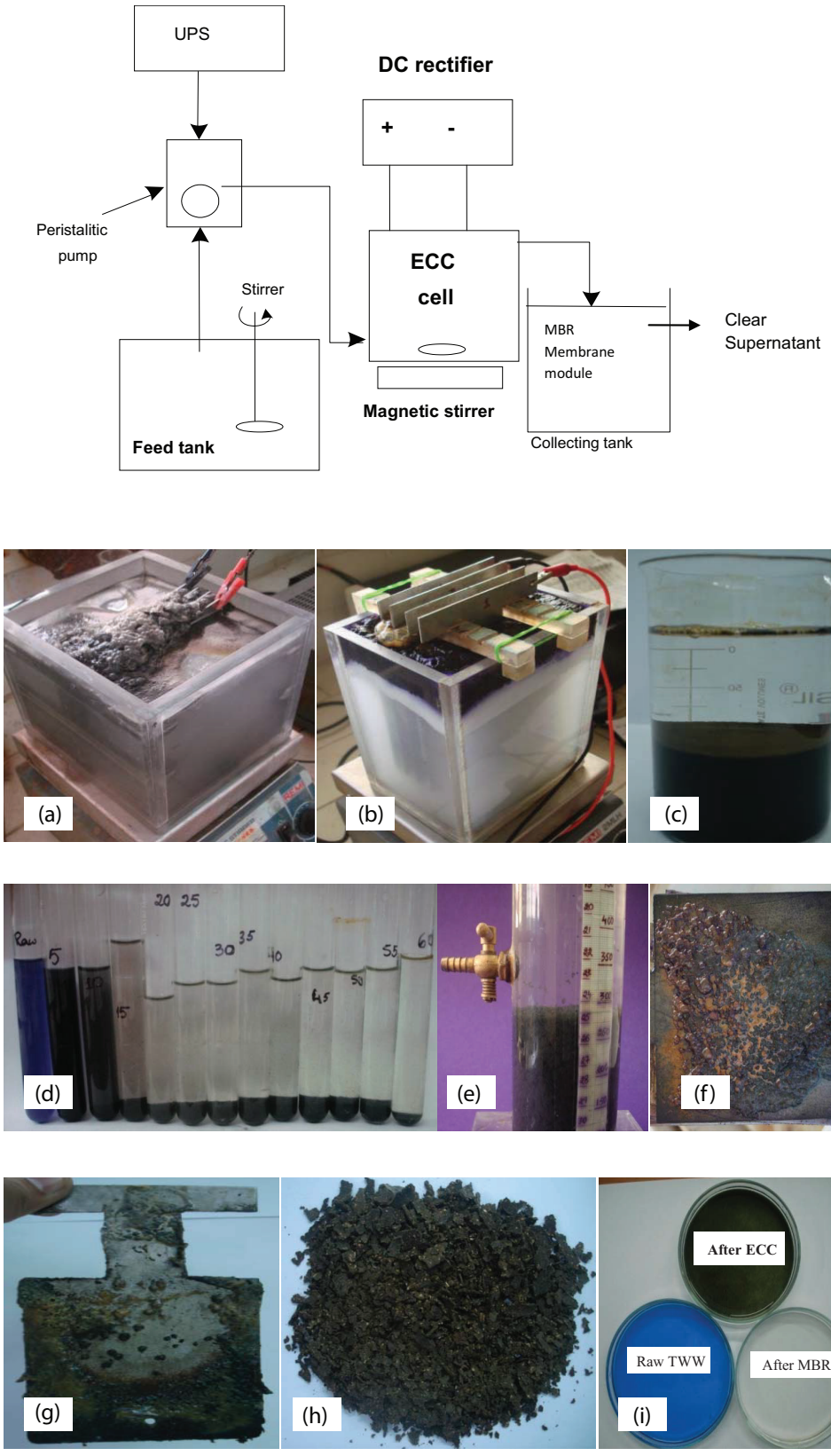

Fig. 1. Schematic of the experimental setup showing ECC + submerged MBR: (a) during ECC of TWW, (b) after ECC (see sludge flotation showing clear water reclaimable) (c) water reclamation after ECC, (d) samples retrieved during ECC of TWW, (e) post ECC slurry in settling column, (f) electrode condition after ECC, (g) flake deposits on anode plates just after ECC, (h) dry sludge after ECC, (i) raw TWW, after ECC, after MBR.

(PAA) (20, 50, 100 and 150 mg/L) were added into the reactor during ECT. All experimental runs lasted for a maximum ET of 75 min. The samples were drawn at regular intervals of 10 min and at the end of the ECC process. All samples were filtered and analyzed for pH, conductivity and COD. The settling and filterability studies of post ECC slurry were also carried out.

The supernatant after ECT excluding the sludge was treated using MBR for further removal of pollutants/solids residues. The membrane-holding unit made up of Perspex material of size 12 cm × 5 cm × 17 cm (L × W × D) was used in the MBR experiments. Tubular PVDF membrane having pore size 0.1 μm, membrane area 0.5 m^2 and size 13 cm × 16 cm (W × H) was used for all the experimental sets. The MBR process was initiated at an initial mixed liquor suspended solids ($MLSS_0$) concentration of 5 g/L, acclimatized overnight. The biomass was provided with oxygen using air diffuser grids. The supernatant from batch ECC was pumped through the membrane in submerged condition. The main operating conditions are summarized in Table 2.

3. Results and discussion

3.1. ECC with and without coagulant aid

Figs. 2(a) and (b) show COD degradation for Fe and Al electrodes with/without the addition of coagulant aid PAA. PAA was added in the dosage of 20, 50, 100 and 150 mg/L. Shen et al. [20] obtained increased color removal with increase in electrolyte concentration for an applied cell voltage >3 V. In this work, for an applied PAA dosage of 50, 100 and 150 mg/L, the COD concentration decreased to ~64 mg/L (~95% removal) from its initial value of 1,350 mg/L using Fe electrodes. Daneshvar et al. [21] reported that color removal directly depends on hydroxyl and metal ions produced during ECC. During ECC, anode dissolution increased slightly with the increase in PAA dosages. For Al electrode, the COD value decreased to a value of <64 mg/L (~95% removal) from its initial value of 1,344 mg/L for an applied dosage of 20, 100 and 150 mg/L. Color removal was much quicker within 10 min ECC for all coagulant dosages than COD removal indicating that electrochemical degradation of azo bond is the first step in treatment process [22,23]. Without aid, complete decolorization was achieved at 20 min ET with an increase in temperature of the bulk solution by 7°C–8°C at 75 min ET.

Fig. 3 shows comparison between iron and aluminum electrodes for COD degradation. Though COD removal efficiency (90%) remained same for both the electrodes after 75 min ET, color removal was achieved within 10 min ET for iron electrodes, whereas for Al electrodes it required 20 min ET for the removal of apparent color (Fig. 4).

Table 2
Operating conditions for submerged MBR module

Parameter	Value
Hydraulic retention time (HRT), h	6
Biomass concentration, g MLSS/L	5
Food/microorganism ratio (F/M), kg COD/(kg MLSS d)	0.15
Operational pH	7.5
Solid retention time (SRT), d	>20
Volumetric loading, kg COD/(m^3 d)	15
Transmembrane pressure, kPa	22
Flux, L/(m^2 h)	5
Operational temperature, °C	26

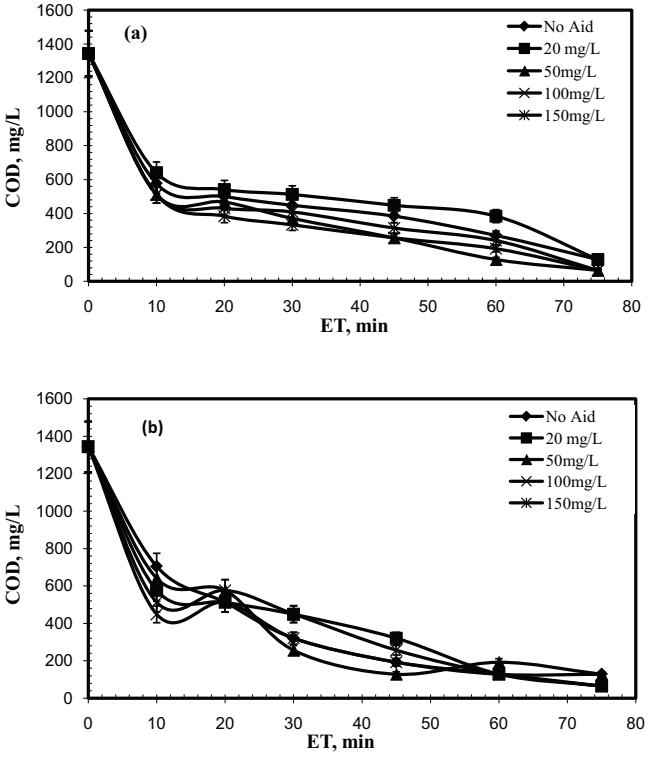

Fig. 2. COD degradation curves for varying dosage of coagulant aid PAA using Fe and Al electrodes for 6 V: (a) iron electrodes and (b) aluminum electrodes (COD_0: 1,350 mg/L, pH: 6.85, number of electrodes: 4).

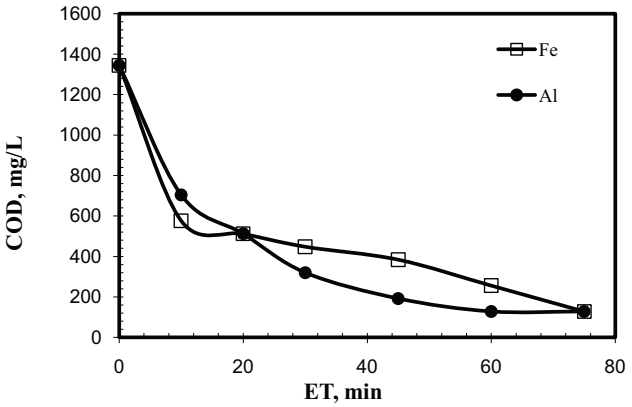

Fig. 3. Comparison of COD degradation curve for iron and aluminum electrodes for 6 V and four electrodes at a specific electrode area to volume (SEA/V): 24 m^2/m^3 and COD_0: 1,350 mg/L.

Complete decolorization was achieved within 60 min ET while using Fe electrode. Vlyssides et al. [24] achieved complete color removal; Naumczyk et al. [25] and Korbahti and Tanyolac [23] obtained maximum color removal after 30 min ET. During ECT, pH increased from 6.84 to 8.93 for Fe electrodes and 6.84 to 7.94 for Al electrodes. The anode dissolution of the iron electrode was nearly twice that of aluminum electrode as shown in Fig. 5, and a marginal increase in electrode dissolution is observed with increase in aid for Fe electrodes.

3.2. Settleability and filterability characteristics of post ECC slurry

Figs. 6(a) and (b) show the settleability curves for varying dosages of PAA and for no aid conditions. Settling characteristics improved with the addition of aid for iron electrode but was poor for aluminum electrodes. The settleability column appeared dark green color for Fe electrode and whitish blue when using Al electrodes.

Higher PAA doses of 50–150 mg/L showed quick settling of ECC flocs in the settling column for Fe electrodes. The bubbles entrapped in the Al floc matrix take more time to settle as seen in Fig. 6(b), and the settling begins after 15–20 min of settling time. It takes 3–4 shuffling of the column slurry to separate gas bubbles from the sludge before the plot in

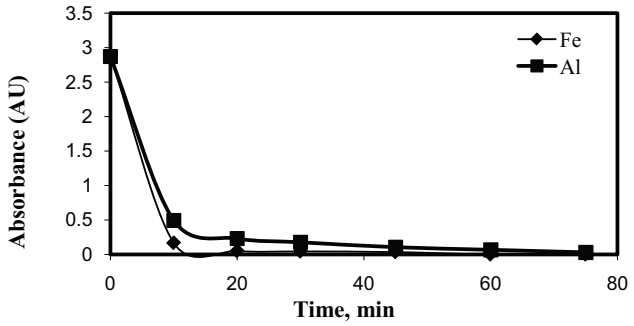

Fig. 4. Comparison of absorbance of iron and the aluminum electrodes for 6 V and four electrodes at specific electrode area to volume (SEA/V): 24 m^2/m^3 and COD_0: 1,350 mg/L.

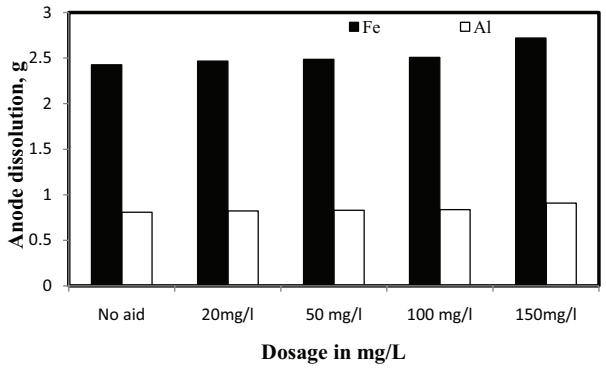

Fig. 5. Anode dissolution for iron and aluminum electrodes for 6 V and four electrodes at specific electrode area to volume (SEA/V): 24 m^2/m^3 and COD_0: 1,350 mg/L.

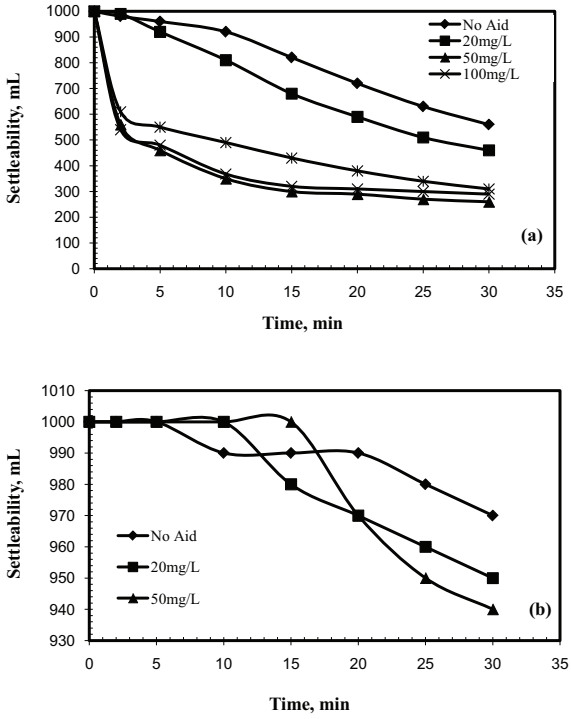

Fig. 6. Settleability of ECC slurry for varying dosage of PAA during batch ECC at optimal operating conditions of four electrodes and 6 V for Fe electrodes and Al electrodes: (a) settling curves for iron electrodes and (b) settling curves for aluminum electrodes.

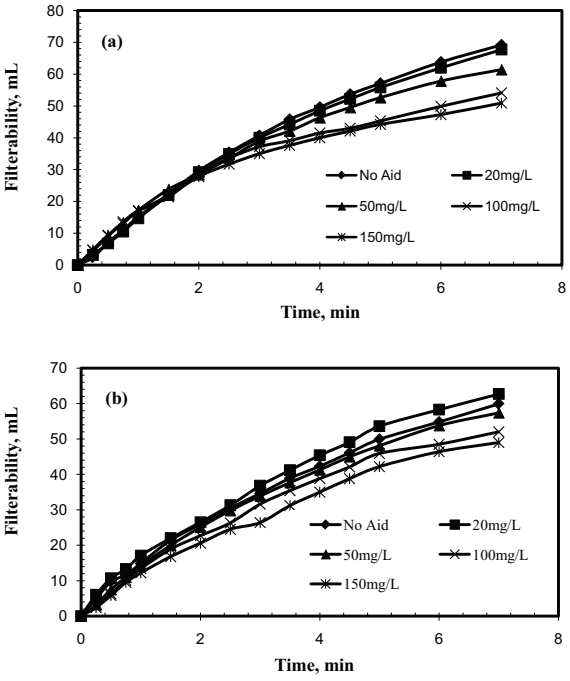

Fig. 7. Gravity filtration of post ECC slurry for varying PAA doses using Fe and Al electrodes at optimal operating conditions of four electrodes and 6 V: (a) filterability of post ECC slurry with iron electrodes and (b) filterability of post ECC slurry with aluminum electrodes.

Fig. 6(b) is obtained. PAA-Al combination electrode showed poor settling characteristics.

Figs. 7(a) and (b) show the filterability curves of post ECC slurry for Fe and Al electrodes. The filterability rate was maximum without aid, and it slowly decreased with the increase in PAA dose (50, 100 and 150 mg/L). The post ECC slurry showed gelatinous behavior, and hence filterability rate was marginally low.

3.3. Characteristics of post-ECC-treated wastewater

The TWW was characterized for various physicochemical parameters after ECC treatment. Table 3 shows the characterization for both Fe and Al electrodes. COD removal ranged from 88% to 92% for both the electrodes. The pH of the treated wastewater increases by ~1.5 units in both cases. The TS, nitrates and phosphates were reduced to the required pollution control discharge standards. The BOD values were between 150 and 168 mg/L for both Fe and Al electrodes demanding secondary treatment applying MBR.

Most of the excess water quality parameters reduced after treatment with MBR. The BOD values were <30 mg/L for both Al and Fe electrodes except for TS which were removed ~50%.

3.4. Spectral analysis showing color removal before and after ECT

The spectral analysis for silk TWW before and after the batch ECC process for different ET is shown in Fig. 8. Changes in the absorbance were observed over a wide range of wavelength (500–1,000 nm) during the ECC process. As observed from Fig. 8, multiple peaks are observed for raw untreated wastewater indicating heterogeneity of raw TWW with high color, organics and solids.

It may also be observed that at 75 and 90 min ET, all peaks are seen nullified showing a smooth curve proving the removal of solid particles in different forms. As the electrolysis proceeds, the peaks disappear at 75 min ET marking the end of the ECC treatment process. This proves the effectiveness of ECC for complete removal of color and COD from raw TWW.

Table 3
Characteristics of raw TWW and post ECC supernatant using Fe and Al electrodes

Parameters	Before treatment	After ECC treatment	
		Fe electrodes	Al electrodes
pH	6.30–6.90	8.2–8.5	7.9–8.0
COD, mg/L	1,088–1,600	128	128
BOD, mg/L	211–254	150	168
COD:BOD	5.15–6.30	1.17	1.31
Nitrates, mg/L	106.58	21.05	19.90
Phosphates, mg/L	17.22	2.32	1.09
Chlorides, mg/L	899.74	837	784
Conductivity, m Mho/cm	3.1	3.0	2.90
Turbidity, NTU	210	56	51
Total solids, mg/L	2,600	1,670	1,420
Total dissolved solids, mg/L	2,286	446	418

3.5. Sludge composition analysis using EDS

Sludge composition was analyzed through energy-dispersive spectroscopy (EDS) as shown in Fig. 9.

Iron, chromium, nickel, carbon, calcium and oxygen by wt% were the main elements present in ECC sludge for applied voltage 6 V (Fig. 9) showing Fe: 43.03%, C: 17.64%, Cr: 16.41%, O: 8.70% and Ni: 5.39%. Other minor elements were Cl^-, S, Si and Mg between 1% and 2%. Na and P were also detected at <1%.

3.6. Membrane filtration of ECC-treated wastewater

The filtration process was carried out for a flow rate of 2.5 Lph. The permeate from membrane filtration unit was then analyzed to assess the change in water quality parameters such as COD, BOD, total suspended solids (TSS), TS and total dissolved solids (TDS). The reject obtained was only 16% while the permeate was 84% proving that ECC as a pre-treatment to MBR provides a larger percentage of clean reclaimable water, with all water quality parameters much below the natural baseline values of aquatic systems. The parameters TS, TSS, TDS and COD were analyzed for the membrane-treated effluent to attain the desired water quality. Table 4 shows the percentage removal of organics from TWW for both ECC and MBR.

The initial COD of TWW was 1,350 mg/L before ECC and was reduced to 42 mg/L after MBR treatment. ECC showed removals of 90.5% COD, 49.9% BOD, 35.7% TS, 17.2% TSS and 80.4% TDS, respectively. Both ECC followed by MBR showed removal of 97% COD, 86% BOD, 96.5% TS, 100% TSS and 96% TDS.

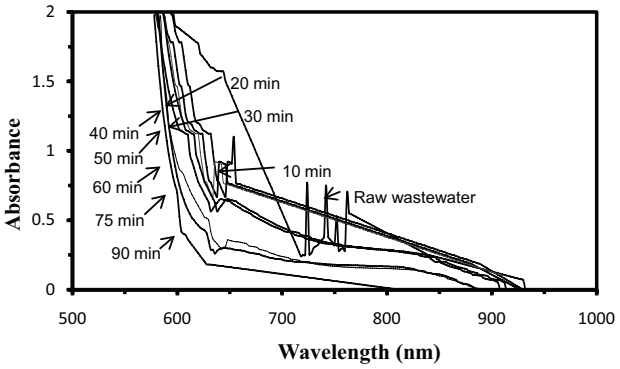

Fig. 8. UV–visible spectra before and after ECT of raw silk textile wastewater.

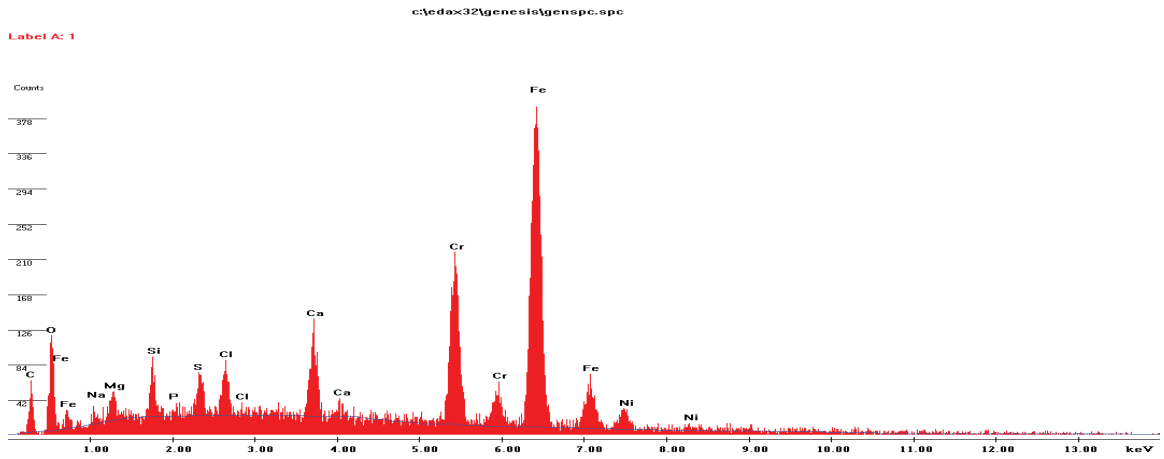

Fig. 9. Dry sludge composition after ECC using EDS for an applied cell voltage of 6 V.

Table 4
Removal of organics from textile wastewater in ECT and MBR

S. No.	Parameters	Raw TWW Values	ECC supernatant		ECC + MBR permeate	
			Values	% Removal	Values	% Removal
1	COD, mg/L	1,350	128	90.5	42	97
2	BOD, mg/L	254	150	49.9	36	86
3	TS, mg/L	2,600	1,670	35.7	90	96.5
4	TSS, mg/L	314	260	17.2	0	100
5	TDS, mg/L	2,286	446	80.4	90	96

4. Conclusions

Batch ECC experiments were carried out using Fe and Al two-dimensional electrodes for an ET of 75 min. Iron electrodes with an SEA/V of 24 m^2/m^3 showed maximum 90% COD removal and 100% color removal at 75 min ET. A 10 min of ET was sufficient in removing apparent color when using Fe electrodes. The pH of the treated effluent increased to ~8 from its initial value of 6.45, but showed a receding value after a small time lapse after ECT. The TS, nitrates and phosphates were reduced to the standards prescribed by the pollution control authorities. The chloride values remained the same even after ECC but well within the discharge limits of 1,000 mg/L for both Fe and Al electrodes. Fe electrodes showed good settling properties ascribed to well-defined morphology of iron hydroxide sludge particles having higher density. Fe electrodes showed good filterability providing clear water compared with Al.

Though Al electrodes with an SEA/V of 24 m^2/m^3 showed maximum COD removal of 90% at 75 min ET, it required 20 min ET for the removal of apparent color. PAA aid and Al combination electrodes showed poor settling characteristics compared with Fe.

The combined effect of both ECC followed by MBR showed removal of 97% COD, 86% BOD, 96.5% TS, 100% TSS, 96% TDS and 100% color. The results proved the suitability of the dual hybrid treatment of ECC and MBR for the effective removal of organics from TWW avoiding a long treatment train of unit operations and unit processes in wastewater treatment facilities.

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