
Effect of electrochemical operational parameters on decolourisation of reactive red-195 using batch process

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Abstract: Reactive red-195 synthetic solution of dye was investigated over different operational parameters in simple electrochemical cell for decolourisation. Effect of operational parameters such as variation in applied electrical potential, electrolyte concentration, dye concentration, inter electrode distance (S), cathode material, H₂O₂ dose and electrolysis time were analysed separately in batch process using iron electrodes. Potential of 20 volts is optimised and applied to successive experiments, considering colour removal efficiency and energy consumption. 100% colour removal was achieved in 4 min for H₂O₂ dose of 6.0ml/L and 2.5gm/L of NaCl respectively. Similarly increase in gap between electrodes to 4.0 cm and concentration of dye to 100 mg/L, process required 18 min to achieve 100% colour removal. Fe-Al and Fe-Steel combination gave good results at initial, but Fe-Fe and Fe-Al gave 100% colour removal at earliest, compared to Fe-Steel. Electrochemical process may prove efficient in such treatments.

Keywords: anode electrode material; cathode electrode material; dye concentration; electrocoagulation; H₂O₂ dose; inter-electrode distance; batch reactor; applied voltage; electrolyte concentration; colour removal.

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1 Introduction

Most of textile wastewater contains high suspended solids, COD, BOD, pH and strong colour from dyes (Kadir and Zuhail, 2009). The dye industry produces thousands of dyes (Magdalena et al., 2009) and over 80,000 tons of reactive dyes are consumed each year in industries with extent of their ability to pollute (Colindres et al., 2011). These dyes are toxic and carcinogenic to human (Belkacem et al., 2010). The cost effective removal of dye from textile effluents still remains a wide mouth to problem (Ravikumar et al., 2007). The synthetic azo dyes extensively used in textile and dyestuff industries are considered recalcitrant xenobiotic compounds due to presence of $-N=N-$ bond and groups such as aromatic rings which are not easily degraded (Maria et al., 2011). Methods which are studied and experimented on colour removal from wastewater are biosorption (Sibel et al., 2009), Fenton (Chia-Chi et al., 2011), photo-catalyst (Subash et al., 2013), activated carbon (Yahya et al., 2008), nano-materials (Satapanajarua et al., 2011), coagulants and polymers (Duk et al., 2007) and biological oxidation (Sibel et al., 2009). Among these methods, electrocoagulation (EC) is one of thriving methods used for treatment of most effluents. EC process propose some merits such as less or no use of chemicals, less sludge, low-cost and compact equipment (Drouichea et al., 2007; Mouedhena et al., 2009).

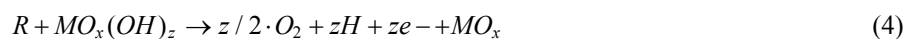
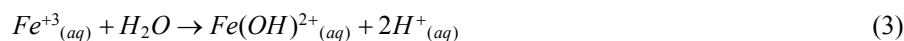
1.1 Mechanism of EC process

EC removes fine colloidal by releasing metal ions through their coagulation ability (Benhadji et al., 2011). EC process reduces pollutants principally by coagulation, adsorption, precipitation and flotation (Carmona et al., 2006; Mohammad and Sivakumar, 2009a). EC produce destabilising agents that neutralise electric charge of pollutants present in solution (Barrera-Díaza et al., 2012). The reactions during electrochemical process in reactor involving iron electrodes are as shown in equations (1) to (6).

- Anode



Due to effect of pH of solution, Fe^{3+} may undergo hydration, forming species like $Fe(OH)^{2+}$, $Fe(OH)^+_2$, and $Fe(OH)^3$, in acidic solution (Muftah et al., 2009), reaction were as shown in equation (3). The hydroxyl radicals are adsorbed at the electrode surface and degradation of organic matter occurs (Yusuf et al., 2010) as shown in equation (4).



where R is pollutant.

Apart from equations (1) to (3), possible anodic reaction for water oxidation (Chafi et al. 2011) takes place as shown in equation (5).



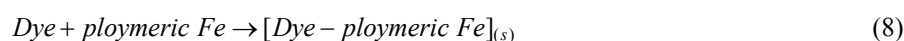
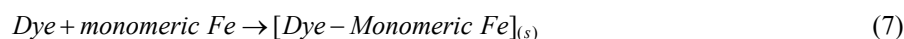
- Cathode (Behbahani et al., 2011)



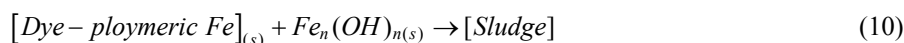
1.2 Mechanism for dye removal

The dye degradation depends on interactive mechanisms between dye molecules, hydrolysis (Gurses et al., 2002) and pH of solution (Kobyta et al., 2003). The insoluble metal hydroxide formed, remains in suspension, helps eliminate pollutants from wastewater under mechanism of precipitation and adsorption (Barunkumar and Sunil, 2017) as shown in equations (7) to (10). Furthermore, at low range of pH, precipitation is major and at $pH > 6.5$ it is explained as adsorption (Kobyta et al., 2003).

- Precipitation



- Adsorption



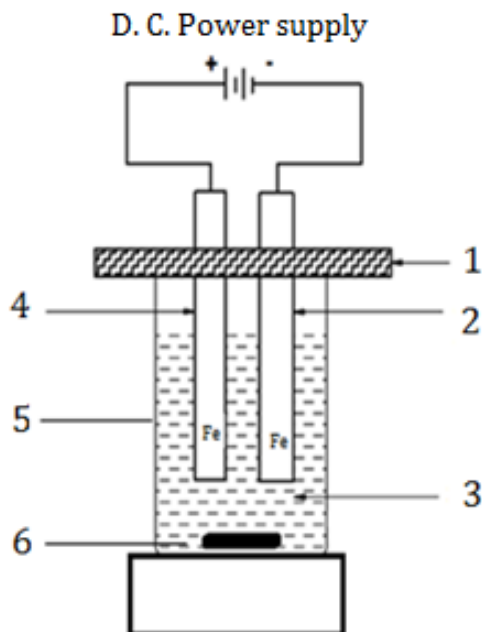
Studies have shown that EC is effectively used to treat contaminants present in water and effluents (Heidmann and Calmano, 2008). Industrial effluents treated using EC process includes, textile wastewater (Magdalena et al., 2009), paint manufacturing wastewater (Abdurrahman, 2012), dye decolourisation (Daneshvar et al., 2007), fluoride (Mohammad and Sivakumar, 2009b), metal plating wastewater (Feryal and Selva, 2011), landfill leachate (Bouhezila et al., 2011), Tannery (Benhadji et al., 2011) and strontium (Murthy and Parmar, 2011). The aim of experimentation was to study feasibility of colour removal of synthetic RR-195 solution using EC process. Parameter range was widen to understand importance of individual operational parameters. Furthermore, investigation may help to focus on parameters applying coagulation, adsorption or fenton like reaction reducing research experiments in decolourisation.

The process will be examined for effects of change in operational parameters such as change in electrical potential, electrolyte concentration, inter-electrode distance, initial dye concentration, H₂O₂ dose concentration, cathode material and electrolysis time.

2 Materials and methods

2.1 Experimental

The experiments were performed by mixing reactive red-195 concentrations of 60 mg/l with tap water for 30 min before experimenting. A reactor volume of 1 litre containing dye solution synthetic was feed with, 1 g /L of NaCl electrolyte to adjust strength of ionic solution. A simple EC cell contains iron electrodes having inter-electrode distance (S) of 1 cm and electrode dimension of 150 mm × 35 mm × 2 mm. The total submerged surface area (A_{sub}) of electrode into reactor was 29.75 cm². Stirring speed of 200 rpm was maintained inside reactor using SPECTRLAB WHIRLMATIC-2 controlled by ELECTRONIC MODULE EM 2. Current was applied through D.C. power supply model FALCON PS 303 D, characterised by range 0–3 amperes and 0–30 volts. The schematic of experimental setup is as shown in Figure 1. Electrodes used during experiments were sanded and washed with dilute HCl before each experiment. The pH and conductivity was detected using SEPTRALAB MULTIPARA MP- 5 to determine characteristics of synthetic solution. The progress of treatment was monitored by collecting supernatant of samples of 10 ml in triplicates after every 2 min. Collected supernatant of samples were then filtered to eliminate sludge formed during process and were examined for absorbance. The dye absorbance was determined using spectrophotometry (Labtronics LT-290) at λ_{max} 542 nm, according to Beer-Lambert law, using UV/vis spectrophotometer. The Table 1 represents structure of dye and characteristics of synthetic solution specified according to (APHA, 1998). Chemicals used for experiments such as NaCl, HCl, NaOH and H₂O₂ (30%) were purchased from MERK and reactive red-195 dye was collected from local dyeing industry.

Figure 1 Experimental set up for colour removal

- Notes: 1 – wooden plate.
 2 – cathode electrode.
 3 – synthetic RR-195 wastewater.
 4 – anode electrode
 5 – reactor (Glass jar 1 L capacity).
 6 – magnetic stirrer.

Table 1 Characterisation of synthetic dye wastewater

Characteristics	Units	Values
pH	-	7.69
Conductivity	mS/cm	2.73
Absorbance	λ_{\max}	542 nm
Structural formula ($C_{31}H_{19}ClN_7Na_5O_{19}S_6$)		

2.2 Calculation

Removal efficiency (η %) was calculated depending in absorbance of colour with respect to electrolysis time of treatment process given in equation (11).

$$\eta\% = \left[\frac{C - Cx}{C} \right] \times 100 \quad (11)$$

where C – initial absorbance, Cx – final absorbance at time of test. The electrical energy consumption (E), an important parameter in process is calculated using equation (12). Where E is the energy consumption (kWh/m³), U is the applied voltage (V), I is the current intensity (A), t is the EC time (hr), and V is the volume of the treated wastewater (L).

$$E = \frac{U * I * t}{V} \quad (12)$$

The theoretical amount of electrode dissolved (C) was calculated by using Faraday's law as shown in equation (13). Where C (g/L) is metal dissolved in the electrolytic cell, I is the current intensity (Amps), t is the EC time (hour), M is the molecular weight of the anode (g/mol), n is the chemical equivalence, F is the Faraday constant (96,500 C/mol) and V is the volume of the treated wastewater (litre).

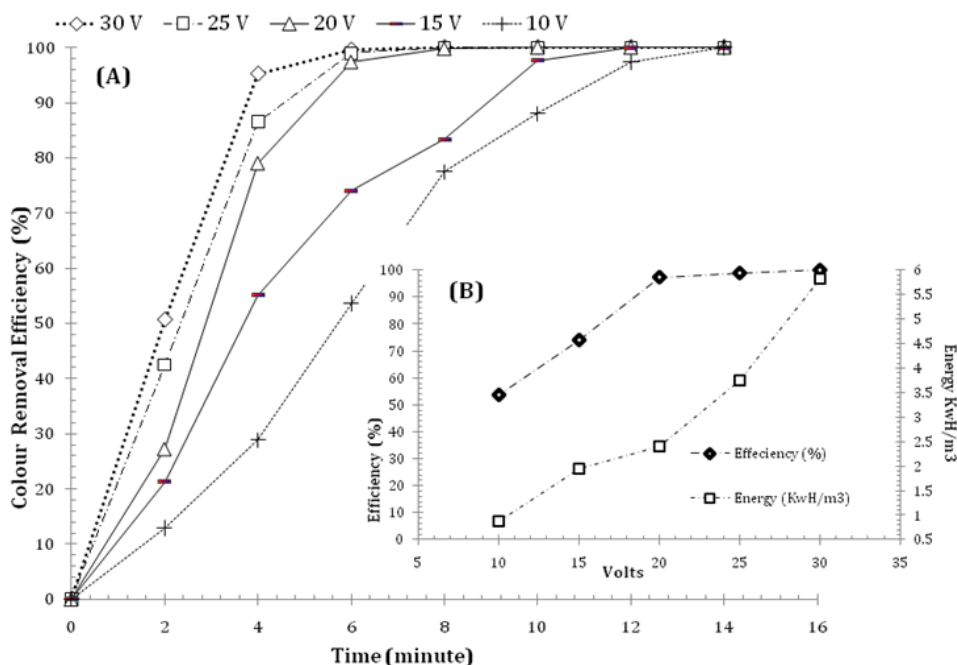
$$C = \frac{I * T * M}{n * F * V} \quad (13)$$

3 Result and discussion

3.1 Effect of change in electrical potential

The current density is defined as current applied per unit surface area of the electrode (Murthy and Parmar, 2011). Current density is one of important parameter which controls reaction rate and coagulant dose during EC process (Mohammad and Sivakumar, 2009a). The synthetic solution of RR-195 having concentration of 60 mg/L was investigated by applying electrical potential in ranges 10 to 30 volts in batch reactor. The electrolysis time increased from 6 to 14 min, with reduction in voltage from 30 to 10 volts. Concurrently, energy consumption reduced from 5.82 to 2.03 kWh/m³ respectively, to achieve 100% colour removal efficiency as shown in Figure 2(a). This phenomenon occurred when metal ion released from anode, depending on applied voltage exposed to electrolysis time according to Faraday's law (Abdurrahman, 2012). Similar results were observed by Modirshahla et al. (2007) with increased current density, colour removal increased. In addition, increase in turbidity was observed with voltage. This was because, rate of anode dissolution increased and generation of hydroxide ions approaching flocculation (PéguyNanseu-Njikia et al., 2009). The study shows result obtained for optimised voltage in orientation to colour removal efficiency and energy. The colour removal efficiency at 6th min, achieved 100% for 30 volts to 53.66% for 10 volts, consuming energy of 5.82 to 0.87 kWh/m³, respectively [Figure 2(b)]. According to Arash et al. (2011), 100% colour removal at 20 volts in 75 min. Consequently, from Figure 2(a) and 2(b), 20 volt was considered optimal for subsequent of experiments. The reason to optimise 20 volts was, with increased voltage from 20–30 volts, small removal efficiency difference was observed. Furthermore, increase in voltage results in increased current density with dissolving rate of anode (Feryal and Selva, 2011), results in surplus energy and electrode consumption.

Figure 2 (a) Effect of change in electrical potential on removal efficiency (b) Optimising volts V/s efficiency and energy during 6 min of electrolysis time



Note: Voltage range of 10–30 volts, RR-195 dye concentration 60 mg/l, NaCl 1g/lit, S = 1 cm, stirring speed of 200 rpm and iron electrode at anode and cathode $A_{sub} = 29.75 \text{ cm}^2$.

3.2 Effect of electrolyte concentration

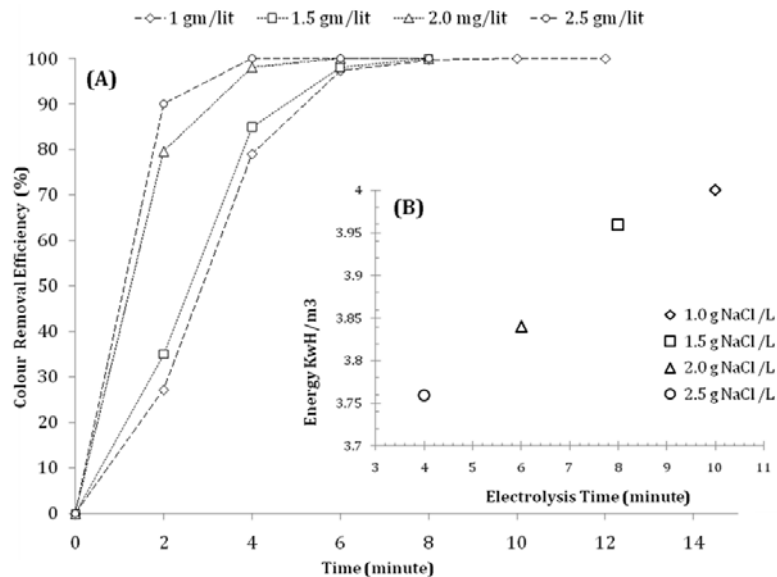
The concentration of electrolyte increases conductivity. Conductivity results in passage of excess electrons per unit time improving removal efficiency (Murthy and Parmar, 2011), with increase in current density at constant voltage (Koby et al., 2007). Many of electrolytes are available in market such as NaCl, KCl, KI, NH_4Cl , Na_2SO_4 , NaNO_3 , K_2SO_4 , BaCl_2 , NaBr , Na_2SO_3 , Na_2CO_3 and KNO_3 (Kashefialasl et al., 2006; Izquierdo et al., 2010). In addition, chloride can reduce the effect of anions (HCO_3^- , SO_4^{2-}) present in synthetic solution (Basiriparsa et al., 2011). In present study effect of NaCl concentration in range 1g/L to 2.5g/L (conductivity of 2.73 to 10.58 mS/cm) on colour removal efficiency was investigated. Removal efficiency increased with increased concentration of electrolyte, resulting in reduction of electrolysis time. The colour removal efficiency of 100% was achieved in 10 to 4 min with increase in conductivity from 2.73 to 10.58 mS/cm, respectively, consuming energy in range 4.0 to 3.76 kWh/m³, respectively as shown in Figure 3(a) and 3(b). In addition, probably with increase in NaCl concentration, increases Cl⁻ anions concentration destroying passive layer, catalysing the dissolution of electrode through localised corrosion by rise in chloride concentration (Chou and Huang et al., 2009). This improves colour removal efficiency by increase in metal hydroxide in wastewater, through dissolution of electrodes with reduction in passivation. Furthermore, the electrolysis time decreased reducing energy consumption

with rise in conductivity, as observed during experiments by Daneshvar et al. (2007). The experiment performed gave 90.08% removal efficiency for 2.5 gm/L of NaCl to 27.26 % for 1 mg/L of NaCl, having energy consumption of 3.76 to 4.0 kWh/m³ respectively at 20 volts. From results it can be considered as increase in NaCl give efficient process in removal and economic.

3.3 Effect of inter-electrode distance

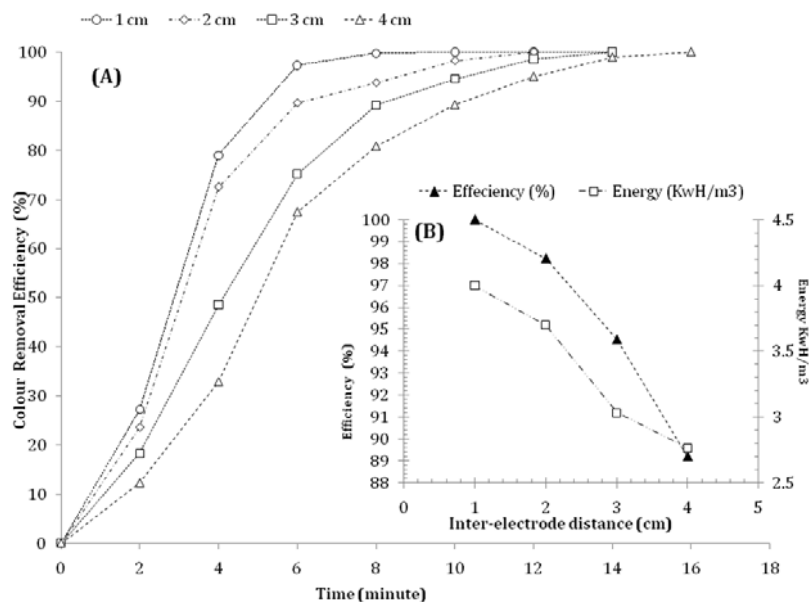
When distance between electrodes increases, increasing IR-drop and resistance, also reducing contact between ions (Song et al., 2008; Modirshahla et al., 2007). In present study effect of inter-electrode distance in range 1 cm to 4 cm on colour removal efficiency was investigated. The inter-electrode distance increased from 1–4 cm, electrolysis time increased from 10–16 min respectively, to achieve 100% removal efficiency (Arslan-Alaton, 2008), at constant voltage of 20 volts as shown in Figure 4(a). Energy consumption was investigated considering colour removal efficiency for 10 minutes of electrolysis time keeping 20 volts as constant. The removal efficiency decreased from 100%, 98.23%, 94.53% and 89.23%, reducing energy consumption from 4.0, 3.7, 3.03, and 2.76 kWh/m³ in 10 minutes, respectively as shown in Figure 4(b). This may be due to decrease in electrostatic effect, resulting in slower movement of ions, reducing formation of flocs required for coagulating contaminants (Khandegar and Saroha, 2013). Furthermore, the increased inter-electrode distance results in change of current density (Bouhezila et al., 2011), for that voltage must be increased to achieve required current density (Modirshahla et al., 2007; Daneshvar et al., 2007). Experiment demonstrated that at same electrolysis time removal efficiency decreases with increase in inter-electrode distance, from experiment 1 cm is appropriate for such kind of treatment.

Figure 3 (a) Effect of NaCl concentration on removal efficiency (b) Energy required V/s colour removal efficiency of 100% at varying EC time



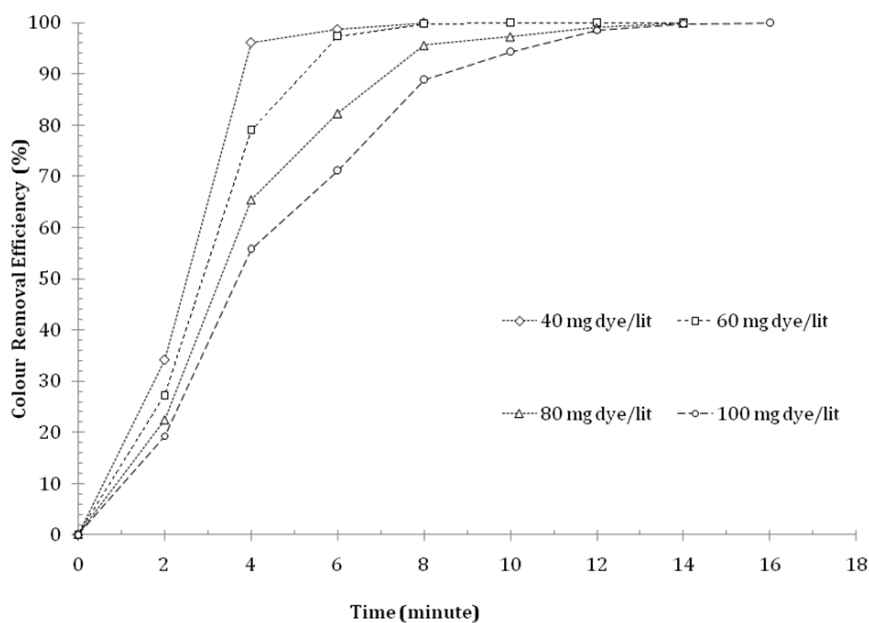
Note: RR-195 of 60 mg/l, Fe-Fe electrodes, S = 1 cm, stirring speed of 200 rpm, 20 volts, Fe-Fe combination and A_{sub} = 29.75 cm²

Figure 4 (a) Effect of inter-electrode distance (S) on removal efficiency (b) Efficiency achieved and energy required V/s inter-electrode distance (S) in 10 min of electrolysis time



Note: RR-195 of 60mg/lit, Fe-Fe electrodes, 1g NaCl/l, stirring speed of 200 rpm, A_{sub} = 29.75 cm² and 20 volts.

Figure 5 The effect of dye concentration on removal efficiency



Note: Fe-Fe electrodes, S = 1 cm, NaCl 1 gm/l, stirring speed of 200 rpm, Fe-Fe combination, A_{sub} = 29.75 cm² and 20 volts

3.4 Effect of initial dye concentration

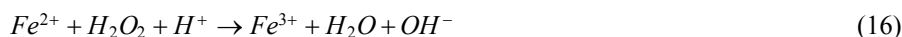
According to studies, as initial concentration of dye increases removal efficiency decreases (Daneshvar et al., 2007; Adhoum et al., 2004) increasing electrolysis time (Mohammad and Sivakumar, 2009a). In present study effect of initial dye concentration in range 40 to 100 mg/L, on colour removal efficiency was investigated. Similarly, 100% colour removal was achieved (Raghu and Basha, 2008) in range of 8 to 16 minutes from dye concentration from 40 to 100 mg/L, respectively as shown in Figure 5. These may be due to constant rate of metal hydroxide generated at invariable voltage of 20 volts (Chou and Huang, 2009). In EC process, colour is adsorbed onto metallic hydroxide flocs and removed by settling (Daneshvar et al., 2007) according to equations (6) to (9). Consequently, less quantity of contaminants adsorbed with increase in dye concentration resulting in reduction efficiency of process.

3.5 Effect of H_2O_2 dose

Currently, electrochemical treatment has developed enormous interest (Pulgarin and Kiwi, 1996), on one hand and advance oxidation process proved potential, on other EC studied extensively for decolourisation of wastewater (Neamtu et al., 2004). In anodic oxidation, pollutants are mineralised by direct electron transfer reactions or action of radical species (hydroxyl radicals) formed on the electrode surface (Babuponnusami and Muthukumar, 2013) as shown in equation (14).



During experimentation H_2O_2 was added externally to the reactor. In addition, ferrous ions were generated from sacrificial anode (Kurt et al., 2007) as shown in equation (15).



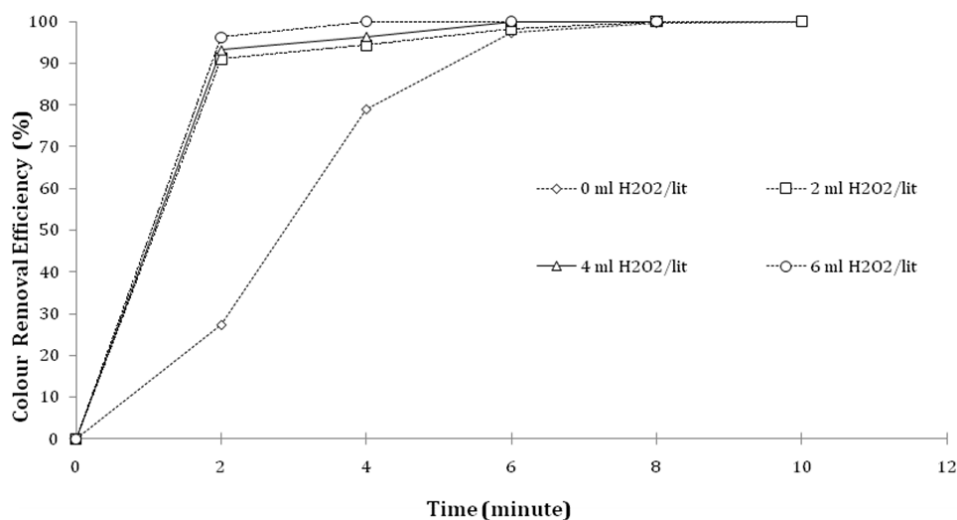
where RH denoting pollutants.



In experiment, effect of H_2O_2 dose in range 0 to 6 ml/l, on colour removal efficiency was investigated. The removal efficiency increased from 27.26% to 96.42% in 2 minutes with addition of H_2O_2 dose from 0 to 6 ml/l in EC cell, respectively as in Figure 6 according to equation (16). Similar results were observed by Nidheesh and Gandhimathi (2012), with increased H_2O_2 dose, colour removal efficiency increased. The reason may be, a very reactive, non selective oxidising hydroxyl radical OH agent in solution is generated which helps colour removal (Nidheesh and Gandhimathi, 2012; Wang et al., 2005) as shown in equation equations (16) to (19). Finally, removal efficiency of 100% was achieved (Mohamed et al., 2011), with reduction in time from 10, 8, 6 and 4 minutes, with increased H_2O_2 dose in range 0 to 6 ml/L, respectively (Figure 6). The removal

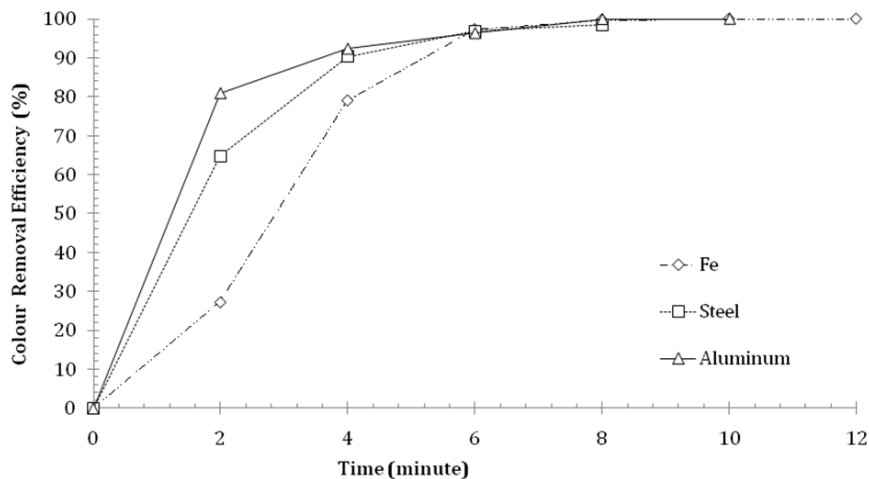
efficiency after 2 ml of H_2O_2 slightly increased, this was due to hydroxyl radical scavenging effect of H_2O_2 (Nidheesh and Gandhimathi, 2012).

Figure 6 The effect of H_2O_2 dose on removal efficiency



Note: RR-195 of 60 mg/lit, Fe-Fe electrodes, $S = 1$ cm, NaCl 1 gm/l, stirring speed of 200 rpm, $A_{sub} = 29.75$ cm² and 20 volts.

Figure 7 The effect of cathode electrode on removal efficiency



Note: RR-195 of 60 mg/lit, Fe-Fe electrodes, $S = 1$ cm, stirring speed 200 rpm, NaCl 1g/l, $A_{sub} = 29.75$ cm² and 20 volts)

3.6 Effect of cathode material

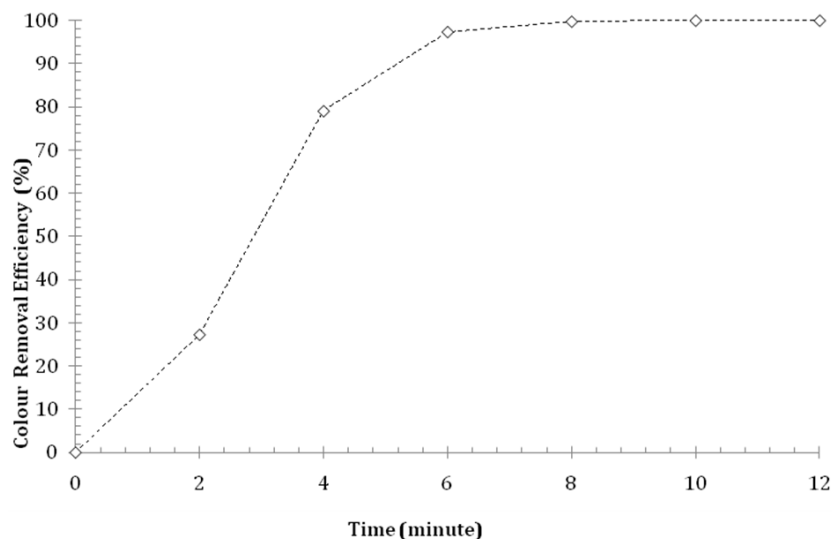
Different metal electrodes are used in EC process, which also majorly contribute to performance (Lai and Lin, 2003). Cathode electrode generates H_2 gas when cell voltage

is applied to process (Benhadji et al., 2011). Increase in voltage increases generation of bubbles at cathode, also size of bubbles depends on cathode material (Chen et al., 2004). In present study effect of cathode materials such as iron, aluminum and steel, on colour removal efficiency was investigated as shown in Figure 7. Removal efficiency of 27.26%, 64.74% and 80.90% was achieved using Fe, Steel and aluminum, respectively in 2 minutes. Colour removal of 100% was achieved in 10 min using Fe-Fe, Fe-Al combination and Fe-steel required 12 minutes. Initial removal efficiency increased in Fe-Al combination, because of intense emission of H₂ gas bubbles evolved at aluminum cathode. After analysis of cathode material in experimentation, it was observed that Fe and Al can be used for such experimentation, with steel, time and efficiency required are more.

3.7 Effect of electrolysis time

Electrolysis time provided to process generates metal hydroxide forming floc, which coagulates with impurities (Murthy and Parmar, 2011) and increasing decolourisation rate (Secula et al., 2011). In present study, effect of electrolysis time was studied on colour removal efficiency applying optimised voltage of 20 volt considering other operational parameters as constant. Increase in time, concentration of hydroxide metal ions increases, resulting in colour removal efficiency from 27.26% to 99.77% in 2 to 8 minutes, respectively. Furthermore, 100% of colour removal was achieved in 10 min as shown in Figure 8.

Figure 8 The effect of electrolysis time on removal efficiency



Note: 20 volts, dye concentration of 60 mg/l, NaCl 1g/lit, S = 1 cm, stirring speed of 200 rpm, A_{sub} = 29.75 cm² and iron electrode at anode and cathode.

4 Conclusions

Results observed showed that EC process could be used for removal of RR-195 from synthetic dye wastewater of concentration of 60 mg/L. The optimal operational parameters observed were 20 volts of electrical potential for 10 min of EC time considering removal efficiency and energy consumption. A 20 volt was chosen as constant for all operational parameter during successive experiments. Therefore, NaCl optimal of 2.0 g /L, could be suitable, as reduces electrolysis time to 4 min to achieve 98.08% removal efficiency, further increase in NaCl concentration has negligible effect on colour removal efficiency. Inter-electrode distance (S) of 1 cm is optimal as it takes 10 min for 100% removal, compared to 2, 3, and 4 cm. In addition, dose of H₂O₂ gave effective results, a 2 ml/L H₂O₂ resulted 91.06% in 2 min with small increase of 96.24% by adding 6 ml/L at same time. Therefore H₂O₂ of 2ml/L can be optimised for 60 mg/L of dye concentration. Cathode material suitably paired with Fe at anode is either Al or Fe instead of steel. Thus above 6 investigation indicates that using Fe-Fe or Fe-Al combination with optimised parameters at constant dye concentration can record effective results for such experiments.

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