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Assessment of electrocoagulation for the treatment of petroleum refinery wastewater

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ABSTRACT

Batch electrocoagulation experiments were carried out to evaluate the removal of sulfate and COD from petroleum refinery wastewater using three types of electrodes: aluminum, stainless steel, and iron. The effects of current density, electrode arrangement, electrolysis time, initial pH, and temperature were investigated for two wastewater samples with different concentrations of COD and sulfate. The experimental results indicated that the utilization of aluminum, as anode and cathode, was by far the most efficient arrangement in the reduction of both the contaminants. The treatment process was found to be largely affected by the current density and the initial composition of the wastewater. Although electrocoagulation was found to be most effective at 25°C and a pH of 8, the influence of these two parameters on the removal rate was not significant. The results demonstrated the technical feasibility of electrocoagulation as a possible and reliable technique for the pretreatment of heavily contaminated petroleum refinery wastewater.

1. Introduction

Wastewater generated by chemical plants including petroleum refineries is often characterized by high concentrations of aliphatic and aromatic petroleum hydrocarbons, which usually have detrimental and harmful effects on plant and aquatic life as well as surface and ground water sources. Wastewater facilities in these plants usually rely on many expensive pretreatment steps to reduce the concentration of these organic contaminants before any final biological purification step. These pretreatment units may include ultrafiltration, adsorption, coagulant and coagulant aides, and electrochemical process (Demirci et al., 1998).

Chemical coagulation refers to the addition of chemicals such as Alum [$Al_2(SO_4)_3 \cdot 18H_2O$] to an aqueous solution to combine small dispersed particles into larger agglomerates which can be removed by some other method such as sedimentation, air floatation, or filtration. Coagulation can also be accomplished by the in situ generation of coagulants by electrolytic oxidation of an appropriate anode material, which is referred to as electrocoagulation (Izares et al., 2006). In recent years, there has been increased interest in the application of electrocoagulation in the treatment and purification of industrial wastewater (Khoufi et al., 2007; Pouet et al., 1992). Electrocoagulation is efficient in removing suspended solids as well as oil and greases (Xinhua and Xiangfeng, 2004). It removes metals, colloidal solids and particles, and soluble inorganic pollutants from aqueous media by introducing highly charged polymeric metal hydroxide species. These species neutralize the electrostatic charges on suspended solids and oil droplets to facilitate agglomeration or coagulation (Yilmaz et al., 2007). The treatment prompts the precipitation of certain metals and salts. The reactions occurring in an electrochemical cell involving aluminum electrodes are as follows (Yilmaz et al., 2007):

At the cathode:

$$3H_2O + 3e^- \longrightarrow \frac{3}{2}H_{2(g)} + 3OH^-_{(aq)} \tag{1}$$

At the anode:

$$Al_{(s)} \longrightarrow Al^{+3}_{(aq)} + 3e^- \tag{2}$$

In the solution:

$$Al^{+3}_{(aq)} + 3H_2O \longrightarrow Al(OH)_3 + 3H^+_{(aq)} \tag{3}$$

For iron, the cathode reaction is the same as reaction (1), whereas the anode reaction is as follows (Mollah et al., 2001; Xinhua and Xiangfeng, 2004):

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At the anode:

$$Fe_{(s)} \longrightarrow Fe^{+2}_{(aq)} + 2e^{-} \tag{4}$$

$$Fe^{+2}_{(aq)} + 2OH^{-}_{(aq)} \longrightarrow Fe(OH)_{2(s)} \tag{5}$$

The Fe^{3+} ions may undergo hydration. Depending on the pH of the solution, $Fe(OH)^{2+}$, $Fe(OH)^{+}_{2}$, and Fe(OH)3 species may be present under acidic conditions. The solution reaction is:

In the solution:

$$Fe^{+3}_{(aq)} + H_2O \longrightarrow Fe(OH)^{2+}_{(aq)} + 2H^{+}_{(aq)} \tag{6}$$

Some of the advantages of the electrocoagulation are the simple equipment required and the easy automation of the process (Izares et al., 2006). Also, since it does not require any addition of chemicals, the dosing of coagulant reagents depends on the cell potential (or current density) applied. Other advantages include the promotion in the flocculation process, caused by the turbulence generated by the oxygen and the hydrogen evolution that produces a soft mix, and helps the destabilized particles generate bigger particles. In addition, the formed oxygen and hydrogen bubbles increase the efficiency of the separation process through electro-floatation (Cansares et al., 2008).

Because of these advantages, electrocoagulation has been used to treat different types of wastewater such as cutting oil–water emulsions (Bensadok et al., 2008), oil suspension used for machining and drilling operations (Cansares et al., 2008; Khemis et al., 2005), olive mill wastewater (Tezcan et al., 2006, 2009; Inan et al., 2004), oily bilge water (Asselin et al., 2008), solutions containing 4-nitrophenol (4-NP) (Modirshahla et al., 2008), and wastewater from different industries, such as textile (Kim et al., 2002), dairy (Sengil and Ozacar, 2006), yeast production (Kobya and Delipinar, 2008), and mechanical polishing (Drouiche et al., 2007). It has been utilized in the reduction of different contaminants such as phosphate (Irdemes et al., 2006), sulfur compounds (Murugananthan et al., 2004), phenolic compounds (Uğurlu et al., 2008), fluoride (Emamjomeh and Sivakumar, 2009a), and heavy metals (Escobara et al., 2006).

In spite of the considerable success of electrocoagulation for the treatment of various types of wastewater, its application as a possible technique for the treatment of petroleum refinery wastewater is rather scarce in the literature. The main objective of the present study, therefore, is to evaluate the reduction of sulfate and COD in petroleum refinery wastewater using three types of electrode materials at different operating conditions. Two refinery wastewater samples with different COD and sulfate concentrations were used in the study.

2. Experimental methods

2.1. Characteristics of wastewater

Real refinery wastewater samples were obtained from a local petroleum refinery and used for the study. The samples were collected from two different locations upstream of the refinery's wastewater treatment system. Analyses of the two wastewater samples, namely WW-A and WW-B, are given in Table 1. A HACH UV Spectrophotometer, Model DR-5000 HACH, Germany, was used for the photometric total COD and sulfate analyses. The concentrations of COD and sulfate were measured according to US EPA Method (410.4) and Method (375.4), respectively.

2.2. Electrocoagulation reactor

Table 1
Characteristics of refinery wastewater samples.

Parameter	WW-A	WW-B
pH	6.6	9.5
Sulfate (mg/L)	887	1222
COD (mg/L)	596	4050
Color	Slightly turbid	Dark green
EC (ms/cm)	9.76	16.36
TSS (g/L)	0.12	0.08
TDS (g/L)	5.87	10.11

Experiments were carried out in a batch electrochemical cylindrical glass cell (ID = 72 mm; $H = 61.4$ mm) with a total volume of 250 ml; a magnetic stirrer was used to provide sufficient mixing inside the reactor. In each experimental run, a total amount of 200 ml of real refinery wastewater was treated in the electrochemical cell; the metal electrodes were dipped into the wastewater sample and connected to an ammeter to provide the needed current, using a DC power source (Tektronix, TM 5006 A). The metal electrodes were made of rectangular metal sheets with dimensions of 40 mm × 60 mm × 1 mm, with a total area of 48 cm^2. A schematic diagram of the experimental set-up is shown in Fig. 1.

At regular intervals of 15 min, 5 ml samples were withdrawn, filtered using Whatman filter paper (Grade 40), and sent for analysis of sulfate and COD contents. The effects of current density, initial solution pH, temperature, electrolysis time, and the distance between the electrodes were investigated.

3. Results and discussion

3.1. Effect of electrode material and current density

The effect of current density on the removal of sulfate from WW-A was investigated at 25 °C using three different electrode materials, namely aluminum (Al), stainless steel (SS), and iron (Fe). Each electrode, with 48 cm^2 area, was tested separately for different current densities in the range of 2–13 mA/cm^2, for a total contact time of 60 min. The percent reductions of sulfate for the three different electrodes are shown in Fig. 2. The results show that the aluminum electrode is by far the most effective, with a reduction in sulfate more than 2.5 times than that of the other two electrodes. This may be attributed to the precipitation of sulfate

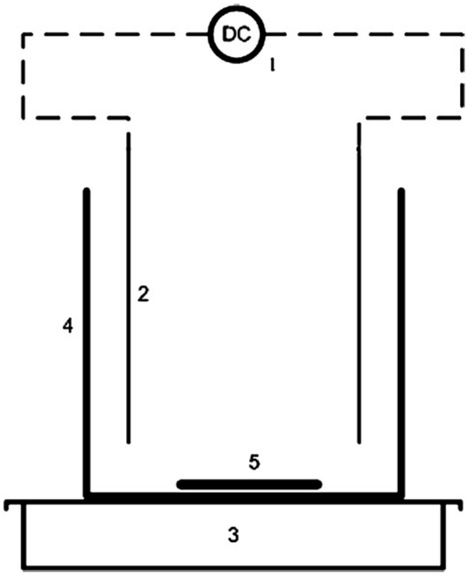

Fig. 1. Schematic diagram of the electrochemical apparatus: (1) DC power supply, (2) metal electrodes, (3) magnetic stirrer, (4) glass beaker, and (5) magnetic bar stirrer.

salts at the bottom of the electrocoagulation cell, which is formed by the reaction of aluminum hydroxide with the sulfate ions in the solution. The sulfate ions may also be enmeshed in the porous metal oxide/hydroxide precipitate (Murugananthan et al., 2004). The results in Fig. 2 show that the reduction in sulfate concentration is higher for aluminum electrode, which is mainly due to the faster formation of aluminum sulfate and its lower solubility in comparison to ferrous sulfate. At 30 °C the solubility of aluminum sulfate is 400 kg m^{-3}, whereas that of ferrous sulfate is 600 kg m^{-3} (Perry and Green, 1997). On the other hand, as stainless steel resists corrosion more than usual iron, the rate of formation of ferrous hydroxide from stainless steel is slower than that from iron.

The results of Fig. 2 also show that the reduction in sulfate increases with increasing the current density. This is partly because increasing current density results in generating more bubbles which cause greater upwards flux and faster removal of pollutants by floatation (Adhoum and Monser, 2004; Tir and Mostefa, 2008). The main effect of increasing current, however, is the higher rate of anode dissolution that increases the concentration of metal ions in the solution, which consequently enhances the pollutants removal rate (Tir and Mostefa, 2008). When iron and stainless steel electrodes were used, the effect of current on sulfate reduction was found to fade at current density values beyond 8 mA/cm^2. Increasing the current density still results in increasing the rate of ferrous ions formation, however, in this range the rate of sulfate reduction is determined by the rate of formation of ferrous sulfate, and the ferrous ions accumulate and increase the electrochemical potential of the solution. On the other hand, when aluminum electrode was used, the sulfate reduction continues to increase with increasing the current throughout the entire range of current investigated. This is because the rate of reaction of aluminum ions with sulfate is fast and the drop in sulfate content is mainly determined by the rate of aluminum ions generated. Therefore, providing more aluminum ions, by increasing the current, always results in an increase in the sulfate reduction. Another reason for this phenomenon can be attributed to the effect of temperature on the solubility of sulfate salts. As the current density increases, the temperature of the solution increases and consequently so does the solubility of aluminum and ferrous sulfates (Perry and Green, 1997). However, at higher temperatures, the solubility of aluminum sulfate increases only by 25% from 365 kg m^{-3} at 20 °C to only 455 kg m^{-3} at 40 °C. Hence, the sulfate is still effectively removed by precipitation. On the contrary, the solubility of ferrous sulfate increases dramatically by 53% with increasing the temperature from 480 kg m^{-3} at 20 °C to 733 kg m^{-3} at 40 °C. Therefore, at higher currents density, and hence higher temperatures, the efficiency of sulfate precipitation is reduced.

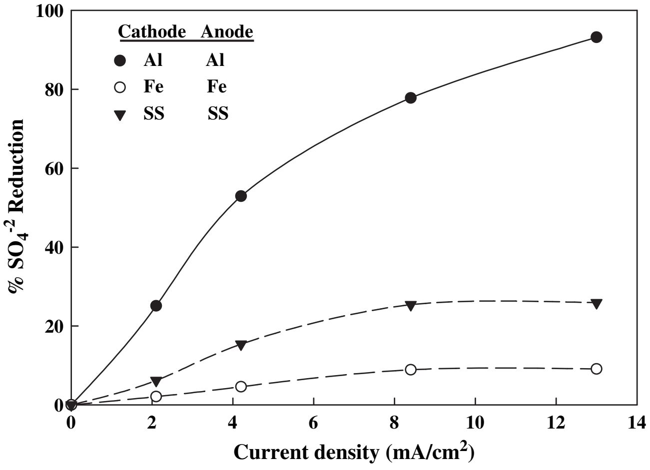

Fig. 2. Sulfate reduction as a function of current density for different electrodes for WW-A using electrode area of 48 cm^2, time of electrolysis of 60 min, temperature of 25 °C, and pH of 6.6.

The amount of electrode material dissolved or consumed during the electrocoagulation process depends heavily on the current density as described by the Faraday's law (Kim et al., 2002; Adhoum and Monser, 2004; Mollah et al., 2004):

$$W = \frac{ItMw}{ZF} \tag{7}$$

where W is the aluminum dissolved (g); I is the current (A); t is the contact time (s); M_w is the molecular weight (g/mol); Z is the number of electrons involved in the redox reaction (equals 3 for aluminum); and F is the Faraday's constant (96,500 C/mol of electrons). Fig. 3 shows the estimated amount of aluminum dissolved as a function of time for a current of 0.6 A (current density of 13 mA/cm^2). The figure shows that the rate of electrode erosion at this current density is about 3.3 mg/min.

A comparison between the percentage reduction in sulfate concentration and COD from WW-A was carried out at 25 °C using aluminum electrodes at 13 mA/cm^2. The results in Fig. 4 show that the sulfate concentration does not change significantly after 60 min of operation. This indicates that the optimum electrolysis time is 60 min and that any operation beyond this time will be unjustified. However, COD continues to drop slightly at times beyond 60 min. Nevertheless, all the subsequent experiments were carried out for 60 min, which was used as a bench mark. It is worth noting that the distance between the cathode and anode was varied between 1 and 4 cm, and the results showed no effect on the reduction of COD or sulfate.

3.2. *Effect of wastewater characteristics*

In order to evaluate the effect of refinery wastewater composition on the electrocoagulation performance, the above experiment was repeated using WW-B which has higher concentrations of sulfate and COD as shown in Table 1. Fig. 5 shows the percentage removal of sulfate from the two wastewater samples, namely WW-A and WW-B. It can be seen that the percentage of sulfate removal reached 93% for WW-A, whereas for WW-B it was in the range of 24%. Similar result was observed for COD, in which the percentage removal was 63% and 42% for WW-A and WW-B, respectively, as shown in Fig. 6.

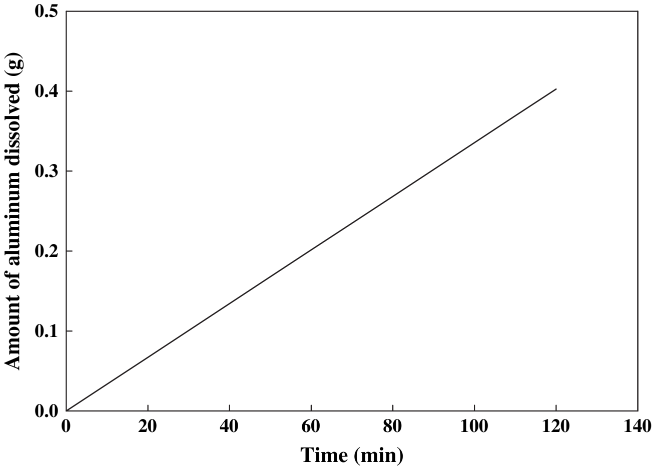

Fig. 3. Weight of aluminum electrode dissolved with time for current of 0.6 A.

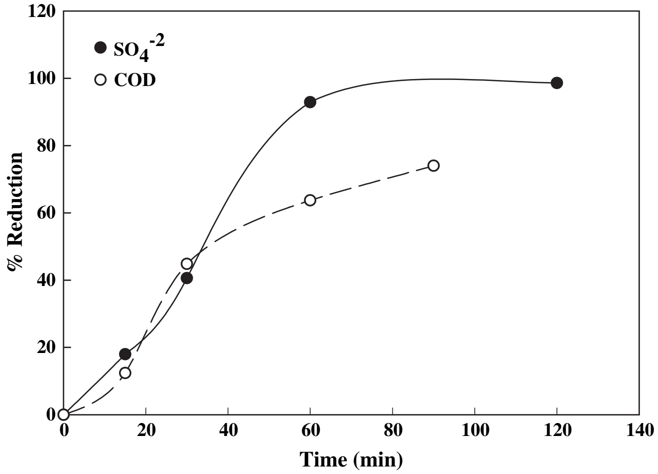

Fig. 4. Sulfate and COD reductions as a function of time for Al–Al electrodes for WW-A using electrode area of 48 cm^2, current density of 13 mA/cm^2, temperature of 25 °C, and pH of 6.6.

It is worth noting here that using percentage removal may give deceiving results, taking into consideration that the initial concentrations of sulfate and COD in WW-A are much lower than those in WW-B. Therefore, the lower percentages of sulfate and COD removal for WW-B are mainly due to their initial high concentrations. Calculation of the total amount of COD and sulfate removed in grams per gram of Al ion generated according to Eq. (7), from both wastewater samples, revealed that there were slightly more contaminants removed from WW-B.

3.3. Effect of electrode arrangement

The effect of cathode–anode arrangement on the reduction of sulfate and COD in WW-B was evaluated, at a constant operating temperature of 25 °C and current density of 13 mA/cm^2, to compare different electrode combinations and to select the best arrangement for the treatment of such wastewater. The percentage removal of sulfate and COD was strongly affected by the electrode arrangements as shown in Figs. 7 and 8, respectively. It is seen from Fig. 7 that the aluminum anode with different cathode materials was more capable of reducing the concentration of sulfate than iron and stainless steel anodes. As explained earlier, aluminum sulfate is formed faster and precipitates easier than ferrous sulfate. In addition, aluminum anode has proven to more effective anode for COD removal, as shown in Fig. 8.

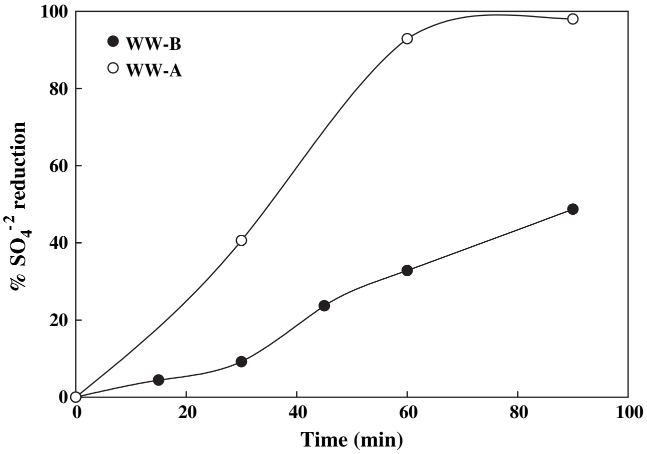

Fig. 5. Effect of wastewater characteristics on the removal of sulfate using a current density of 13 mA/cm^2 and temperature of 25 °C.

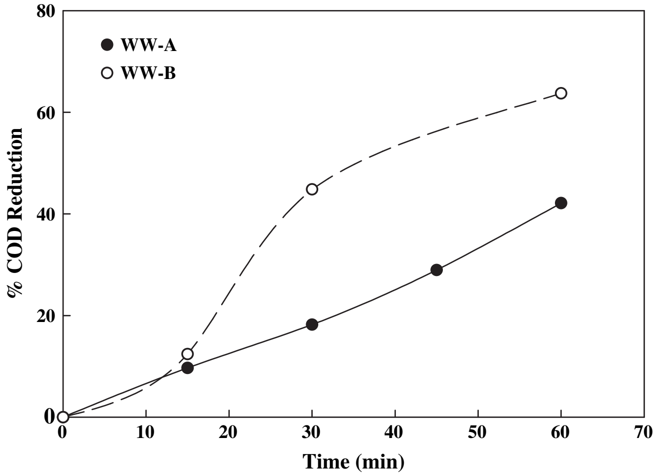

Fig. 6. Effect of wastewater characteristics on the removal of COD using a current density of 13 mA/cm^2 and temperature of 25 °C.

On the other hand, the type of cathode played an important role in sulfate removal with aluminum cathode showing the best performance, as shown in Fig. 7. For aluminum anode, the percentage removal of sulfate reached 33% and 16% when the cathode material was aluminum and stainless steel, respectively. However, it is noticed from Fig. 8 that when aluminum anode was used, the percent removal of COD was almost the same regardless of the material of cathode used. These results indicate that a certain electrode arrangement may prove to be more efficient or more economical for a specific application. Since Al–Al electrode arrangement was by far the most effective in reducing the sulfate and COD concentrations, all the subsequent investigations were carried out using aluminum electrodes.

3.4. Effect of temperature and pH

Temperature and pH are always considered important parameters in any chemical or electrochemical separation process. The

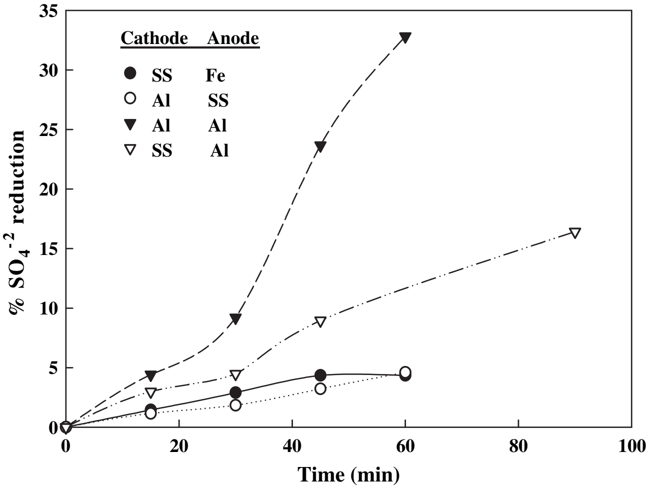

Fig. 7. Effect of electrode arrangements (cathode–anode) on the removal of sulfate for WW-B using a current density of 13 mA/cm^2, temperature of 25 °C, and pH of 9.5.

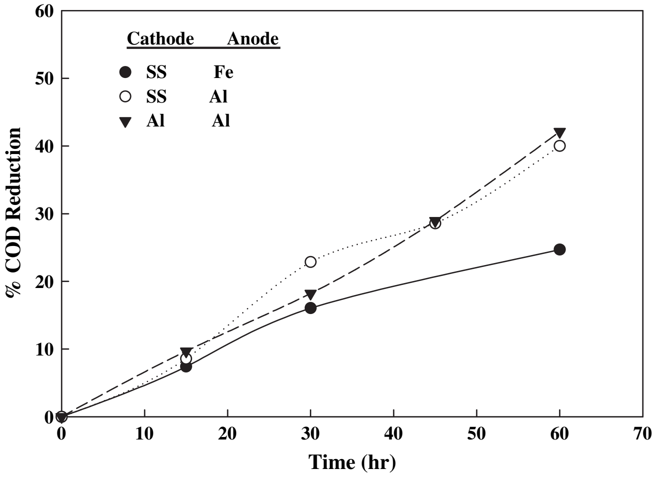

Fig. 8. Effect of electrode arrangement (cathode–anode) on the removal of COD for WW-B using a current density of 13 mA/cm^2, temperature of 25 °C, and pH of 9.5.

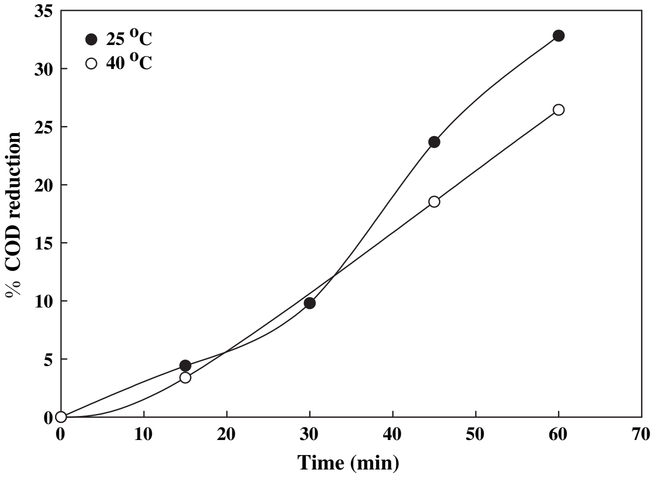

Fig. 10. Effect of temperature on the removal of COD for WW-B using a current density of 13 mA/cm^2 and pH of 9.5.

effect of temperature on the removal of sulfate and COD from WW-B was evaluated at two different temperatures, namely 25 and 40 °C and a constant current density of 13 mA/cm^2, where the temperature was controlled using a water bath (Schutzart DIN 4050-IP 20, Germany). The results are presented in Figs. 9 and 10 for the percentage removal of sulfate and COD, respectively. Fig. 9 shows that for the first 45 min of electrolysis, the temperature has negligible effect on the sulfate removal. However, after 45 min, the removal rate at 25 °C becomes slightly higher than that at 40 °C. Similar behavior was observed for COD shown in Fig. 10. As explained earlier, the increase in the temperature of the solution results in increasing the solubility of aluminum sulfates (Perry and Green, 1997). Therefore, the precipitation of the aluminum sulfate is enhanced at lower temperatures, which results in a better removal.

Solution pH is a significant factor in the solubility of aluminum sulfate, and hence in the sulfate removal. The effect of the initial pH on the sulfate removal from WW-A was evaluated by varying the initial pH from 6 to 9 using HCl or NaOH (0.1 M). The results presented in Fig. 11 show that the best pH for the reduction of sulfate concentration was found to be about 8. It is believed that the pH affects the reduction of sulfate and COD from the solution in two ways. The first is the solubility of aluminum sulfate, which increases as the pH decreases. As the acidity increases (i.e., pH decreases), the concentration of H_3O^+ in the solution increases, which reacts with the sulfate to form HSO_4^-. The solubility of aluminum hydroxide has been reported to be minimum at the pH range of 6–8 (Emamjomeh and Sivakumar, 2009b). The solid aluminum hydroxide, Al(OH)3, is also believed to contribute to the reduction of COD through coagulation and co-precipitation with the organic compounds in the wastewater. Aluminum hydroxide precipitates at pH 6–7, and its solubility increases as the solution becomes either more acidic or alkaline (Tezcan et al., 2009). Therefore, both very low and very high pH values do not favor the precipitation, and the most favorable condition would be somewhere in between, which was found to be 8 in this work.

4. Conclusions

Electrocoagulation was assessed as a possible technique for the reduction of sulfate and COD concentration in petroleum refinery wastewater. Wastewater samples, collected from two different locations in the wastewater treatment system of a local petroleum refinery, were treated in a batch electrochemical cell using three types of electrode materials: aluminum, stainless steel, and iron.

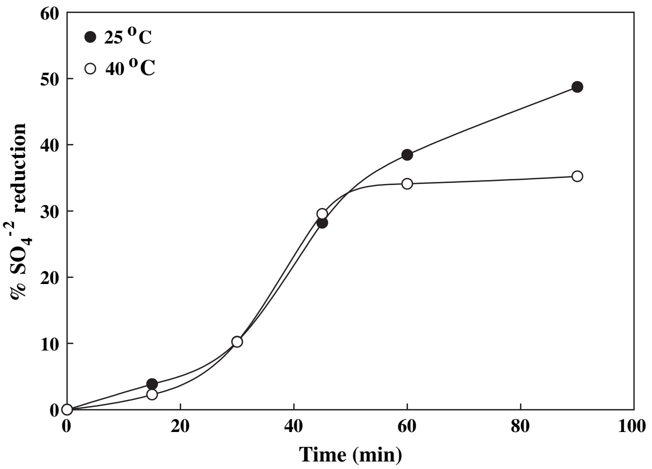

Fig. 9. Effect of temperature on the removal of sulfate for WW-B using a current density of 13 mA/cm^2 and pH of 9.5.

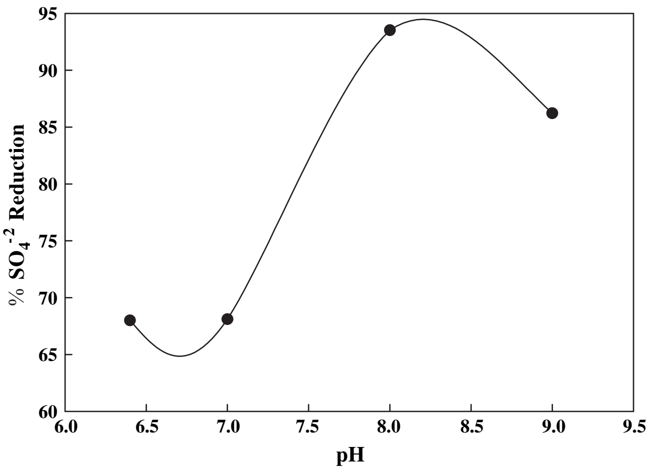

Fig. 11. Sulfate reduction as a function of pH for WW-A using a current density of 13 mA/cm^2 and temperature of 25 °C.

The experimental results showed that electrocoagulation can achieve percentage removal of up to 93% and 63% for sulfate and COD, respectively. It was shown that the utilization of aluminum, as anode and cathode, was by far the most effective arrangement in the reduction of both sulfate and COD. The performance of the electrochemical reactor was found to be highly influenced by the initial composition of the wastewater and the current density. The electrocoagulation process was most effective at 25°C and a pH value of 8. The study demonstrated that electrocoagulation may prove to be a practical approach for the pretreatment of petroleum refinery wastewater.

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