Removal of COD from Petroleum refinery Wastewater by Electro-Coagulation Process Using SS/Al electrodes

Sajjad S. Alkurdi and Ali H. Abbar
Al-Qadisiyah University, Chemical Engineering Department, Al-Diwaniyah, Iraq
E-mail: engsajad89@yahoo.com

Abstract. In the present study, the effectiveness of a procedure of electrocoagulation for removal of chemical oxygen demand (COD) from wastewater of petroleum refinery has been evaluated. Aluminum and stainless steel electrodes were used as sacrificial anode and cathode respectively. The effects of current density (4-20mAcm⁻²), pH (3-11), and NaCl concentration (0-4g/l) on the efficiency of COD removal were investigated. The results have shown that increasing of current density led to increase the efficiency of COD removal while increasing NaCl concentration resulted in decreasing of COD removal efficiency. Effect of pH was found to be lowering COD removal efficiency when the pH increased or decreased from pH=7. The best conditions were found to be a current density of 12mA/cm², pH=7 and NaCl concentration of 2g/l at a treatment time of 60 minutes, where a maximum COD removal efficiency of 96.8%, phenol removal efficiency of 64.7%, and total dissolved solid (TDS) removal efficiency of 20.6% were obtained at energy consumption of 29.12 kWh/kg COD. The results of the present work gave COD of 8mg/l which is lower than the standard limit for discharging the petroleum refinery effluent. The electrocougulation was proven to be efficient and reliable technique for treatment Al-Dewaniya petroleum refinery effluent to get an effluent with features in agreement with the standard limits for discharge the pollutants to the environment at lower cost.

Keywords: Electrocoagulation, COD removal, aluminum anode, Petroleum refinery wastewater.

1. Introduction

The treatment of wastewater, generated by different sectors of industries, represents a significant environmental pollution challenge because of its large amount and the variety of pollutants existing. One of these industries is the petroleum refinery, in which huge quantities of water are used especially for distillation, hydro-treating, thermal cracking and catalytic cracking, desalting and cooling systems [1]. Petroleum refining processes generate wastewater of about 1.6 times the volume of the processed crude oil. Furthermore, approximately 80-90 percent of the water supplied to the petroleum refinery comes out as wastewaters, therefore treatment of these wastewaters is not only to avoid environmental pollution issues, but also to reuse of the water again hence another
economic benefit for oil refineries [2]. The characteristics of petroleum refinery effluents are usually influenced by production mode, plant configuration and types of oil [3]. However, wastewaters from refinery processes are generally defined by having high aliphatic and aromatic hydrocarbon concentrations that typically have harmful and detrimental effects on the plants and the aquatic life, as well as the surface and sources of ground water [4]. Therefore, effective and feasible methods for treating of petroleum refinery wastewater are needed.

Different technological methods have been used in this field including biodegradation, ultrafiltration, adsorption, coagulation, and electrochemical processes [4]. Electrochemical techniques such as, electro-flotation (EF), electro-decantation (ED), electrocoagulation (EC), and electrooxidation (EO) have the benefit of being easily distributed, and require minimum amount and number of chemicals. In recent years, several studies have focused on electrocoagulation, which is an effective process used to treat wastewaters with finely dispersed particles [5]. This technique is robust and compact, and hence has the potential to replace sophisticated processes that require large volumes and/or number of chemicals [6]. This technique depends on the electrochemical dissolution of a sacrificial anode for generating various polymeric and monomeric composites of metal ions [7]. The produced species of metal in the solution have a destabilizing effect on the colloidal contaminants in the waste-water, form aggregated particles and flocs and adsorb the dissolved composites [8]. For instance, aluminum ions, produced from dissolution of aluminum anode, react with hydroxyl ions generated by reduction of water at cathode to make flocs of amorphous $\text{Al}($OH$)_3(s)$ (Eqs. (1)–(4)). At the same time, gaseous hydrogen and oxygen liberate at the cathode and the anode, respectively as a result of application the electricity in the system (Eqs. (2) and (4)). Those gases enable collecting flocs at the liquid surface through flotation [9] whereas heavier flocs settle down at the bottom of cell via sedimentation.

At the anode:

\[ (\text{Al})_{(s)} \rightarrow \text{Al}^{3+}_{(aq)} + 3e^- \]  
\[ 2\text{H}_2\text{O} \rightarrow \text{O}_2(g) + 4\text{H}^+ + 4e^- \]

At the cathode:

\[ 2(\text{H}_2\text{O} + e^- \rightarrow 0.5\text{H}_2 + \text{OH}^-) \]

In solution:

\[ \text{Al}^{3+} + 3\text{OH}^- \rightarrow \text{Al}($OH$)_3(s) \]

Electrocoagulation has been efficaciously used to treat numerous types of wastewaters. total petroleum hydrocarbon from the waste-water of oil refinery [10]; Manganese, Copper and Zinc from oil refinery wastewater [11]; wastewaters produced from textile industry [12]; Chromium removal from tannery wastewater [13]; phosphate removal from mining wastewater [14]; color removal from sugar beet molasses [15]; Al and Zr removal from can manufacturing wastewater [16]; and arsenic from groundwater by electrocoagulation [17].

In the present study, the process of electro-coagulation has been implemented for removing COD from the wastewater of petroleum refinery that resulted from Al-Dewaniya petroleum refinery plant using a batch electrochemical reactor in which the impacts of different parameters like current density, pH, and NaCl concentration on the efficiency of COD removal have been researched.
2. Experimental work

Samples of petroleum refinery effluent were delivered by Al-Dewaniya petroleum refinery plant. Sample (20L) was collected from the feed tank to the biological treatment unit and stored in closed containers at temperature 4 °C until use. Table 1 illustrates the characteristics of this sample. It is also included the properties of effluent taken from the settling tank of the final stage of the biological treatment that was measured by petroleum refinery plant administration with the permissible limit for comparison.

| Test          | feed tank sample | settling tank* | Permissible limit* |
|---------------|------------------|----------------|--------------------|
| COD (mg/l)    | 250              | 65             | 100                |
| pH            | 7.8              | 7.5            | (6-9.5)            |
| T.D.S (mg/l)  | 1610             | 1680           | -----              |
| Cl⁻ (mg/l)    | 931              | 119            | 100                |
| SO₄²⁻ (mg/l)  | 560              | 400            | 400                |
| Turb. (NTU)   | 4.17             | 6.44           | 41.3               |
| Cond. (μS/cm) | 3130             | -----          | -----              |
| Phenol (mg/l | 0.715            | (0.01-0.05)    | 0.06               |

*provided by Al-Dewaniya petroleum refinery plant administration

A rectangular lab-scale batch electrochemical reactor made from Perspex glass having a working volume of 1.25 L (120 mm width x 120 mm length x 120 mm height) provided with Perspex cover (150 mm width x 150 mm length x 28 mm thickness) was used for the electrocoagulation treatment experiments. The cover contains slits for electrodes fixation and holes for inserting the probe of pH-meter and conductivity meter. A parallel plate configuration was adopted for the electrochemical reactor where three stainless steel plate cathodes (130 mm length x 80 mm width x 3 mm (thickness)) and two Aluminum anodes (130 mm length x 80 mm width x 3mm (thickness)) have been utilized. The inter-electrode gap has been fixed at 1.5 mm. A digital power supply of the direct current, Type (UNI-T, UTP3315PF) was used to provide constant current throughout the experimental process. The test solution has been agitated with a magnetic stirrer to achieve proper mixing then put in the cell. All the experiments were carried out at constant temperature 30 °C using water bath (Memmert, type: WNB22, Germany). Schematic diagram of the EC experimental setup is shown in Fig.1 with the supporting information. The pH of electrolyte was measured using a digital pH meter (HNNA Instrument Inc, PH211, Romania) and the electrolyte acidity was adjusted using HCl or NaOH for the appropriate experimental conditions. Conductivity and TDS were measured by using (HM digital Inc. model COM-100, Korea). The samples were collected and analyzed for pollutant degradation for every 10 minutes of electrochemical treatment.
The pollutant concentration is given in terms of chemical oxygen demand (COD). Amount of COD in the effluents of petroleum refinery has been specified by taking a sample (2ml) of effluent digested by an oxidizing agent (K$_2$Cr$_2$O$_7$) for 120 minutes at 150°C in a COD thermos-reactor (RD-125, Lovibond). The digested sample was cooled down to the room temperature and after that analyzed in spectro-photometer (MD-200, Lovibond). Phenol was measured by using Method 8047 assigned by Hach Company/Hach Lange GmbH, USA [18]. COD and phenol detection were performed for three times and the mean values have been considered in this work.

The removal efficiencies of COD and phenol were calculated according to equation (5), where $C_i$ is the initial (mg L$^{-1}$) and $C_f$ is the final concentration (mg L$^{-1}$) [19]:

$$RE\% = \frac{C_i-C_f}{C_i} \times 100$$  \hspace{1cm} (5)

The electrical energy consumption (EC) is the amount of energy which has been consumed in the process for digesting one kg of COD. EC in (kWh/kg) can be obtained using the equation (6), [20]:

$$EC = \frac{U \cdot I \cdot t \times 1000}{(COD_i-COD_f) \cdot V}$$  \hspace{1cm} (6)

Where EC is the energy consumption (kWh/kg COD), U is the applied cell voltage (Volt), I is the current (A), t is the electrolysis time (h), COD$_i$ and COD$_f$ are the initial chemical oxygen demand and final chemical oxygen demand (mg/l), and V is the volume of effluent (L).

3. Results and discussion

3.1 Effect of current density

It has been stated that the performance of an electrocoagulation process is entirely influenced by the current density amongst the others operating parameters since current density determines the dissolution rate of the coagulant from the electrode into the solution [21]. Figure 2 shows the abatement of COD with time at different current densities using effluent with an initial COD of 250...
ppm and containing NaCl at concentration of 2 g/l with adjusted acidity initially at pH 7. It was clear that increasing current density leads to decrease the final COD value of the effluent. It could be also observed that the degradation efficiency goes down gradually as degradation continues. This behavior can be interpreted as the increasing of current density results in increasing the rate of anodic dissolution of aluminum which in turn causes higher amount of the production of precipitant and coagulant. Accordingly, this has led to higher efficiency in organics removal. Besides, previous works confirmed that increasing current density results in a decreasing in bubble size with increasing the rate of bubble generation hence higher efficiency of organics removal by the flotation of H₂, in combination with the coagulation impact was observed [22]. Table 2 shows the removal efficiency of COD at different current densities. It may be ascertained, from Table 2, that the efficiency of COD removal enhances with enhancing the applied current density where a complete COD removal was obtained at current density equal or higher than 16 mA cm⁻² at electrolysis time of 60 min. Energy consumption and its cost is one of the most efficient parameters in applying any approach for waste-water treatment. Amongst the electro-coagulation treatment operating cost, the consumption of electric energy and aluminum electrodes consumption are of the highest importance. Electric (EC) can be characterized as the quantity of electric energy consumed per unit mass of the removed organic load [21]. As it has been listed in Table 2, increasing the density of the current would increase the electric EC by the increase in ohmic voltage losses in the cell. Using current density higher than 12 mA cm⁻² results in increasing power consumption higher than (29.12 Kwh/Kg COD). Therefore it is the best to choose this current density for further investigation. Similar observations were found in previous studies [19, 23, 24].

In general, the medium pH varies throughout the operating process and this variation is dependent upon the initial pH and the electrode material type. However, the process of energy consumption shows some capacity of buffering, particularly in alkaline mediums, preventing large variations in the value of pH and decreasing the effectiveness of pollutant removal [25]. Figure 3 illustrates how the pH affected by the current density through the operation where pH was decreased for the first 20 min then started to increase for further electrolysis time. Similar trend was observed by previous studies [26, 27, 28].

![Figure 2](image-url)

**Figure 2.** Abatement of chemical oxygen demand with time at various current densities. pH = 7, [NaCl] = 2 g/l, Temp. = 30°C.
Table 2. Effect of current density on COD removal efficiency

| Current density mA/cm² | COD Removal (%) at 30 min | COD Removal (%) at 60 min | EC (kWh/kg) at 30 min |
|------------------------|---------------------------|---------------------------|-----------------------|
| 4                      | 68                        | 89.2                      | 5.35                  |
| 8                      | 72                        | 91.6                      | 14.99                 |
| 12                     | 88                        | 96.8                      | 29.12                 |
| 16                     | 96                        | 100                       | 45.43                 |
| 20                     | 96.8                      | 100                       | 75.23                 |

Figure 3 pH profile with time at different current density

3.2 Effect of NaCl concentration

NaCl solution was designated as supporting electrolyte due to its several benefits, i.e., the ability of chloride ions in reducing the adverse effects of other anions, like HCO₃⁻ and SO₄²⁻, for instance, existing of carbonate ion would result in the precipitation of Ca²⁺ or Mg²⁺ ions as an insulating layer on the electrodes surface leading to abruptly increase electro-chemical cell ohmic resistance and resulted in significantly reducing the effectiveness of COD removal [29]. Figure 4 displays the abatement of chemical oxygen demand with time at different NaCl concentration using effluent with acidity adjusted initially to pH 7. It was clear that increasing NaCl concentration leads to a decrease in the final COD value of the effluent. Table 3 reveals this behavior where the effect of NaCl concentration on the removal efficiency of COD is reported. It can be seen that as sodium chloride concentration increases from no addition to the addition of 4 g/L, the removal efficiency of COD was decreased from 100% to 68% at abatement time of 30 min, while from 100% to 85.2% at abatement time of 60 min. It was observed that a complete COD removal could be obtained with and without addition of NaCl up to 1g/l at abatement time of 60 min. This is an interesting result which confirm that chlorine ions which originally existing in the petroleum refinery effluent have the ability to degrade the pollutants even at low concentration. Table 3 shows the energy consumption at different NaCl concentration. Increasing NaCl concentration resulted in increasing
conductivity leading to less resistance in the solution which in turns decreases the required voltage for reaching the needed density of current. As a result, this decreases the electric EC [30]. It is preferred to conduct further experiments at NaCl concentration of 2 g/L in which a removal efficiency of COD is not decreased than 96.8%. Besides a negligible change in the energy consumption was observed at NaCl concentration higher than 2 g/L. Similar observations were found in previous studies [19, 23, 24].

Figure 5 shows how the pH affected by NaCl concentration through the operation where pH was decreased for the first 10 min then started to increase for further electrolysis time. Similar trend was observed by previous studies [26, 27, 28].

![Image of COD removal efficiency with NaCl concentration](image)

**Figure 4.** Chemical oxygen demand abatement with time at various concentrations of NaCl.
I=12mAcm\(^{-2}\), pH=7, Temp. = 30°C.

**Table 3.** Effect of NaCl concentration on COD removal efficiency

| NaCl Conc. (g/l) | Initial conductivity μs/cm | COD Removal (%) | EC (kWh/kg) |
|------------------|-----------------------------|-----------------|-------------|
|                  |                             | at 30 min       | at 60 min   |             |
| 0                | 3130                        | 95.2            | 100         | 49.3        |
| 1                | 4800                        | 93.6            | 100         | 40.28       |
| 2                | 6980                        | 88              | 96.8        | 29.12       |
| 3                | 7070                        | 80              | 92.4        | 31.52       |
| 4                | 8500                        | 68.4            | 85.2        | 31.03       |
Effect of pH

Aluminum ions may be existed in various forms and phases, depending on the solution chemical characteristics and pH. At values of pH which are < 4, ions of aluminum presents in the form Al(H₂O)₆⁺³, whereas, a pH that ranges between 5 and 6, aluminum presents in the forms Al(OH)⁺² and Al(OH)₂⁺. When the pH increases to higher values (5.5 to 8.8), aluminum altered to the Al(OH)₃ form. pH values that are > 8.8 could result in aluminum dissolution as ions again[31].

According to the mentioned pH impact on Al’s chemical form, it is expected that the effectiveness of electro-coagulation would depend on the pH, to some level. Five experiments have been carried out at initial values of pH that were equal to 3, 5, 7, 9, and 11 for investigating this impact. Figure 6 displays the abatement of COD with time at different pH using effluent containing NaCl at concentration of 2g/l at a current density of 12mA/cm², while Table 4 illustrates the impact of pH on the removal efficiency of COD. It can be seen that the preliminary value of pH has a significant impact on COD removal efficiency. Increasing or decreasing pH beyond 7 resulted in decreasing removal efficiency and a maximal efficiency of chemical oxygen demand removal has been recorded at pH value of 7. Similar observations were found in previous studies [19, 23, 24].

The pH profile with time at different initial pH is illustrated in Figure 7 where the pH was decreased for the first 10 min then started to increase for further electrolysis time. Similar trend was observed by previous studies [26, 27, 28].

It was clear that the optimum conditions can be attained by adjusting the initial pH to a value of 7, adding NaCl at concentration of 2g/l, and applying a current density of 12
mA cm$^2$ with operating the system for an interval of time equal to 60 min. In this case a removal efficiency of COD (96.8%) with a phenol removal efficiency of 64.7% and TDS removal efficiency of (20.6%) were achieved in which an energy consumption of (29.12kWh/kgCOD ) was required.

**Figure 6.** Abatement of chemical oxygen demand with time at various pH values.
I=12mAcm$^2$, [NaCl] = 2g/l, Temp. = 30°C.

**Table 4.** Effect of pH on COD removal efficiency

| pH | COD Removal (%) | EC ( kWh/kg ) |
|----|----------------|---------------|
|    | at 30 min | at 60 min |               |
| 11 | 76       | 90       | 32.30         |
| 9  | 82       | 92.8     | 33.47         |
| 7  | 88       | 96.8     | 29.12         |
| 5  | 80       | 88.4     | 32.27         |
| 3  | 74       | 86.8     | 33.77         |
4. Conclusions

In this work, chemical oxygen demand removal from a petroleum refinery waste-water which is generated by Al-Dewaniya petroleum refinery plant has been researched with the use of the electro-coagulation technique. The results of the present work have shown that electrocoagulation can be utilized successfully for removing COD. The effects of different parameters such as current density, NaCl concentration, and pH on the treatment of this wastewater in a batch electrocoagulation system were investigated. Current density was found to be the most important and crucial parameters. Addition of NaCl improves the economy of the process via reducing energy consumption. It can be concluded that the optimal initial value of pH of petroleum refinery waste-water to be treated via electrocoagulation with the use of the electrodes of aluminum ranges between 6 and 7, where the majority of the ions of aluminum are in Al(OH)$_3$ form [31]. By adjusting initial pH to a value of 7, adding NaCl at concentration of 2g/l, and applying current density of 12 mA cm$^{-2}$ for an interval of time equal to 60 min, a removal efficiency of COD(96.8%) with phenol removal efficiency of 64.7% and removal efficiency of TDS (20.6%) were achieved in which energy consumption of (29.12 kWh/kg COD) was observed. The energy consumption in the present work is relatively closed to that reported by previous works in the field treatment of petroleum refinery effluent by electrocoagulation technique [19, 23, 24].

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