



## Electrochemical removal of phenol from oil refinery wastewater

O. Abdelwahab<sup>a,\*</sup>, N.K. Amin<sup>b</sup>, E-S.Z. El-Ashtoukhy<sup>b</sup>

<sup>a</sup> Environmental Division, National Institute of Oceanography and Fisheries, Kayet Bay, El-Anfushy, Alexandria, Egypt

<sup>b</sup> Chemical Engineering Department, Faculty of Engineering, Alexandria University, Alexandria, Egypt

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### ABSTRACT

This study explores the possibility of using electrocoagulation to remove phenol from oil refinery waste effluent using a cell with horizontally oriented aluminum cathode and a horizontal aluminum screen anode. The removal of phenol was investigated in terms of various parameters namely: pH, operating time, current density, initial phenol concentration and addition of NaCl. Removal of phenol during electrocoagulation was due to combined effect of sweep coagulation and adsorption. The results showed that, at high current density and solution pH 7, remarkable removal of 97% of phenol after 2 h can be achieved. The rate of electrocoagulation was observed to increase as the phenol concentration decreases; the maximum removal rate was attained at 30 mg L<sup>-1</sup> phenol concentration. For a given current density using an array of closely packed Al screens as anode was found to be more effective than single screen anode, the percentage phenol removal was found to increase with increasing the number of screens per array. After 2 h of electrocoagulation, 94.5% of initial phenol concentration was removed from the petroleum refinery wastewater. Energy consumption and aluminum Electrode consumption were calculated per gram of phenol removed. The present study shows that, electrocoagulation of phenol using aluminum electrodes is a promising process.

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### 1. Introduction

Treatment of industrial wastewaters is a problem of major concern nowadays. More strict regulations are being imposed, which persevere on the need to develop and employ treatment technologies capable to deal with the hazardous pollutants present in many industrial waste streams [1]. Wastewater containing phenolic compounds presents a serious discharge problem due to their poor biodegradability, high toxicity and ecological aspects [2]. Phenols are widely distributed as environmental pollutants. They exist in different concentrations in wastewaters disposed from many industrial processes, including coking, synthetic rubber, plastics, paper, oil refineries, petrochemical, ceramic, steel, conversion processes and phenolic resin industries [3,4]. Phenols are considered as priority pollutants since they are harmful to organisms at low concentrations and many of them have been classified as hazardous pollutants because of their potential harm to human health [3]. Due to the high toxicity of phenols, they are subjected to specific

regulations, and their industrial use is increasingly avoided by substituting them with harmless compounds [5]. The Environmental Protection Agency (EPA) calls for lowering phenol content in the wastewater to less than 1 mg L<sup>-1</sup> [6].

Wastewaters containing phenols and other toxic compounds need careful treatment before discharge into the receiving bodies of water. Biological treatment, activated carbon adsorption, solvent extraction, chemical oxidation and electrochemical methods are the most widely used methods for removing phenol and phenolic compounds from wastewaters [7–12]. Such problems as high cost, low efficiency, and generation of toxic by-products are associated with the above methods [13]. Biological and electrochemical methods have little or no harmful effects on the environment, because these techniques do not involve the use of harmful reagents. On the other hand, electrochemical reactions are more or less independent of the condition of the wastewater and can proceed as long as a current is supplied to the electrode [14].

Electrocoagulation is the process utilizing “sacrificed” anodes to form active coagulant which is used to remove pollutant by precipitation and flotation in situ. Compared with traditional chemical coagulation, electrocoagulation has, in theory, the advantage of removing the smallest colloidal particles; the smallest charged particles have a greater probability of being coagulated because of the electric field that sets them in motion. It has also the advantage of producing a relatively low amount of sludge [15]. EC process

Abbreviations: c.d, current density; COD, chemical oxygen demand; EC, electrocoagulation; E.Consumption, energy consumption; Al Consumption, aluminum electrode consumption.

\* Corresponding author. Tel.: +20 121093161.

E-mail address: [olaabdelwahab53@hotmail.com](mailto:olaabdelwahab53@hotmail.com) (O. Abdelwahab).

### Nomenclature

|       |  |
|-------|--|
| $C_f$ | final phenol concentration ( $\text{mg L}^{-1}$ )    |
| $C_i$ | initial phenol concentration ( $\text{mg L}^{-1}$ )  |
| $F$   | Faraday's constant ( $\text{C mol}^{-1}$ )           |
| $I$   | applied current (A)                                  |
| $m$   | weight of treated wastewater (g)                     |
| $M$   | molecular weight of aluminum ( $\text{g mol}^{-1}$ ) |
| $t$   | retention time (s)                                   |
| $V$   | voltage (V)  |
| $z$   | number of electron transfer                          |

### Greek letter

|           |                 |
|-----------|-----------------|
| $\lambda$ | wavelength (nm) |
|-----------|-----------------|

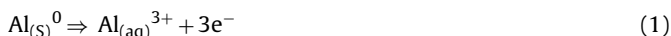
reportedly requires less space and does not require chemical storage, dilution, and pH adjustment [16]. Therefore, electrocoagulation has been widely used to treat water containing food and protein wastes, oil wastes, synthetic detergent, effluent mine wastes and heavy metal containing solutions [17–19].

In the present work, the removal of phenol from aqueous solution was investigated using a horizontally oriented electrodes made of Al electrodes; an Al cathode resting on the cell bottom while the anode was made of a horizontal single screen or an array of closely spaced Al screens placed at a short distance from the cathode. The use of an array of closely spaced screens as anode offers the advantage of prolonging the anode life. Several parameters, namely initial pH, conductivity, current density, phenol concentration, NaCl concentration and electrolysis time, were investigated for their effects on the removal efficiency. The corresponding energy consumption and electrode consumption are also determined as principal cost parameters. Furthermore, the removal efficiency was studied using actual wastewater effluent to explore the applicability of the technique in industry.

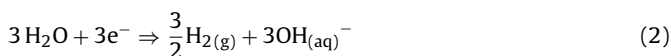
## 2. Theory of EC

Electrocoagulation occurs via serial steps such as (i) electrolytic reactions at electrode surfaces, such as Al, Al ions form at the anode and hydroxyl ions are generated at the cathode, (ii) in situ oxidation of Al ions and subsequent precipitation of aluminum hydroxide in aqueous phase, and (iii) adsorption of soluble or colloidal pollutants on coagulants which are removed by sedimentation or flotation. The main reactions are as follows [20]:

Anode:

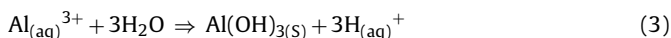


Cathode:



$\text{Al}^{3+}$  and  $\text{OH}^-$  ions generated by electrode reactions (1) and (2) react to form different monomeric and polymeric species, which transform finally into  $\text{Al}(\text{OH})_{3(s)}$ .

Solution reaction:



Freshly formed amorphous  $\text{Al}(\text{OH})_{3(s)}$  "sweep flocs" have large surface areas which is beneficial for a rapid adsorption of soluble organic compounds and trapping of colloidal particles. Finally, these flocs are removed easily from aqueous solution by sedimentation or floatation [21,22]. On the other hand, at high pH values,

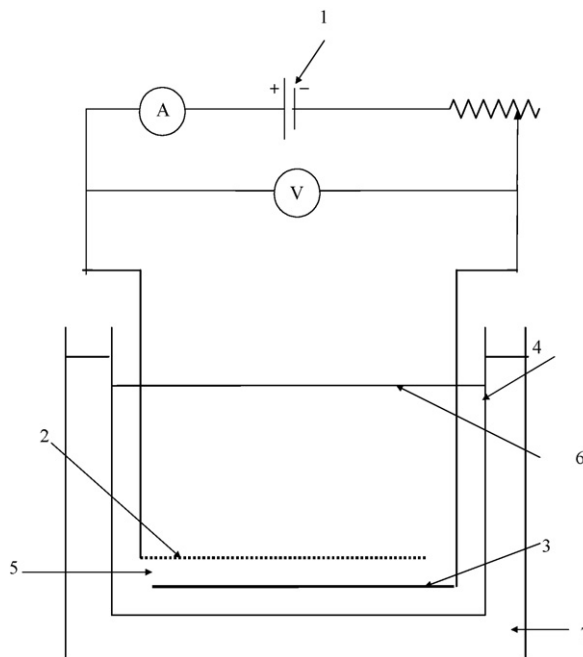
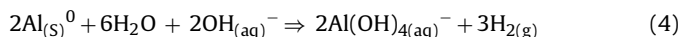


Fig. 1. Schematic view of the experimental system. (1) dc power supply, (2) anode, (3) cathode, (4) reactor, (5) electrode gap, (6) level of solution and (7) water bath.

both Al cathode and anode may be chemically attacked by  $\text{OH}^-$  ions in view of the amphoteric nature of Al [21].



Accordingly, two major interaction mechanisms are being proposed namely: precipitation and adsorption, each one being suggested for a separate pH range. Flocculation in the low pH range is explained as precipitation while the higher pH range (>6.5) as adsorption [22,23].

## 3. Experimental

### 3.1. Chemicals and analytical

Stock solution of the phenol ( $1000 \text{ mg dm}^{-3}$ ) was prepared by weighing the purified grade chemicals and dissolving it in distilled water. Experimental solutions of the desired concentration were obtained by successive dilution. All reagents were of AR grade. pH of the solution was adjusted by means of HCl and/or NaOH solution.

An oil refinery wastewater was obtained from SIDI KERIR petrochemicals company, a petroleum refinery located in Alexandria, Egypt. It was exposed to electrocoagulation using the experimental apparatus shown in Fig. 1. The analysis of wastewater is illustrated in Table 1.

The analytical determination of phenol was carried out using UV-spectrophotometer (Shimadzu, Model UV-160) by analyzing the colour resulting from the reaction of phenol with

Table 1  
Physical characteristic of wastewater before and after electrocoagulation treatment

| Parameter            | Analysis before treatment | Analysis after treatment |
|----------------------|---------------------------|--------------------------|
| pH                   | 8                         | 9                        |
| Phenol content (ppm) | 13                        | 1                        |
| TSS (ppm)            | 22.8                      | 8                        |
| BOD (ppm)            | 40.25                     | 39                       |
| COD (ppm)            | 80–120                    | 100                      |
| Turbidity (NTU)      | 7.4                       | 4.8                      |

4-aminoantipyrine at maximum wavelength  $\lambda = 500$  nm. COD was measured using COD reactor (HACH) and spectrophotometer. Current to the cell was supplied by a dc power source (Statron Type 3240.2) and was measured by a multimeter (Fluke 26III True RMS multimeter).

### 3.2. Electrocoagulation test

The cell used in this study was a Plexiglass rectangular container having the dimensions (25 cm  $\times$  25 cm  $\times$  30 cm). The cell was surrounded by thermostatic water bath for temperature control. The performance of the cell was evaluated in batch mode. The experimental setup is shown in Fig. 1. A horizontal aluminum plate was used as the cathode while, a horizontal aluminum screen (mesh no. 10) acted as the sacrificial anode. The dimensions of the electrodes were 25 cm  $\times$  25 cm. The two electrodes were separated by a distance of 1 cm. During electrolysis, the current density was maintained constant by using a voltage regulator. For each run, 3.5 L of phenol solution was placed into the cell. The electrolyte, NaCl, was added to the phenol solution at the required concentration, then the current density was adjusted to the desired value and electrolysis was conducted for a certain time. Samples were drawn periodically during each experiment, filtered to separate the sludge then were analyzed for phenol concentration.

Before each run, organic impurities on electrode surfaces were removed by washing with acetone and HCl solution (10 wt%), respectively and then washed with distilled water prior to use. The reproducibility of the experiments presented was carefully checked. The efficiency of phenol removal, % Removal, was calculated as

$$\% \text{ Removal} = \frac{C_i - C_f}{C_i} \times 100 \quad (5)$$

where  $C_i$  is the initial phenol concentration ( $\text{mg L}^{-1}$ ) and  $C_f$  is the final phenol concentration ( $\text{mg L}^{-1}$ ).

## 4. Results and discussion

### 4.1. Influence of initial pH

It has been established that the pH has a considerable influence on the performance of electrocoagulation process [24]. The phenol removal efficiency was determined in the pH range from 3 to 10. The results are shown in Fig. 2, from which the variation of removal efficiency of phenol with the solution initial pH could be clearly identified. The efficiency of removal was very low either at low

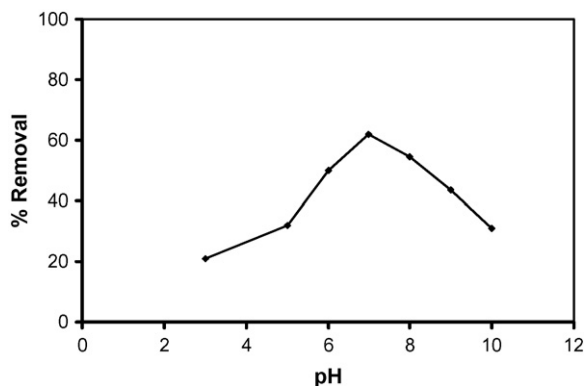


Fig. 2. The effect of pH on the phenol removal efficiency (c.d: 19.3 mA/cm<sup>2</sup>, initial phenol concentration: 50 mg/L, NaCl: 2 g/L, temp: 20 °C and time: 2 h).

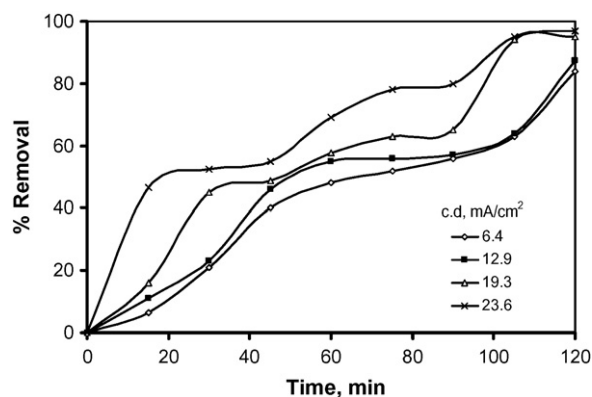


Fig. 3. Effect of electrolysis time and c.d. on phenol removal efficiency ( $C_0$ : 50 mg/L, NaCl: 2 g/L, pH: 7, and temp: 20 °C).

pH or at high pH. The optimal pH was 7, at which higher phenol removal efficiency could be reached. This behavior was attributed to the amphoteric character of aluminum hydroxide which does not precipitate at very low pH [25]. Moreover, high pH leads to the formation of  $\text{Al}(\text{OH})_4^-$ , which is soluble and useless for adsorption of phenol [16]. Therefore, further increase of the influent pH would decrease the phenol removal efficiency.

### 4.2. Influence of operating time and current density

Operating time experiments were carried out at pH 7 for initial phenol concentration of 50 mg L<sup>-1</sup> in the presence of 2 g L<sup>-1</sup> NaCl. Batch electrocoagulation experiments were conducted for 2 h for different current densities. Fig. 3 depicts the effect of current density, c.d., on electrocoagulation, removal efficiency increases with increasing time. After 120 min the maximum removal efficiency of phenol were 84, 88, 95 and 97% for current densities of 6.4, 12.9, 19.3 and 23.6 mA cm<sup>-2</sup>, respectively. This is ascribed to the fact that at high current densities the dissolution of anode electrode increases according to Faraday's law, the resulting aluminum hydroxides produces more sludge with a consequent significant removal of phenol due to sweep coagulation [26]. Furthermore, more bubbles are generated at higher current and this improves the degree of mixing of  $\text{Al}(\text{OH})_3$  and phenol which enhances floatation ability of the cell with a consequent increase in the phenol removal efficiency [27].

For the same operating conditions, after 2 h of electrocoagulation, consumption of energy and electrode material are also

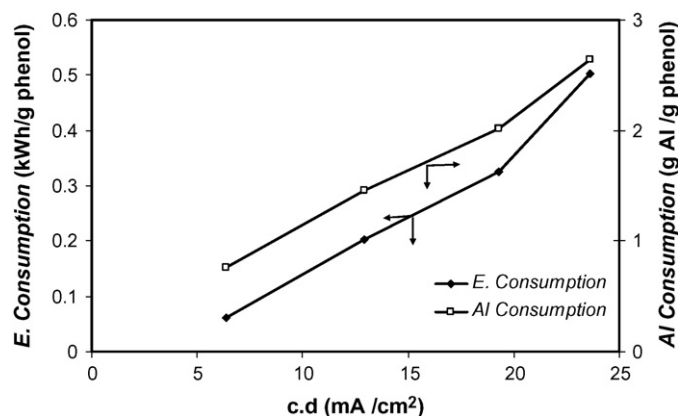


Fig. 4. Effect of current density on energy consumption and electrode consumption ( $C_0$ : 50 mg/L, NaCl: 2 g/L, pH: 7, temp: 20 °C, and time: 2 h).

represented in Fig. 4. Energy consumption is expressed as

$$E. \text{Consumption} = \frac{IVt}{m} \quad (6)$$

where  $E. \text{Consumption}$ ,  $I$ ,  $V$ ,  $t$  and  $m$  are energy consumption ( $\text{kWh g}^{-1}$  of phenol removed), applied current (A), voltage (V), retention time (s) and the weight of the treated wastewater (g), respectively. The aluminum electrode consumption (Al Consumption) having a unit of  $\text{g Al g}^{-1}$  of phenol removed is calculated from Faraday's law from the following relation:

$$\text{Al Consumption} = \frac{ItM}{zFm} \quad (7)$$

where  $F$  is the Faraday's constant ( $96,500 \text{ C mol}^{-1}$ ),  $M$  the molecular weight of aluminum ( $27 \text{ g mol}^{-1}$ ) and  $z$  is the number of electron transfer ( $z_{\text{Al}} = 3$ ), respectively. Al consumption and  $E. \text{consumption}$  were found to increase with increasing the current density (Fig. 4). An increase in current density from  $6.4$  to  $23.6 \text{ mA/cm}^2$  causes an increase in energy consumption from  $0.061$  to  $0.502 \text{ kWh g}^{-1}$  and increase electrode consumption from  $0.761$  to  $2.638 \text{ g Al g}^{-1}$  of phenol.

#### 4.3. Influence of initial phenol concentration

Fig. 5 represents the relation between the percentage removal of phenol and initial solution concentration. The percentage removal was gradually decreased from 99 to 34% as the phenol concentration increased from 30 to  $250 \text{ mg L}^{-1}$ . This is may be attributed to the fact that at a constant current density the same amount of aluminum ions passes to the solution at different phenol concentrations. Consequently, the formed amount of complex aluminum hydroxide was insufficient to coagulate the greater number of phenol molecules at higher phenol concentrations [22]. By increasing the concentration of phenol the energy consumption was decreased from  $0.52$  to  $0.18 \text{ kWh g}^{-1}$  phenol, while electrode consumption slightly increased from  $0.02$  to  $0.07 \text{ g Al g}^{-1}$  phenol as shown in Fig. 6.

#### 4.4. Influence of sodium chloride

A nominal value of NaCl concentration in the range from 1 to  $2.5 \text{ g L}^{-1}$  was used to increase the ionic conductivity of the solution in order to minimize energy consumption. Furthermore, the presence of chloride ions remove the passive oxide layer form on electrode surface, hence it increases the availability of aluminum hydroxide in the solution and improves the efficiency of phenol removal (Fig. 7) [27]. Fig. 8 shows that, the energy consumption and

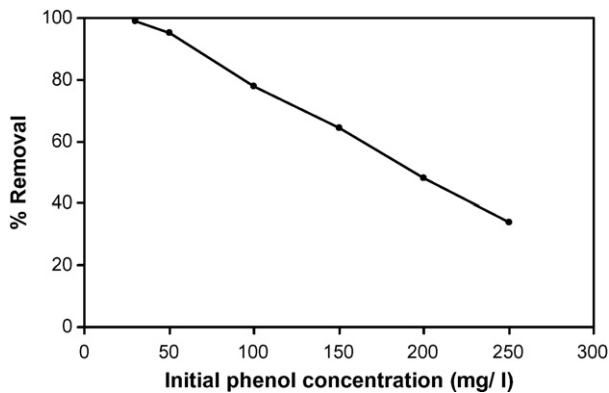


Fig. 5. Effect of initial phenol concentration on phenol removal efficiency (NaCl:  $2 \text{ g/L}$ , pH: 7, time: 2 h, c.d:  $19.3 \text{ mA/cm}^2$ , and temp:  $20^\circ\text{C}$ ).

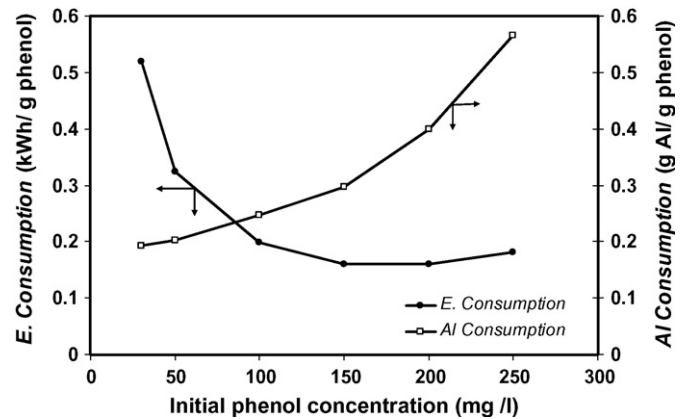


Fig. 6. Effect of initial phenol concentration on energy consumption and electrode consumption (NaCl:  $2 \text{ g/L}$ , pH: 7, time: 2 h, c.d:  $19.3 \text{ mA/cm}^2$ , and temp:  $20^\circ\text{C}$ ).

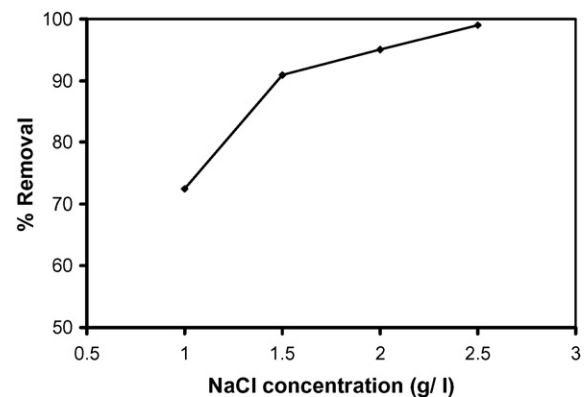


Fig. 7. Effect of NaCl concentration on phenol removal efficiency ( $C_0$ :  $50 \text{ mg/L}$ , c.d:  $19.3 \text{ mA/cm}^2$ , pH: 7, and temp:  $20^\circ\text{C}$ ).

the aluminum consumption decreased from  $0.85$  to  $0.2 \text{ kWh g}^{-1}$  and from  $0.26$  to  $0.19 \text{ g Al g}^{-1}$  phenol removed with increasing NaCl concentration from 1 to  $2.5 \text{ g L}^{-1}$ . This indicates the importance of the presence of NaCl in solution on the economy of electrocoagulation process.

#### 4.5. Influence of anode thickness

The possibility of using an anode made of an array of closely packed screens was investigated in order to prolong the anode life in practice and eliminate the downtime required to fix a new single

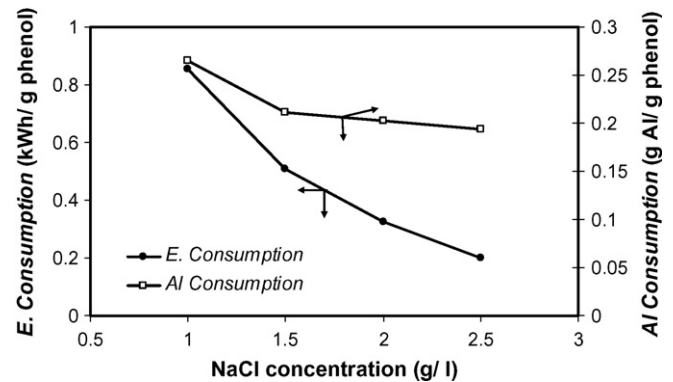
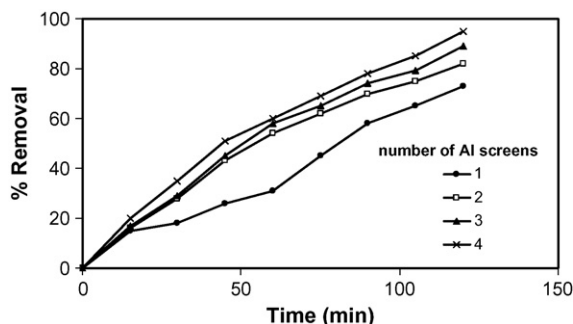
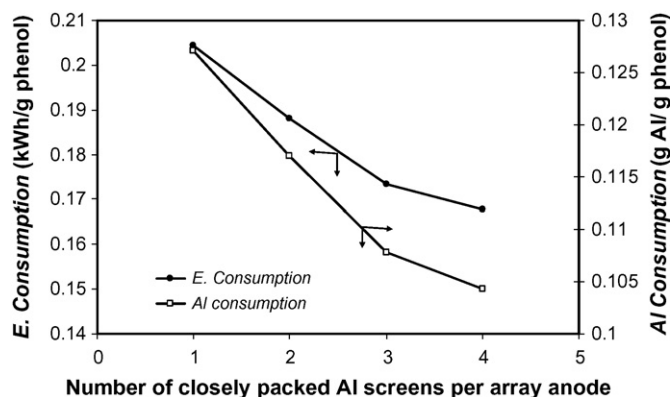


Fig. 8. Effect of NaCl concentration on energy consumption and electrode consumption ( $C_0$ :  $50 \text{ mg/L}$ , c.d:  $19.3 \text{ mA/cm}^2$ , pH: 7, temp:  $20^\circ\text{C}$ , and time: 2 h).

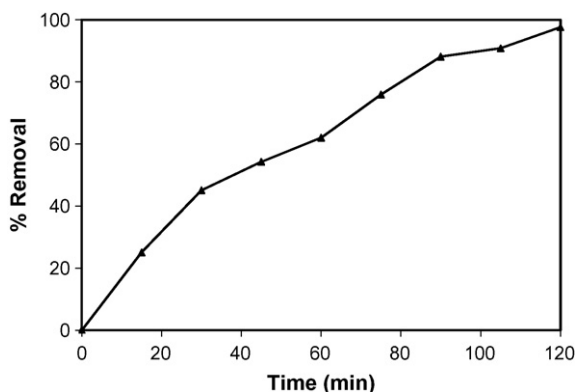


**Fig. 9.** Effect of number of packed Al screens per array anode on phenol removal efficiency ( $C_0$ : 100 mg/L, c.d: 19.3 mA/cm<sup>2</sup>, NaCl: 2 g/L, time: 2 h, pH: 7, and temp: 20 °C).

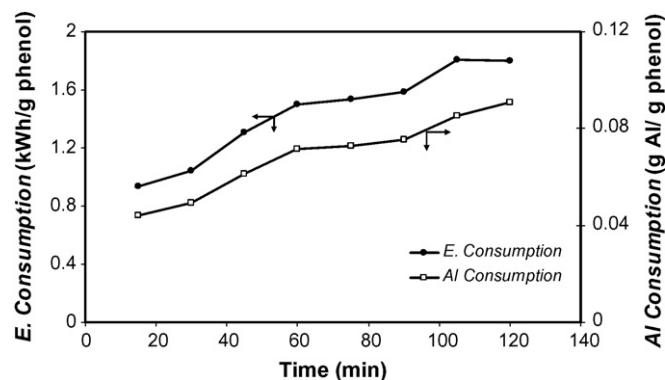


**Fig. 10.** Effect of number of packed Al screens per array anode on energy consumption and electrode consumption ( $C_0$ : 100 mg/L, c.d: 19.3 mA/cm<sup>2</sup>, NaCl: 2 g/L, time: 2 h, pH: 7, and temp: 20 °C).

screen anode. A number of experiments were conducted using an array anode made of 1, 2, 3, 4 closely packed screens. Fig. 9 shows that for a given current density, phenol removal efficiency increases from 75.5 to 92% with the increase in number of screens per array anode from 1 to 4, respectively. This may be attributed to the fact that the rising swarm of H<sub>2</sub> bubbles receives higher resistance from the thick anode with a consequent decrease in its rise velocity; this improves the floating ability of H<sub>2</sub> bubbles and increases the degree of phenol removal. By increasing the number of screens per array anode from 1 to 4, both energy consumption and aluminum consumption were decreased from 0.20 to 0.16 kWh g<sup>-1</sup> phenol and from 0.13 to 0.10 g Al g<sup>-1</sup> phenol, respectively (Fig. 10).



**Fig. 11.** Phenol removal efficiency from oil refinery wastewater (c.d: 19.3 mA/cm<sup>2</sup>, NaCl: 2 g/L, pH: 7, and temp: 20 °C).



**Fig. 12.** Variation of energy consumption and electrode consumption for phenol removal from oil refinery wastewater (c.d: 19.3 mA/cm<sup>2</sup>, NaCl: 2 g/L, pH: 7, and temp: 20 °C).

**Table 2**

Comparison of % phenol removal obtained from different wastewater effluents

| Source of effluent       | Phenol concentration (mg L <sup>-1</sup> ) | % Removal | Reference |
|--------------------------|--|-----------|-----------|
| Petrochemical wastewater | 2.5  | 60        | [28]      |
| Paper mill effluent      | 0.535                                      | 99        | [29]      |
| Present work             | 13   | 92        | –         |

#### 4.6. Treatment of petroleum refinery wastewater

Petroleum refineries are the main sources of phenolic wastewaters. Under the present optimum experimental conditions, a real oil refinery wastewater, which was obtained from SIDI KERIR petrochemicals company, was exposed to electrocoagulation using the experimental apparatus at 23.6 mA cm<sup>-2</sup> current density and pH 7 for 120 min (Fig. 11). The characteristics of wastewater used in the studies before and after treatment are presented in Table 1. After electrocoagulation period, 94.5% of initial phenol concentration was removed from the petroleum refinery wastewater. Compared to phenol removal, the removal of other contaminants in wastewater was not excessively changed. Practically, the effluent met the discharged standards after 2 h electrocoagulation time with energy consumption of 1.8 kWh g<sup>-1</sup> phenol and electrode consumption 0.091 g Al g<sup>-1</sup> phenol (Fig. 12). A few studies reported the removal of phenol from wastewater by electrocoagulation. Table 2 presents a comparison of % phenol removal obtained from different studies on real wastewater effluents.

## 5. Conclusion

Electrocoagulation of phenol using Al screen as a sacrificed anode seems to be a very efficient method for wastewater treatment as revealed by the present study. Experimental parameters such as pH, time, current density, electrolyte concentration, initial phenol concentration and an array of closely packed Al screen anode were investigated for phenol removal. The experimental results revealed that the phenol percentage removal is enhanced by sweep coagulation and adsorption. Increase in the current density, NaCl concentration and thickness of anode resulted in increase in the phenol removal. While it decreases with the increase in initial phenol concentration. Treatment efficiency, in terms of both energy and electrode consumption, is affected by the operating conditions. Energy consumption and electrode consumption of electrochemical coagulation was found for current densities 6.4–23.6 mA cm<sup>-2</sup> to be in the range of from 0.061 to 0.502 kWh g<sup>-1</sup> and from 0.761 to 2.638 g Al g<sup>-1</sup> phenol removed, respectively. An oil refinery

wastewater containing phenol was exposed to electrocoagulation using the present optimum conditions. Oil refinery wastewater of volume 3.5 L containing 13 mg L<sup>-1</sup> phenol, 2 g L<sup>-1</sup> of NaCl as supporting electrolyte having initial pH 8 after electrocoagulation at current density of 19.3 mA cm<sup>-2</sup> for 2 h brings down the phenol concentration to 1 mg L<sup>-1</sup>. It can be concluded from this study that electrocoagulation is a promising technique for phenol removal.

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