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# EFFICIENCY OF AN ELECTROCOAGULATION TREATMENT OF WATER CONTAMINATED BY HYDROCARBONS IN A CONTINUOUS MODE POWERED BY PHOTOVOLTAIC SOLAR MODULES

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### Abstract

The aim of this study is the treatment of liquid effluents from a petroleum industry (NAFTAL /Algeria) by an electrochemical treatment process (electrocoagulation). The electrocoagulation has proved to be useful for the treatment of wastewater, but in order to enhance its ecological characteristic we found it would be essential to carry out our experiments with ecological energy derived from a photovoltaic solar module, which would contribute to sustainability objectives. The electrocoagulation process in a continuous mode using anodes (aluminum and iron) and a graphite cathode was studied. Changes in the Chemical Oxygen Demand were analyzed which stated the efficiency of the method. The current density, the duration of treatment, the pH and the amount of electrolyte added in order to optimize these operating parameters lead to a better reduction of the pollution load. After 1h of electrolysis, optimum conditions were: current density of 100 A/m<sup>2</sup>, with initial pH 7.4 and a flow rate of 3.94.10<sup>-3</sup> L/s. More than 94% of COD were removed with an aluminum anode.

Key words: COD removal, electrocoagulation, electrode, petroleum oil, photovoltaic energy

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# 1. Introduction

Oil is a mixture of hydrocarbons, which are combination of carbon and hydrogen molecules. It is less dense than water, has a characteristic odor and its color varies from black to brown (Cerqueira and Da Costa Marques, 2012). Depending on its origin, it may contain a variety of chemicals such as organic salts, aliphatic and aromatic hydrocarbons, oils and grease, metals and radioactive material (Henderson, 1999, Oliveira et al., 2005). In order to meet the needs of the growing demand for oil, its extraction has risen dramatically in recent decades producing huge amounts of toxic effluents worldwide - 33.6 million barrels a day (Diya'uddeen et al., 2011). A large portion of recoverable wastewater comes from petrochemical activities such as oil extraction and its subsequent refining processes (Rastegar et al., 2017; Maretto et al., 2014); the release of these wastewaters into the environment largely affects the ecosystem. In addition, the failure to observe safety standards and current equipment maintenance in the oil industry aggravates water pollution problems. In order to protect the environment, the treatment and reuse of wastewater has become an absolute necessity. Improved water management in a refinery must reduce the volume and the coast of raw water used in refinery operation, but firstly contaminants should be reduced (Davoudi et al., 2014). For these reasons, innovative and inexpensive techniques for the treatment of

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wastewater in the oil industry should be developed. In recent years, there has been a growing interest for the use of electrochemical methods for the treatment of industrial wastewater especially electrocoagulation (EC). This process is well known in wastewater treatments (Martinez-Huitle and Brillas, 2009; Mohammed et al., 2009). In fact, it has been successfully used for the treatment of drinking water (Pop et al., 2017; Vik et al., 1984) and wastewaters generated from aquaculture (Lin and Chen, 1997), industries (Ramirez, 1981), textile industry (Lin and Peng, 1996; Pajootan et al., 2016), tannery industry (Benhadji et al., 2011), restaurants (Chen et al., 2000), landfill leachate, municipal sewage (Biwyk et al., 1980) and urban wastewater (Pouet and Grasmick, 1995). This process was used to remove phenol (Awad and Abuzaid, 2000) and surfactants (Ciorba et al., 2000) and even the oil industry (El-Naas et al., 2009; Hariz et al., 2013) used it to cut oil-water emulsions (Bensadok et al., 2008), oil suspension used for machining and drilling operations (Pansares et al., 2008) and for oil drilling mud treatment (Ighilahriz et al., 2013). Electrocoagulation (EC) is an effective method wherein the flocculating agent is produced by the electro-oxidation of a single-use anode made of iron or aluminum mostly. This process turns a suitable pH into an insoluble metal hydroxide that can remove pollutants. In this process, the treatment is done without the addition of chemical flocculants or coagulants, thereby reducing the amount of sludge that should be removed.

This technique is easy and requires simple equipment and a small amount of sludge (Mollah et al., 2004; Yehya et al., 2015) is produced. Its technology's efficiency and reliability were stated and it provides an environmentally compatible method to reduce a wide variety of pollutants (Ciorba et al., 2000).

During the electrocoagulation process, the electrochemical reactions of electrodes in aluminum are summarized as follows: At the anode Eq. (1):

$$Al_{(s)} \to Al_{(aq)}^{3+} + 3e^{-}$$
 (1)

# At the cathode:

The reaction occurring at the cathode is dependent on pH. Indeed, under neutral or basic conditions hydrogen is produced through Eq. (2), whereas under acidic conditions, Eq. (3) better describes the hydrogen evolution:

$$2H_2O_{(l)} + 2e^- \to H_{2(g)} + 2OH_{(ag)}^- \tag{2}$$

$$2H_{(aq)}^+ + 2e^- \to H_{2(g)} \tag{3}$$

In this study, EC using aluminum and iron electrodes in continuous mode was studied in the treatment of an oily wastewater generated from a petroleum industry at Dar el Beida (Algiers, Algeria). The first purpose of this study was to observe the effects of the main operating variables on the efficiency of the EC process, like the anode materials, the operating time and the current density in order to determine the optimal conditions for it to remove pollutants. The sludge formed was characterized. The second purpose of the study was to explore a new solution for treating oily wastewater with a renewable energy through a photovoltaic solar power which is a new form of non-polluting, abundant and renewable energy. Photovoltaic modules last long and involve low maintenance costs (Valero et al., 2008); they can produce a high direct current when exposed to the sunlight and connected in series and/or in parallel. Based on the above facts, the photovoltaic energy can be used directly in electrocoagulation to treat wastewater, and this is becoming a popular trend in the environmental protection field. The use of the photovoltaic solar power, an eco-friendly energy can produce a high direct current when exposed to sunlight. The photovoltaic module can be installed near a remote or absent power region (Navarro-Solis et al., 2010; Valero et al., 2008; Zhang et al., 2013).

# 2. Material and methods

# 2.1. Water samples

An effluent was selected from NAFTAL (Algeria) oil industry which contains kerosene/ water emulsions taken downstream of the local sewage treatment plant at the industrial site. The sample containers were preserved in a refrigerator at 4°C before use. At the beginning of the study, the main characteristics of the collected samples were analyzed and the water temperature was increased to the room temperature before each electrocoagulation test. Table 1 shows the characteristics of the effluent used during the process.

Parameters	Values	Permissible levels [Decree, 2006]
COD (mg O <sub>2</sub> /L)	292	100
BOD <sub>5</sub> (mg O <sub>2</sub> /L)	30	25
pН	7.50	5.5-8.5
Conductivity (mS/cm)	0.36	-
Turbidity (NTU)	2.32	-

Table 1. The main characteristics of oily wastewater

# 2.2. The electrocoagulation unit

It is composed of two compartments. The first consists of a 10-L cylindrical tank. The second compartment is provided with a cathode of graphite and anodes of cylindrical shapes made of aluminum (with a 0.8-cm internal diameter, a 12.1-cm height with an active area of  $30.39 \text{ cm}^2$ ) and iron (with 0.3-cm internal diameter, a 12.1-cm height with an active area of  $11.39 \text{ cm}^2$ ). The electrodes were placed at the middle bottom of the reactor, parallel to each other in the cell at a fixed distance of 1 cm. The EC cell was fed continuously by a peristaltic pump (Iwaki Magnet

Pump; 16-19 L/min) with the effluents from a wastewater tank. The reactor operates as a closed reactor with full recirculation, the total solution does not exceed 2-L, and electrodes were connected to a DC power supply (HEP-613; 0-2 A; 40-V). The cell was powered by a photovoltaic system. The battery had an internal resistance of 17 m $\Omega$ , a 4.8 A maximum charging current and a minimum safe end discharge voltage of 1.60 V/cell. The SOLAR23 solar module line was especially designed for PV off grid systems and covered modules in the range from 5 to 100 Wp. Samples were taken at various time interval. The experimental equipment is schematically shown in Fig. 1.

The operation lasted 2 h; samples were taken from the reactor every 15 min and were analyzed. Their pH, conductivity and COD were determined. The pH measurement was carried out with a HANNA Digital Display model SED pH meter using 12500 V. The conductivity measurements were made by using a HANNA.COD conductivity meter. COD was measured using a thermo ECO reactor refluxing by titration method following the NFT 90-101 standard. Boiling under reflux in the presence of mercury sulfate (II), a test sample, in the presence of a known amount of potassium dichromate and a silver catalyst, strongly acidified with sulfuric acid for a period of time during which a portion of dichromate was reduced by the oxidizable matter present in the solution. The dichromate excess was titrated with a standard solution of iron sulfate (II) and ammonium. Calculation of COD was done from the amount of the reduced dichromate. BOD<sub>5</sub> analysis was carried out by a Schrank Aqua Lytic Thermostat following the NF T90-103 standard. The sample was incubated for 5 days at 20 °C. The microorganisms consumed the dissolved oxygen and the carbon dioxide released was trapped by sodium hydroxide. The amount of sludge generated after the EC treatment under optimal conditions using aluminum electrodes was characterized using a FT-IR spectrometer (Perkin-Elmer N°200139).

#### 3. Results and discussion

The EC process is quite complex and is affected by several operating parameters such as the

initial pH, the duration of the EC, the nature of the electrode material and the current density. In order to extend the process performance, the effects of those parameters were studied and the results are stated below. The solution was prepared with 1 L of pure effluent and 1 L of distilled water which led us to add electrolyte support to raise the solution conductivity, to decrease the voltage between electrodes due to the decreased resistance of polluted water and to reduce energy consumption.

# 3.1. Effect of current density and electrolysis duration on EC process

The current density is considered as a key parameter in EC (Khemis et al., 2006; Xu and Zhu, 2004) especially for the reduction kinetics of COD. It is knowledged that current density determines the production rate of coagulant as well as the adjustment of bubble production, and hence it affects the growth of flocs (Chen et al., 2004; Mollah et al., 2004). In order to assess the effect of the applied current density, a set of experiments was planned at an initial pH 7.4 (since it was the natural pH of the solution) and varying applied current density (50-125 A/m<sup>2</sup>) for a concentration of supporting salt NaCl of 0.5 g/L.

The COD reduction kinetics goes fast during the first 15–30 min of EC and is almost stabilized thereafter. One can say that when the current density increases, the processing time decreases thanks to the strong electrodes dissolution. In fact, when current density increases the production rate of hydrogen bubbles increases and their size decreases. This highlights an effective elimination of the pollutant by flotation (Ben Hariz et al., 2013). Moreover, the increase of current density entails the increase in the amount of coagulant as a consequence of greater removal rate. At a lower current density, the anode dissolution does not provide a destabilization of particles because of the low anode dissolution rate.

However, some COD removal is noticeable which can be explained by an indirect oxidation of the oil suspension provided by oxidizing species such as CIO<sup>-</sup> allowing the indirect electrochemical treatment of the wastewater (Adhoum et al., 2004). The most significant reduction rate (92 %) occurred at a current density of 100 A/m<sup>2</sup> for 1 h.



Fig. 1. Electrochemical cell

The fact that for higher current densities the COD removal decreases can be explained by an overdose of coagulant, which can carry an excessive adsorption of cations and reversing the charge of the particles. The particles could then be stabilized again (Amirtharajah and O'Melia, 1990). Fig. 2 (a) proves that duration has effects on experimental change results in COD reduction for different current densities.

From Fig. 2 (b) one can infer that the decrease in conductivity, being related to the current density during this spell of time, is due to the precipitation of salts produced during the EC reaction with aluminum electrodes. For the case of the experiments made at  $150 \text{ A/m}^2$  the conductivity is lower. The reason of this finding may be that the increment of current density causes an excessive presence of ions produced during EC leading to lower conductivity measured after decantation. The forthcoming experiments were all run at the current density of  $100 \text{ A/m}^2$ .

#### 3.2. Effect of initial pH on EC process

It is established that the initial pH is a significant factor affecting the electrocoagulation process (Asselin et al., 2008; Yildiz et al., 2008). In order to study its effect on the COD removal and the

final pH, the treated solution was adjusted between pH 4 and pH 10 with 0.1 M sodium hydroxide or 0.1 M hydrochloric acid. The current density was set at  $100A/m^2$  and likewise the NaCl concentration was fixed at 0.5 g/L. Fig. 3 shows that COD removal increased from 34 % for pH 4 to 92 % for pH 7.4. Former studies already revealed that EC gave best results when the initial pH varied from pH 3 to neutral pH 7 forming a majority of aluminum complexes which played an active part during the removal of pollutant through EC (Alaton et al., 2009).

With a pH 8 solution the removal efficiency decreased because of the amphoteric character of the aluminum hydroxide  $Al(OH)_3$  whose solubility increases when the solution becomes either more acidic or alkaline (Bensadok et al., 2008; Koparal and Ogutveren, 2002; Mollah et al., 2004). Moreover, a high pH entails the formation of  $Al(OH)_4^-$ , which is soluble and useless for the removal of oil emulsion (Koren and Syversen, 1995). Therefore, a further increase of the influential pH does not help the COD removal.

During the electrolysis the solution pH changes as shown in Fig. 4 and thus, the final pH was 6.3, 7.2, 7.3, 8.3 for the initial pH of 4, 5, 6, 7.4 respectively, which was due to the OH<sup>-</sup> ions produced in the solution by the water electrolysis.



Fig.2. Effect of current density on: (a) COD and (b) conductivity vs. time. pH=7.4,  $C_{NaCl}=0.5g/L$ ,  $Q=5.79.10^{-3}$  L/s (Aluminum anode)



Fig.3. Effect of initial pH on COD, *j*=100 A/m<sup>2</sup>, t=1h, C<sub>NaCl</sub>=0.5g/L, Q=5.79.10<sup>-3</sup>L/s (Aluminum anode)

We can observe that for a medium alkaline pH of 8, the final pH was not that different (pH = 8.5). For a basic pH of 10, the final pH decreased to 9.3 by the production of H<sup>+</sup>. On one hand, metal hydroxides Al(OH)<sub>3</sub> are formed and on the other hand, the oxidation of water to oxygen can help to reduce the pH of the water to be treated (Chen et al., 2004).



**Fig.4.** Effect of initial pH on pH,  $j=100 \text{ A/m}^2$ , C<sub>NaCl=</sub>0.5g/L, Q=5.79.10<sup>-3</sup> L/s (Aluminum anode)

#### 3.3. Effect of electrolyte concentration on EC process

The conductivity of the reaction mixture is one of the main parameters that influence water treatment by EC and can be increased by the addition of salt (Mollah et al., 2004; Holt, 2005). In our study NaCl was used because it is non-toxic and its cost is moderate (Calvo et al., 2003). Adding some electrolvte support would decrease power consumption and destroy the passivation layer and hence improve the dissolution rate of Al electrodes (Lee and Pvun. 1999). Moreover. the electrochemically generated chlorine was found to be effective in water disinfection (Muthukumar, 2007). When there are chlorides in the liquid waste, Cl<sub>2</sub> and OCl- can be produced from anodic discharge of chloride ions (Chen et al., 2004) by Eqs. (4-6). The hypochlorite ion OCl- is a strong oxidant which could oxidize some of the organic molecules present in wastewater therefore it contributes to the water treatment.

$$2Cl^- + 2e^- \to Cl_2 \tag{4}$$

$$Cl_2 + H_2 0 \rightarrow HOCl + Cl^- + H^+$$
(5)

$$HOCl + H_2 O \to OCl^- + H_3 O^+,$$
 (6)  
pKa: 7.3 at 25°C

In order to study the effect of NaCl on the COD removal (Fig. 5), various concentrations were analyzed (from 0.25 to 1.0 g/L).

#### 3.4. Flow rate effect on EC process

In order to investigate the effect of time on the COD removal efficiency, the flow rate at start (Q) was

gradually increased from  $3.94.10^{-3}$  to  $16.3.10^{-3}$  L/s at 100 A/m<sup>2</sup>; pH = 7.4 and C<sub>NaCl</sub> = 0.5 g/L.



Fig.5. Rate abatement as function of electrolyte concentration: j =100A/m<sup>2</sup>, t=1h, pH=7.4 (Aluminum anode)

According to Fig. 6, the reduction of COD decreased with increasing flow rate. We can conclude that when the flow rate raises the COD reduction drops. Indeed, the lowest the flow rate the highest the residence time is. Therefore, the effluent remains longer time in contact with the coagulant into the reactor and improves the treatment efficiency, in agreement with the results previously obtained by other researchers (Emamjomeh and Sivakumar, 2009). The optimal inlet flow rate could therefore be considered to be 3.94.10<sup>-3</sup>L/s.



**Fig.6.** COD as function of flow rate using *j*=100 A/m<sup>2</sup>, pH=7.4, C<sub>NaCl</sub>=0.5g/L (Aluminum anode)

#### 3.5. Effect of anode material on EC process

The composition of the electrode material is a key element in the electrochemical treatment; the choice of the adequate electrode material is important to reach better removal efficiency.

Aluminum and iron are commonly used because they are cheap. Thus, experiments were conducted for both aluminum and iron anodes under the same operational conditions.

Fig. 7 shows the effect of current density on COD removal. We found out that the rate of COD reduction at a current density of 100 A/m<sup>2</sup> was greater with aluminum electrode whereas it was almost the same at a current density of 50  $A/m^2$ . With a density of 125 A/m<sup>2</sup>, removal was more effective with iron anodes. The trends for COD removal in Fig. 7 by different anode materials (aluminum and iron) did not have the same trends at different current densities this can be explained by the generation of various shapes and multiples species of Aluminum such as monomeric species including,  $Al(OH)^{2+}$ ,  $Al(OH)^{+}_{2}$ , Al(OH)<sup>-</sup><sub>4</sub> and Al<sub>2</sub>(OH)<sup>4+</sup><sub>2</sub> and the polymeric species such as  $Al_2(OH)^{+}_5$ ,  $Al_6(OH)^{3+}_{15}$ ,  $Al_{13}(OH)^{5+}_{34}$ , Al<sub>7</sub>(OH)<sup>4+</sup><sub>17</sub>, Al<sub>8</sub>(OH)<sub>20</sub><sup>4+</sup>, Al<sub>13</sub>O<sub>4</sub>(OH)<sup>7+</sup><sub>24</sub> (Mollah et al., 2001).



Fig.7. Rate abatement as function of current density using pH = 7.4, t=1h,  $C_{NaCl}=0.5$  g/L,  $Q=3.94.10^{-3}$  L/s for aluminum and iron electrodes

Both followed the same trends and the scatter diagram shows that the optimum pH obtained with the aluminum electrodes (7.4) is not the same as with iron (6.0). This can be explained by the fact that aluminum hydroxide has a greater compatibility with the pollutant compared to iron hydroxide.

Fig. 8 compares the COD reduction according to pH with both electrodes and shows the worse results obtained with iron electrodes under the same operating conditions as the experiments with aluminum - namely pHi equal to 7.4, Q = $3.94.10^{-3}$  L/s, j = 100 A/m<sup>2</sup> and [NaCl] = 0.5 g/L. Indeed, a reduction of 95% was achieved with aluminum electrodes against 69% with iron.

#### 3.6. Electrical energy consumption

One of the most important factors to be taken into account for the selection of a water treatment process is the cost. Electrodes, chemicals and the energy used during EC add up to the process operating costs (Ayhan Şengil and Özacar, 2006; Gebologlu, 2010; Kobya et al., 2003, Khandegar and Saroha, 2013). The electrical energy required for the removal of COD using aluminum electrodes was calculated in terms of kWh/kg COD using Eq. (7):

$$E_C = \frac{UIt}{V \times \Delta COD} \times 10^3 \tag{7}$$

where U is cell voltage (V), I is the applied electrical current (A), t is the reaction time (h), V is the reactor volume (L) and  $\triangle$ COD is the subtraction of COD (mg O<sub>2</sub>/L) before and after the electrolysis (COD removed during electrochemical treatment).



**Fig.8.** COD as function of pH using  $j=100 \text{ A/m}^2 \text{ t} = 1 \text{ h}$ , C<sub>NaCl</sub>=0.5g/L, Q=3.94.10<sup>-3</sup> L/s for aluminum and iron electrodes

The chemical consumption (CHC) was calculated in terms of kg of chemicals/kg COD using Eq. (8):

$$CHC = \frac{Chemicals used}{V \times \Delta COD} \times 10^3$$
(8)

The electrode consumption (kg/kg COD) was calculated according to Eq. (9):

$$ENC = \frac{I \times t \times M}{n \times F \times \Delta COD \times V} \times 10^3$$
(9)

where: *M* is the molar mass (kg/mol), *n* is the valence number (= 3) and *F* is the Faraday constant (96485  $\text{C.mol}^{-1}$ ).

As seen in Fig. 9, a band appeared at 3434 corresponding to O-H stretching (Jewel et al., 2007), whereas the peak towards 1643 cm<sup>-1</sup> is due to the presence of bending hydroxyl (Drouiche et al., 2009). The operating cost was calculated from the following expression Eq. (10) (Khandegar and Saroha, 2013):

$$Operating \ cost = aEc + bCHC + cENC \quad (10)$$

where: a is the electrical energy price (0.022 US\$ per kWh), b corresponds to the chemical consumption price (12.60 US\$ per kg of added NaCl) and c is the electrode material consumption (3.8 US\$ per kg of aluminum). Working at 100 A/m<sup>2</sup> during 1 h of electrolysis, Q=  $3.94.10^{-3}$  L/s and C<sub>NaCl</sub> = 0.5 g/L and pH 7.4, the operating cost of EC was 0.0761US\$ per kg of COD, with Algeria price electricity. These costs are in agreement with those given in the literature (Saeedi and Khalvati-Fahlyani, 2011).



Fig.9. FT-IR spectrum of the sludge produced in the electrochemical process

#### 3.7. Characterization of sludge obtained with the EC

The sample obtained under the optimal conditions using aluminum electrodes was filtered to get the sludge formed during the EC in order to be analyzed with the Fourier Transform Infrared Spectroscopy (FT-IR). The characteristic of Al–O-H bending is represented by the band at 872 cm<sup>-1</sup> (Jewel et al., 2007). These results account for a mechanism in which the pollutant was removed from wastewater in the EC process. In fact, the sludge contains aluminum hydroxide in a major part which proves that the Al was turned into sludge.

#### 4. Conclusions

This study confirms that the electrocoagulation process is satisfactory with results reaching 94 % of oil removal (liquid effluent from a petroleum industry). The optimum conditions are 1 h of electrolysis and a current density of  $100A/m^2$  with an initial pH 7.4 and a  $3.94.10^{-3}$ L/s flow rate with an aluminum anode.

In the best treatment conditions, the COD residual (18 mg  $O_2/L$ ) was less than the permissible level of 100 mg  $O_2/L$ . The operational cost of EC was 0.0761US\$ per kg of COD. Overall, the results confirm the efficiency of EC process, powered directly by a photovoltaic module, in removing pollution from oil wastewater.

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