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ELECTROCOAGULATION OF OIL/WATER EMULSION IN A CELL WITH VERTICAL ROTATING ALUMINUM CYLINDER ELECTRODE

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ABSTRACT

Large volumes of oily wastewater are daily produced. Nowadays, environmental wastewater treatment methods attracted more attention. As a result, many treatment methods are concerned with oily wastewater. Electrocoagulation (EC) technology using aluminum as a sacrificial electrode has been widely applied to separate emulsified oil.EC was investigated as an efficient and financial strategy for the treatment of such wastewater by studying the impact of current density and pH on the 500 mg / l oil-in - water emulsion. The used cell reactor consists of a vertical rotating aluminum cylinder electrode as an anode and a fixed cylindrical aluminum tube as a cathode.

The impact of few factors on the reactor performance was considered, such as the impact of the speed of tumult, the separate between the terminals and the concentration of salt. In the event that the current density rises and the hole between the anodes diminishes, the lessening of turbidity rises. The increase in the speed of electrode agitation (rpm), decreases the diffusion layer and decreases the elimination of turbidity; decreasing the NaCl content of the aqueous process induces an increase in the degree of turbidity elimination. The ideal conditions of this study about have appeared that the highest separation efficiency (92 percent) was accomplished after 15 min of electrolysis at an introductory pH of 8, current density of 400 A/m^2, and initial NaCl concentration of 3 g/l.

Keywords: *Electrocoagulation, Water pollution, Turbidity removal, cylindrical electrodes.*

1. INTRODUCTION

Chemical plants produce polluted water; this involves oil refineries that are marked by high concentrations of aliphatic and aromatic petroleum hydrocarbons that have toxic and adverse effects on plant and marine life in addition to surface and ground water supplies. [1]Several oily wastewater treatment approaches have been used, such as chemical destabilization using dissolved inorganic Salts[2], flocculation[3], liquid airflotation[4], membrane processes[5, 6],electro-flotation or electro-coagulation[7, 8], biological processes[9-10] and adsorption[11].

The electrocoagulation process is conducted by means of electrodes. The electrode material is selected on the premise of some parameters, such as, oxidation potential. In spite of the fact that there are distinctive materials, such as steel and aluminum are the foremost compelling and competitive terminal materials to produce $Al(aq)^{3+}$ or $Fe(aq)^{3+}$ ions. Aluminum has

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been used in the current study, cause of to its cost-effectiveness, ready availability and demands comparatively low oxidation potential. [12]

The anode material undergoes oxidation while the cathode is subject to the degradation of elemental metal.

At the anode:

$$M_{(s)} \rightarrow n\, M^{+}_{(aq)} + n\, e^{-} \qquad (1)$$

$$H_2O \rightarrow 4\, H^{+}_{(aq)} + O_{2\,(g)} + 4\, e^{-} \qquad (2)$$

At the cathode:

$$n\, M^{+}_{(aq)} + n\, e^{-} \rightarrow M_{(s)}(3)$$

$$2\, H_2O_{(l)} + 2\, e^{-} \rightarrow H_2 + 2\, OH^{-} \qquad (4)$$

The electrocoagulation cycle is carried out by dissolving Fe / Al ions into water streams which are hydrolyzed into polymeric iron or aluminum hydroxides. These polymeric hydroxides are fabulous as a coagulation specialist. As these metal cations are blended with negative particles transported by electrophoretic movement to the anode, coagulation happens. Oxygen and hydrogen bubbles shaped at the anode and cathode can cause the coagulated particles to drift to the surface of the arrangement and kick off the treated water at the foot of the arrangement. [13] The gasses produced within the terminals increases the productivity of the division handled by electro-flotation. [14]

Electrocoagulation can neutralize the pH of the treated water to some degree by following reactions compared to chemical coagulation.

$$Al \rightarrow Al^{3+} + 3e \qquad (5)$$

Acid gives protons hydroxide to water and hydrated metal particle, so aluminum hydroxide, which produces three particles of OH^{-} per particle of Al, in this way requires three H^{+} ions:

$$Al\,(OH)_{3\,(S)} + 3\, H^{+}_{(aq)} \rightarrow Al^{3+}_{(aq)} + 3\, H_2O \qquad (6)$$

In fluid arrangement, Al^{3+} shapes the complex particle [Al (H2O) 6]3+.

Within the basic solution, OH^{-} is added to the compound to
make a dissolvable and steady poly(hydrox) complex:

$$Al(OH)_{3\,(s)} + OH^{-}_{(aq)} \rightarrow (Al(OH)_4)^{-}_{(aq)} \qquad (7)$$

The dominant route of elimination, sedimentation or floatation of contaminants amid the final arrange is decided by
the connected current. [15, 16]

There have been many applications for electrocoagulation within the treatment and decontamination of wastewater for a long time [17, 18]. Electrocoagulation is an excellent method for extracting suspended solids, tar, greases [14], dyes [19-20], heavy metals [21, 21], and Strong agricultural products [23]. A few of the points of interest of the electrocoagulation prepare are required for straightforward hardware. [24] Which does not require any expansion of chemicals and no era of auxiliary toxins. [25]

In the present research, the analysis of the applied current density effect on the efficiency of the EC system with regard to the removal of turbidity from oil / water emulsion was carried out. Optimization of factors such as current density (range 100 to 400 A / m^2), concentration of NaCl (range 1 to 4g / l), anode diameter (4, 6 and 7.5 cm), original pH of solution (1, 2, 4, 7,9,10 and 11) and agitation velocity (50. 60, 70, 80. 90, 100.8, 151.2 and 200 rpm) on turbidity removal of solution are taken in consideration.

2. EXPERIMENTAL METHODOLOGY

2.1. Experimental setup:

The experimental apparatus as illustrated in Figure (1):

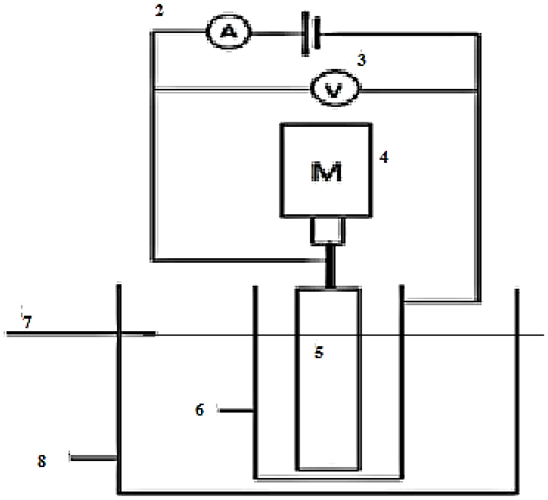

Fig.1. Test plan design and electrical circuit.

1. D.C power supply
2. Ammeter.
3. Voltmeter.
4. Agitator.
5. Al cylinder (anode) (height = 20 cm fixed to the stirrer).
6. Al cylinder (cathode) (Closed bottom cylinder, its back is isolated with epoxy).
7. Emulsion level
8. Plastic container (with diameter = 15 cm and height = 15 cm)

2.2. Materials used

Materials used are crude oil, distilled water, NaCl (AR) and S_1 emulsifier (poly ethylene + additive + ethylene oxide + nonionic phenol+ salicylic acid).

2.3. Variables investigated

Factors such as current density (ranged from 100 to 400 A/m^2), NaCl concentration (from 1 to 3g/l), Anode diameter (4, 6 and 7.5 cm), initial pH of solution (1, 2, 4, 7,9,10 and 11) and agitation velocity (50. 60, 70, 80. 90, 100.8, 151.2 and 200 rpm) are considered.

2.4. Procedure

2.4.1. Emulsion Preparation

The demulsification of oil-water emulsion by electro-coagulation requires the arrangement of a stable manufactured emulsion to recover the genuine oil emulsions shown in here. Consequently, to get ready oil-in-water emulsion with 500 mg/l oil concentration, the following steps were carried out:

1- Five grams of crude oil and 6 ml of S_1 emulsifier [provided by The Egyptian Starch and Yeast Co. (Alexandria – Egypt)] mixed to 1000 ml with the required amount of tap water in 1 L measuring flask to prepare a stock solution of 5000 mg/l concentration.

2- A volume of 50 ml was taken from the stock solution completed to 1000 ml tap water to prepare initial concentration of the experiment (250 mg/l).

3- The solution was stirred vigorously with a mechanical stirrer.

4- The pH esteem was balanced at the specified value by utilizing hydrochloric acid (HCl) or caustic soda (NaOH), before starting the run.

2.4.2. Sampling

The process was carried out in a reactor with Aluminum cylinder (anode) (height = 20 cm fixed to the stirrer) and Aluminum cylinder (cathode) (Closed bottom cylinder).

Samples were collected every 10 minutes; each time the tests were taken from the same cell area. The duration of a single run was 50 minutes, and each run consisted of five samples. Analyze the samples using a spectrophotometer to analyze their oil concentration.

In the second step, after each cycle, the contaminants were removed. Aluminum hydroxide flocs, $Al(OH)_3$, are known to be suitable for the adsorption and precipitation of broken down contaminants, though little hydrogen bubbles streaming around the cathode are mindful for the buoyancy of poisons. [26, 12].

2.4.3. Oil analysis

The concentration of the emulsified oil was determined using spectrophotometer (Helois Alpha, NC 9423 UV A1002E). The suitable wavelength which gives maximum absorbance and minimum interference was determined.

2.4.4. Data treatment

By the end of each run, electrodes must be cleaned by tap water and scrubbed to remove any oxide film formed during experiment,The low ionic conductivity reduces the anodic dissolution. Calculate the percentage removal using the following formula:

$$\%\, Removal = \frac{C_o - C_t}{C_o} * 100 \qquad (8)$$

Where: C_o: initial crude oil concentration (mg/l).

$C_{t:}$ concentration of crude oil at time t (mg/l)

3. RESULTS AND DISCUSSION

The percent of oil removal from emulsion was studied using different variable parameters; current density, time, agitation velocity, initial pH, and salt concentration.

3.1. Effect of current density:

By and large, current density is the foremost basic parameter to control the response rate. Four current densities of 100, 200, 300 and 400 A /m^2 were checked. Figure 2 shows that when the current thickness increases, the effectiveness of the yield of Al^{3+} in anode andcathode increases concurring to Faraday's law. This leads to an increment within the generation of herds. As higher sums of aluminum broken down, higher coagulation effectiveness and more prominent destabilization of the emulsion are accomplished. The higher the streams, the higher the hydrogen generation rate, favors the buoyancy of flocculated matter. [27, 28]

The number of bubbles delivered amid electrocoagulation increments as the current thickness increments, permitting the suspended solids to drift to the surface of the reactor. Bubbles formed in large numbers that interfere with the removal of turbidity due to a decrease in the collision between oil droplets and a decrease in the size of the floc. Slowly, tiny bubbles have been raised more thanbig ones and can be added to neutralized oil drops. [29]

Also, the percent removal of turbidity increased at higher current density due to a rise in anodic aluminum dissolution, resulting in further precipitation of contaminants. There was an increase in the production of bubbles and a decrease in the size of the bubble as the current density increased. [30]

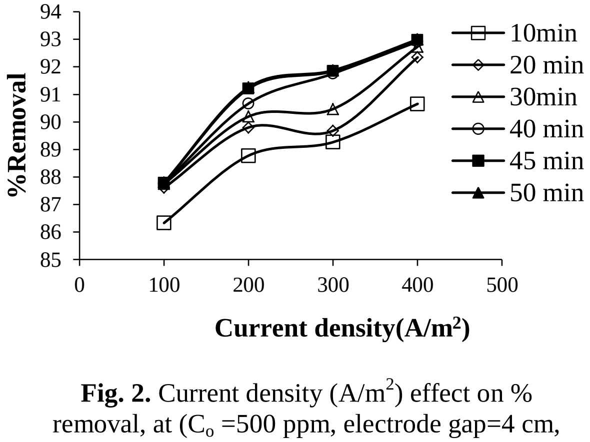

Fig. 2. Current density (A/m^2) effect on % removal, at (C_o =500 ppm, electrode gap=4 cm, NaCl conc. =1 g/l and v=100.8 rpm).

3.2. Effect of initial pH:

After disintegration, the Al^{3+} (aq) particles experience numerous responses to form different monomeric and polymeric compounds, such as Al $(OH)_2^+$ and $Al13O4(OH)_{24}^{7\ +}$, which in effect easily coagulate flocs-forming contaminants.Aluminum speciation (hydrolyzation of dissolved ions) is, in general, profoundly directed by the pH of the arrangement being treated where $Al(OH)_2^+$ is the transcendent species of aluminum for the pH extend (5–6), $Al(OH)_4^-$ is the overwhelming species for the pH extend over 9, whereas $Al(OH)_3$ is overwhelming for the unbiased or marginally soluble pH run. [31]

Figure 3 indicates that the amount of removal rises to pH 8 and then begins to decrease. The data shown in Table (1) also indicates that the initial pH effect on the productivity of the EC cycle [32, 33, 34] as well. Neutralization between the Al^+ ions and the Al^-ions was responsible for the stabilization of the oil emulsion between the oil drops and the water. [35, 36]

Table 1: Effect of the medium pH on the forms of AI ion species

pH	Hydrolysis product
< 4	$Al(H_2O)_6^{3+}$; $[Al(H_2O)_5(OH)]^{2+}$; $[Al(H_2O)_4(OH)_2]^+$
4-5	$[Al_6(OH)_{15}]^{3+}$; $[Al_8(OH)_{20}]^{4+}$
5.5	$Al[(H_2O)_3(OH)_3]^0$
> 7	$[Al_2(OH)_7]^-$; $[Al(OH)_4]^-$

In arrange to decrease the interfacial pressure and to debilitate the interfacial film quality, ingested surfactant can be supplanted by demulsified atoms adsorbed on the oil bead surface to play down its soundness. Moreover, the demulsifier neutralizes the negative charge on the surface of the oil beads, decreasing the electrostatic shock between them, which empowers the accumulation of oil droplets. Upon demulsification of oil beads by charge neutralization, the flocculent adsorption bridging and clearing work together in such a way that the oil beads shape totals, at that point the engineered activity of the demulsifier and the flocculent permits the oil bead film to crumble and the oil droplet to coalesce in arrange to attain the objective. [37, 38]

When the pH of the original water is greater than 9, the pH of the treated water decreases. Therefore, the pH of the chemical coagulation cycle must be modified as the pH of the solution decreases with the addition of the coagulant.

Under acid conditions, and with the evolution of H_2, O_2, and CO_2 can be purged. Particularly, Al dissolution as shown by the previous equations.

And, the Al $(OH)_3$ shaped, also dissolved, and easily appears to the left. These reactions are responsible for the increase in pH of the solution. [39, 40]

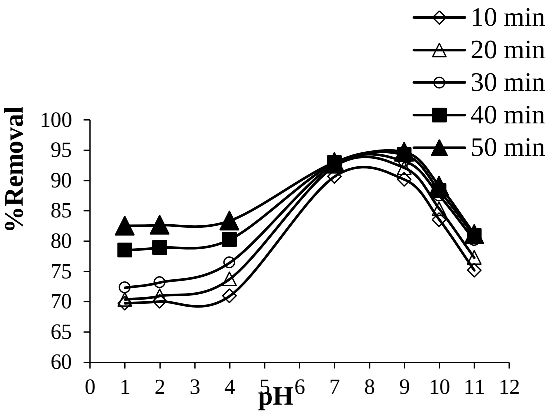

Fig.3. pH effect on %Removal at (C_o =500 ppm, electrode gap= 4 cm, *NaCl* conc. =1g/l ,v=100.8 rpm, i=400 A/m^2).

3.3. Effect of electrolysis time:

This research is used to measure percent removal of turbidity of the oil /water solution. The turbidity of solution changes with time of electrocoagulation process as shown in Figure 4. The figure indicates that the percent of turbidity removal directly proportional with the time. When the electrocoagulation cycle time increases, the water turbidity decreases and the rate of output of hydroxyl and metal ion on the electrode increases. As shown in the figure the electrocoagulation process, passing through two steps: destabilization and aggregation. [41]

The fast step is the first one, and the second step is relatively slow. In the first step within first 15 min the percent removal increases. Pursuant to Faraday's law: the amount of electricity applied to the cell decreases as the time of current passage decreases, which allows the anode release of aluminum ions. The

Positively charged ions neutralize negatively charged oil droplets.

This impact increments the division of neutralized oil beads and makes them coast on the water.

As the treatment time increases, at the cathode there will be a part of hydrogen gas bubbles. And by the activity of bouncy constrain, the hydrogen bubbles will be pushed upward with the oil drops which leads to increase the efficiency of turbidity removal of the emulsion.

The droplets decrease in the second step after 15 minutes and this cause the oil separation to slow down with time. [31]

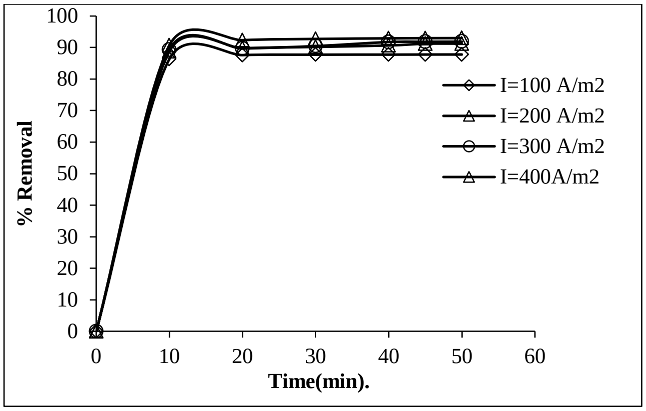

Fig. 4. Time and current density effect on %removal (C_o =500 ppm, electrodes gap = 4 cm, NaCl conc. =1g/l , v = 100.8 rpm).

3.4. Effect of electrode agitation speed:

Electrocoagulation was conducted at different stirring speeds of 50, 70, 80, 90, 100, 151 and 200 rpm to test the effect of agitation level. The effect of electrode agitation on percent removal of turbidity shown in Figure 5. The findings show that the reduction of turbidity by coagulant aluminum hydroxide $Al(OH)_3$ happened exceptionally successfully at a blending level of 50 to 90 rpm. Therefore, for agitation speed of 150 to 200 rpm a slight decrease occurs in percent removal of turbidity. This may be due to the excessive agitation that breaks the flocs and the reduction in diffusion layer. [42]

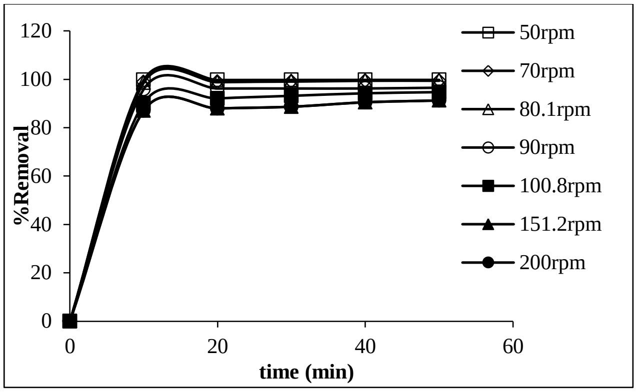

Fig.5. Agitation velocity and EC time effect on %Removal (C_o=500ppm, electrodes gap=4cm, NaCl conc. =1 g/l, i=400A/m^2, pH=9).

3.5. Effect of electrode gap:

The electrode gap is theInter- electrode distance which also affects the current supplied. Larger inter electrode distance generates more resistance, which decreases the current density.

Figure 6 shows that decreasing the spacing between the electrodes causes a slight improvement in the turbidity removal. And also, the decrease in electrode gap decreases of electrolyte resistance. [43]

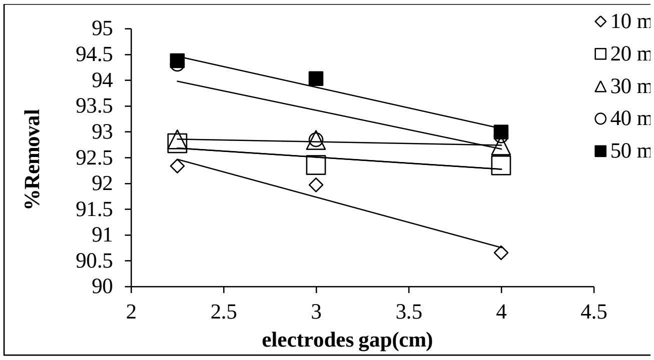

Fig.6. Electrode gap effect on %Removal with time, (C_o =500ppm, v=100.8 rpm, i=400A/m^2, pH=7, NaClconc=1 g/l).

3.6. Effect of NaCl conc. (conductivity) on removal efficiency:

Different concentrations of NaCl (from 1 to 3g / l) have been used. Figure 7 shows that the electrolyte concentration has a vital impact on the removal capacity of the turbidity of the solution. Increased electrolyte concentration achieved better turbidity reduction.

The arrangement of the anodes, alongside components such as the concentration of electrolytes, plays a noteworthy part within the partition effectiveness of oil from wastewater. In order to provide an effective electrocoagulation feature in the treatment of water, it is proposed that 20% of the anion must be Cl$^-$.

Typically, NaCl is used to improve electrolytic conductivity. The presence of NaCl has two main functions which are: first, it increases the conductivity of the electrolyte; second, it increases the conductivity of the active chloride ions during electrolysis, which contributes to the sanitization of the water additionally kills the arrangement of a detached layer on the anode and makes strides current productivity.[44, 45]

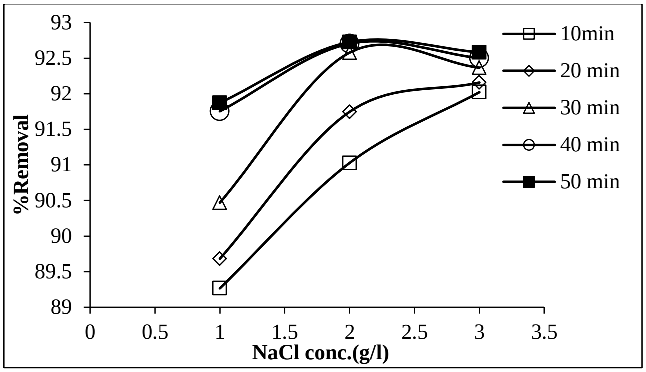

Fig.7. NaCl conc. effect on %Removal at (C_o=500ppm, v=100.8 rpm, i=300A/m^2, g=4cm, pH=7).

4. CONCLUSIONS

It was shown that working parameters, such as current density,pH arrangement and response time, have a critical impact on the response of turbidity removal.

- Current density increments, the proficiency of the yield of Al^{3+} in anode and cathode increments concurring to Faraday's law and contributes to an advancement within the disposal of turbidity.
- The percentage of turbidity removal increases with pH until it reaches pH 8 and then begins to decrease.
- The electrocoagulation process, passing through two steps: destabilization and aggregation. The fast step is the first, and the second step is relatively slow.
- As the electrocoagulation process time increases the oil/water emulsion turbidity decreases.
- The reduction of turbidity by coagulant aluminum hydroxide Al(OH)$_3$ occurred very effectively at a stirring level of 50 to 90 rpm. As a result, there is a slight increase in agitation speed from 150 to

200 rpm when the turbidity is reduced by percent.
- Decreasing the spacing between the electrodes causes a slight improvement in the turbidity removal.
- Increasing the electrolyte concentration achieves better percent removal of turbidity.

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Appendix

Specifications of Emulsifier used

The S_1 emulsifier is a brown liquid. Nonionic surfactant resulting from esterification of oleic acid ($C_{17}H_{33}COOH$) with carbowax200 in the presence sulfuric acid as a catalyst to give oleicperol that has scientific name divinyl phenyl polyperoxy ethylene and ethoxy vinyl phenol. It forms a stable emulsion of oil with water. It is used as emulsifier for some mineral oils.