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SEPARATION OF OIL FROM O/W EMULSION BY ELECTROFLOTATION TECHNIQUE

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ABSRACT

Dilute emulsified oil in water (50-500 ppm) was removed by electrocoagulatin and electroflotation process. Effects of various parameters such as current density, initial pH, sodium chloride concentration, different electrodes material, gap, temperature, electrodes surface area, and treatment time on the removal efficiency were studied in batch and continuous mode.It was found that the higher removal efficiency (99%) achieved at pH of 7.5 for Al/Al electrodes while for iron electrodes at pH 8 in the batch mode.

The removal efficiency increased and the treatment time decreased with increasing the current density and decreasing the gap distance between electrodes.

The removal efficiency was (99%) at 10 min for Al/Al electrodes, (98.6%) at 12 min for Al/St.St. electrodes, and (97.4%) at 12 min for Fe/St.St. electrodes.

The present results showed that the best temperature was 60ºC .The best concentration of sodium chloride was found to be (400 ppm) when the oil concentration was (500 ppm). Also it was found that a vertical Al/Al electrode was the best type with an electrical energy consuming (3.36 kWh/m^3).

In the continuous experiments, the removal efficiency enhanced with increasing flow rate. A multiple linear regression model was used in order to relate experimental data to a statistical model with a correlation coefficient of (0.909) and variance (0.827).

KEYWORDS: emulsion, electroflotation, separation

الخلاصة

. الزيت المستحلب في الماء (ppm 500-50) فصل بطريقة التطويف الكهربائي . تم دراسة تأثير عدة متغيرات مثل كثافة التيار,الدالة الحامضية ,تركيز كلوريد الصوديوم,نوعية الاقطاب ,درجة الحرارة,المساحة السطحية ,المسافة بين الاقطاب ,وزمن المعالجة على كفاءة ازلة الزيت وبأسلوب الوجبة. ومن التجارب وجد ان اعلى كفاءة (99%) وبدالة حامضية (pH=7.5) لاقطاب Al/Al اما بالنسبة لاقطاب Fe/St.St عندما تكون الدالة الحامضية هي (pH=8).

وقد تبين ان كفاءة الازالة تزداد وزمن المعاجة يقل بزيادة كثافة التيار الكهربائي في حين عند زيادة المسافة بين الاقطاب يؤدي الى نقصان كفاءة الازلة مع زيادة زمن المعالجة. ومن خلال النتائج المختبرية الكفاءة المستحصلة (99%) بزمن (10 min) لاقطاب Al/Al وكفاءة (98.6%) وزمن (12 min) بالنسبة لاقطاب .Al/St.St واما اقطاب .Fe/St.St تكون الكفاءة (97.4%) وزمن (12 min).

أفضل درجة حرارة هي (60 ºC) وأفضل تركيز لملح كلوريد الصوديوم هو(400 ppm) .ونستنتج من التجارب المختبرية ان اقطاب Al/Al العمودية هي الافضل باسنهلاك للطاقة الكهربائية بمقدار (3.36 kWh/m^3).

وجد ان كفاءة الازالة تتحسن مع زيادة الجريان.من خلال احتساب الانحدار اللاخطي المتعدد للنتائج المختبرية تم ايجاد معادلة تربط كافة المتغيرات المدروسة وبمعامل تصحيح (90.9%) وتباين (82.7%).

INTRODUCTION

Oily wastewater has remained problematic in various industries four decades. The major industrial sources of oily wastewater include petroleum refining, metals manufacturing, and food processing. This wastewater contains lubricating oil, grinding oils, cutting oils, and coolant oil–water emulsions with a possible soluble and emulsified oil content that varies from 100 to 5000 mg/l (Yang, 2006).

Types of oil-water mixture may be classified as oil and grease present as free oil, dispersed oil, emulsified oil or dissolved oil. Free oil is usually characterized by an oil-water mixture with droplets greater than or equal to 150 microns in size while a dispersed oil mixture has a droplet size range between 20 and 150 microns, and an emulsified oil mixture have droplet sizes smaller than 20 microns. Wastewater with an oil-water mixture where the oil is said to be soluble is a liquid where oil is not present in the form of droplets (the oil particle size would be typically less than 5 microns) (Rhee et al., 1985).

SOURCES OF OIL AND GREASE IN OILY WASTEWATER

Petroleum refining and oil re-refining, used oil and re-refining operation, from fractions oil and primary distillation through final treatment, contains various oils and organosulfur compounds in their wastewaters. Crude oil producing facilities emulsified of wastewater from oil field operations may contain drilling muds, brine, free and emulsified oil, tank-bottom sludge and natural gas. Many oil-bearing beds have brine-bearing formations. Oil and gas must then be separated from the wastewater; this wastewater is typically a brine waste containing some oil contamination and must be disposed (Rhee et al., 1985).

CONVENTIONAL METHODS OF TREATING OILY WASTEWATER

The treatment of oily wastewater has been addressed by different techniques; the most commonly used are the physical methods include heating, centrifugation, precoat filtration, ultrafiltration, and membrane process. ultrafiltration and membrane processes have been actively pursued (Um et al., 2001; Gryta et al., 2001).

The chemical destabilization (conventional coagulation) is used directed toward the destabilization of the dispersed oil droplets or the destruction of emulsifying agents present in a first stage followed by the removal of the separated oil. This treatment accomplished by addition of hydrolyzing metal salts (Fe^{3+} or Al^{3+}) as coagulant reagents, which are still the most widely used reagents for demulsification. The process usually consists of rapid mixing of the coagulant with the wastewater followed by flocculation and flotation or settling.

Currently the treatment of oily wastewater applies a primary treatment to separate the floatable oils from the water and emulsified oils. A secondary treatment phase is then required to break the oil–water emulsion and separate the remaining oil from the water. Primary treatment takes advantage of the differences in the specific gravities of the oils and grease and the water. Then subsequently skimmed oils and grease floated from the wastewater surface.

Dissolved-air flotation is another process for the supplemental treatment of oil-water separator effluents for reducing oil and suspended solids to low levels. Its success depends on the use of very fine air bubbles to increase the rate of suspended particles so they can be floated to the surface for

removal. It is, in effect, the opposite of sedimentation. Dissolved-air flotation can be used alone or in combination with flocculation (flocculation-flotation) (Canizares et al., 2007).

ELECTROCHEMICAL TECHNOLOGIES FOR WASTEWATER TREATMENT

Electrochemical technologies have recuperated their importance in the world during the past two decades. There are different companies supplying facilities for metal recoveries, treating drinking water or process water, treating various wastewaters resulting from tannery, electroplating, dairy, textile processing, oil and oil-in-water emulsion, etc. At the present time, electrochemical technologies have reached such a state that they are not only comparable with other technologies in terms of cost, but also more efficient and compact.

The development of design and application of electrochemical technologies in water and wastewater treatment have been focused particularly to some technologies such as electroflotation, electrocoagulation, and electrooxidation.

This new rise of electrochemical has also been due to the relative reduction in the operation and investment costs. The electrochemical has the potential to be competitive with respect to both economical and environmental criteria for treatment of wastewater and other related water management issues (Saur et al., 1996; Hosny, 1992; Mollah et al., 2001; Chen et al., 2002).

EXPERIMENTAL WORK AND PROCEDURE

This paper contains description of apparatus, materials and analysis measurement methods of oil emulsion which were used in the test of experimental work.

The experimental work was performed in two parts (batchwise and continuous) for the treatment of emulsified oil. The effect of oil concentration, electrode type, gape, pH, NaCl concentration, applied potential, surface area and treatment time were studied.

EQUIPMENTS AND APPARATUS

The following measuring devices were used in experiments

- Digital pH meter, model orion 3star, thermo electron, USA.
- Combo meter, model combo (Conductivity/TDS/Temp.), HM Digital, Korea, with a range (0-9990 µS/cm), Temp. =0-80 ºC.
- Power supply, Iraqi model VS/CS 25A, 35 V, Iraq.
- Blender mixer, AL-Arabi blender MX-5200, Egypt.
- Ultraviolet spectrophotometer (model GENESYS^{TM} 10 spectrophotometers; thermo), USA.
- Dosing pump ,H.R. flow inducer ,Watson –Marlow limited ,England
- Electrical balance, Sartorius, digital indicator with capacity (210gm).
- Beaker, pipettes of various size.
- Electrical heater, power= 100 w, china.

O/W EMULSIONS PREPARATION

The oily phase of the emulsion is prepared from using a common lubricant oil (Babel-40 provided by AL-Doura refinery, Iraq), and an emulsifying agent (R.T.K. provided by BRB CO., UK), 0.5 gm oil and 0.12 gm emulsifying agent mixed together in separate beakers, to prepared 500 ppm of emulsified oil then stirred until a homogenous liquid was obtained. The salt content of the emulsion was adjusted by adding a suitable amount of sodium chloride solution which prepared separately by dissolving 0.2 gm of NaCl in 1 liter of distilled water. Conductivity of Sodium

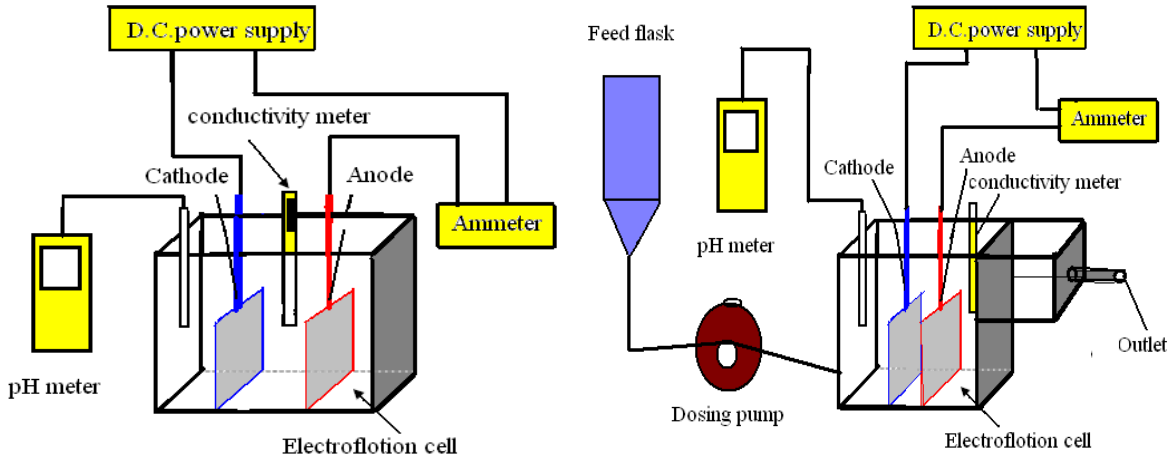
chloride (NaCl) solution was measured by combo meter to achieve a conductivity of (up to 452 µS/cm). Then the contents of the two beakers were mixed and stirred by using blender mixer to obtain the oil emulsion desired.

BATCH EXPERIMENTS

The experiments were carried out in batch laboratory electroflotation cell (Fig.1-a) a glass cell of internal size 12 cm × 12 cm × 30 cm (width × length ×height) with an effective volume of 4L. It is provided with two electrodes: aluminum or iron as anode and stainless steel as cathode. In the electroflotation cell configuration, the monopolar aluminum or iron plate electrodes were used. The spacing between electrodes varies from 1-4 cm. whereas, the oil emulsion batch 3L for each experiment we used. Electrodes were placed in the oil emulsion and connected to terminals of a DC Power Supply. The voltage and current flowing through the cell were measured with a multimeter. The emulsion was maintained at the desired temperature by portable immersed heater. Samples were taken from the cell using a pipette tube to avoid passivation of the electrodes, the electrochemical cell and electrodes were entirely cleaned after each experiment with detergent and ethanol. All experiments were carried out at room temperature near 30ºC. pH of the emulsion was adjusted at the desired value by adding 1M (NaOH or H_2SO_4) . pH meter was used to measure the pH value. While conductivity of emulsion was measure using Combo meter. The same procedure applied in batch mode was applied in continuous experiments.

CONTINUOUS EXPERIMENTS

In continuous experiments, the electroflotation cells with a total volume of 3.5 liters are shown in (Fig.1-b). It is divided into two compartments. The first size of compartment is (9cm×12 cm × 30 cm) which provided with two electrodes. This compartment receives the influent from a hold conical flask by a dosing pump. The size second compartment is (9 cm× 12 cm × 9 cm) which received the effluent and undergoes to settling of the suspended solids .The bubbles formed at electrodes and effluent are in co-current movement. Electrodes were connected to terminals of a DC Power Supply. Multimeter was used to measure the current passing through the circuit and the applied potential, respectively. The pH values in the cell were measured using a pH meter and adjusting pH of emulsion by 1M (NaOH or H2SO4).while, the conductivity of emulsion was measured using Combo meter.



(a) **(b)**

Fig.(1): Schematic diagram of batch and continuous experimental.

(a) batch mode, (b) continuous mode

RESULTS AND DISCUSSION

This research is mainly focused on the electrocoagulatin treatment of emulsified oil. The effects of: initial pH, initial oil concentration, temperature, NaCl concentration, electrodes type, surface area, applied potential and treatment time value were studied to evaluate the EC progress. The oil removal efficiency was estimated from experimental tests for all electrodes types. Two parts were used during electroflotation treatment:

(i) Batch experiments.

(ii) Continuous experiments.

BATCH EXPERIMENTS

- Effect of applied potential

The variation of removal efficiency and energy consumption with time for different applied potentials are shown in figs.(2 and 3) from these figures it can be seen that both the energy consumption and the removal efficiency increase with increasing applied potential (5V- 35V). This can be explained by the fact that with an increased in the current density, the aluminum production on both anode and cathode increased resulted in an increased in the floc production in the solution .in addition bubbles density increases and their size decreases resulting in both greater upward momentum flux and increased mixing. Therefore, the highest removal efficiency 98.2% with lowest electrolysis time (28 min) at applied potential 35V by using horizontal (A/Al) electrodes was achieved.

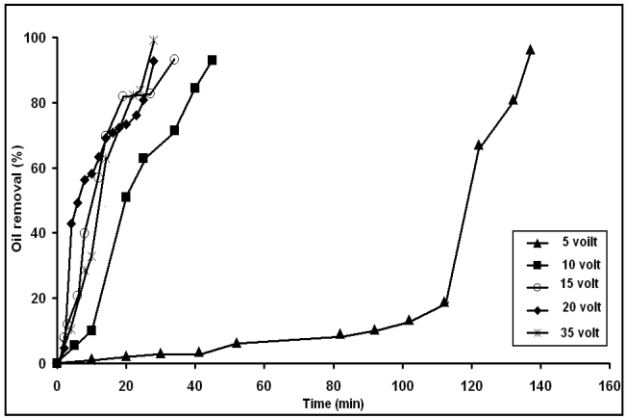


Fig.(2)Variation of oil removal efficiency with different applied potential (Horizontal Al/Al electrodes, [oil] =500 ppm, [NaCl] =200 ppm, gap=1 cm,30ºC, AAA=182.47cm^2).

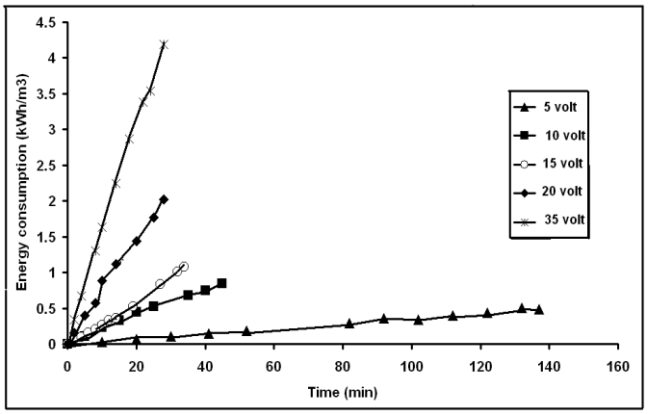


Fig.(3)Variation of energy consumption with different applied potential (Horizontal Al/A electrodes, [oil] =500 ppm, [NaCl] =200 ppm, gap=1 cm,30ºC, AAA=182.47cm^2).

- Effect of initial pH

pH is an important parameter in chemical and electrochemical coagulation. The effect of pH on the removal efficiency is plotted in fig.(4). Adjusting the pH in the range (3.5-8.5) using 1M (NaOH or H_2SO_4) and applying 35V, this figure shows the removal efficiency as a function of treatment time at different initial oil emulsion pH values. The higher oil removal efficiency (98.2-98.3%) attained in the pH range of (3.5–8.5). The pH effect in the removal efficiency is very significant in pH 7.5 (98.2%) with electrolysis time 28 min and energy consumption of 4.19 kW/m^3. While for the other values of pH the time required to achieve the same removal efficiency increased in the

range (46-62 min) in the acidic medium and about (40 min) at pH 8.5. With increasing the treatment time (28-62 min) the electrical energy consumption increased from (0.93 -8.19 kWh/m^3), this is shown in fig.(5) by plotting energy consumption against time at different initial pH. It is that increasing pH from acidic to neutral values lead to stands for the whole oil droplets/precipitate particles system. This behavior can be explained in terms of the charge reversal of the aluminum hydroxide precipitates from positive to negative, for pH values over 8. These negative charges produce repulsion forces between the oil droplets (negatively charged) and the particles of precipitate, and consequently avoid the attachment of more than one droplet to a particle of precipitate, and the subsequent coalescence of the droplets, causing therefore increase time of oil removal efficiency.

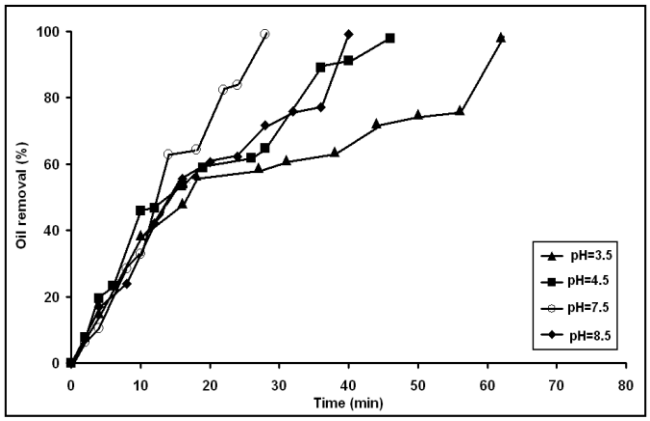


Fig.(4) Variation of oil removal efficiency with different pH (Horizontal Al/Al electrodes, 35 V, [oil] =500 ppm, [NaCl] =200 ppm, gap=1 cm ,30ºC, AAA=182.47 cm^2).

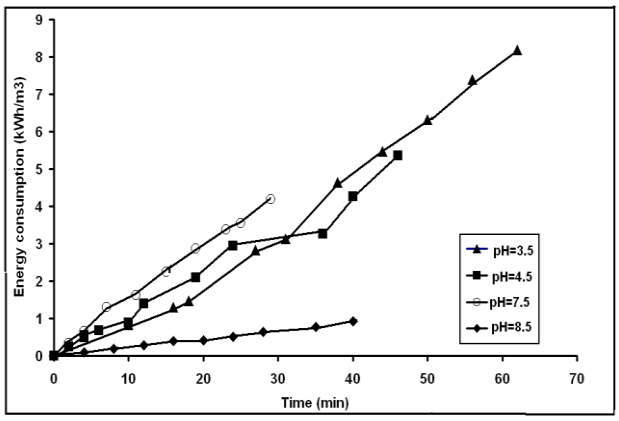


Fig.(5) Variation of energy consumption with different pH (Horizontal Al/Al electrodes, 35 V, [oil] =500 ppm, [NaCl] =200 ppm, gap=1 cm ,30ºC, AAA=182.47 cm^2).

-Effect of initial oil concentration

The removal efficiency of emulsified oil at various initial oil concentration (50-500ppm) with different electrodes direction are shown in figs.(6 and 7).In the case of horizontal (Al/Al)electrodes, figure(4.5) the time required to achieved the higher removal efficiency (98.2%) increased from (16-28 min) with increasing initial oil concentration from (50 -500 ppm).while vertical (Al/Al) electrodes had a better removal efficiency (98.6%) achieved at time 10 min for initial concentration (300 ppm). From the two figures below it can be seen that the electrodes direction effect on time and removal efficiency.

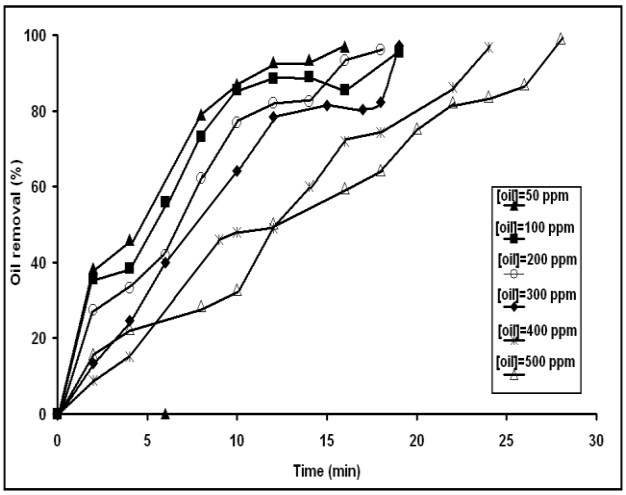


Fig.(6)Variation of oil removal efficiency with different oil concentration (horizontal Al/Al electrodes, 35V, gap=1cm, pH=7.5, 30 ºC, AAA=182.47 cm^2).

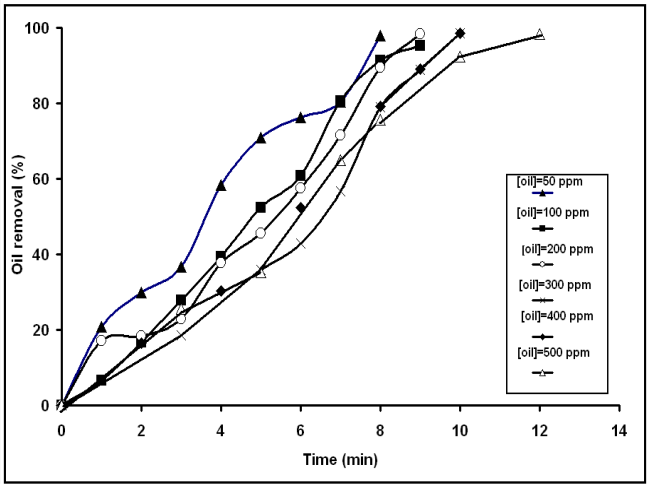


Fig.(7)Variation of oil removal efficiency with different oil concentration (horizontal Al/Al electrodes, 35V, gap=1cm, pH=7.5, 30 ºC, AAA=182.47 cm^2).

-Effect of initial temperature

The effect of the electroflotation cell temperature on the oil removal efficiency is shown in fig.(8).The removal efficiency showed a tendency to increase with an increase in temperature. The higher removal efficiency (98.8%) achieved when the solution temperature 60ºC with treatment time 9 min. An increase in the temperature resulted a decrease in the oil emulsion stability due to an increase in the adsorption rate, because the Van der Waals forces and Brownian motion increase and decrease the viscosity of the continuous phase film. This would contribute to increase coalescence rate, through an increased probability of film rupture (Becher, 1966).

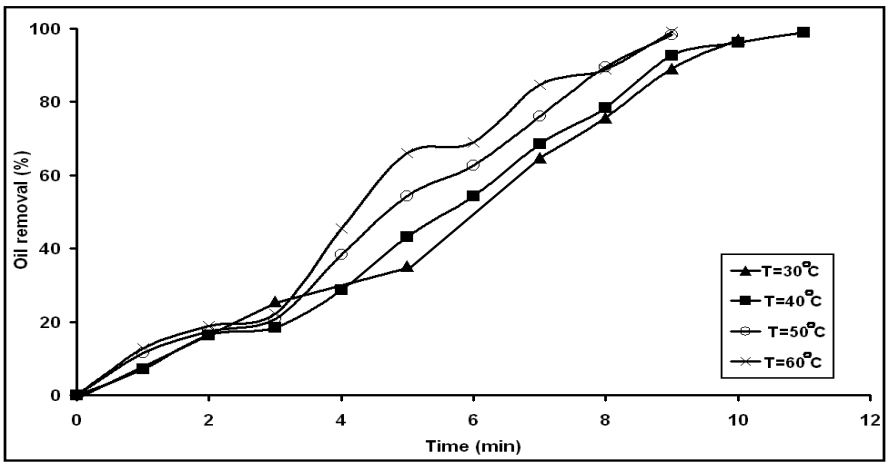


Fig.(8) Variation of oil removal efficiency with different temperature (Horizontal Al/Al electrodes, 35V,[oil] =500 ppm, [NaCl] =200ppm, gap=1 cm, pH=7.5, AAA=182.47 cm^2).

-Effect of sodium chloride concentration

Sodium chloride concentration was varied to evaluate the impact of solution conductivity on electrocoagulation. Figure(9) shows the relationship between sodium chloride concentration and removal efficiency with time. From this figure it can be seen that increasing sodium chloride concentration (50-400 ppm) leads to an increase in the removal efficiency with decreasing in treatment time required to achieve the highest removal percent. When using 50 ppm NaCl, the maximum removal efficiency achieved (98%) at time 13 min. while increasing the NaCl concentration to 400ppm resulted in 98.9% removal efficiency at 7 min. This means that the time decreased as the solution conductivity increases.

The increase in the oil removal efficiency attributed to a change in the ionic strength due to changing conductivity of the emulsion medium. Ionic strength affects on the oil emulsion destabilizing which occurring between charged species and oil droplets during EC.

The relationship between solution conductivity and energy consumptions is plotted in fig.(10). From this figure it can be concluded that the energy consumption increases from 1.99 to 4.12 kWh/m^3 with increasing conductivity.

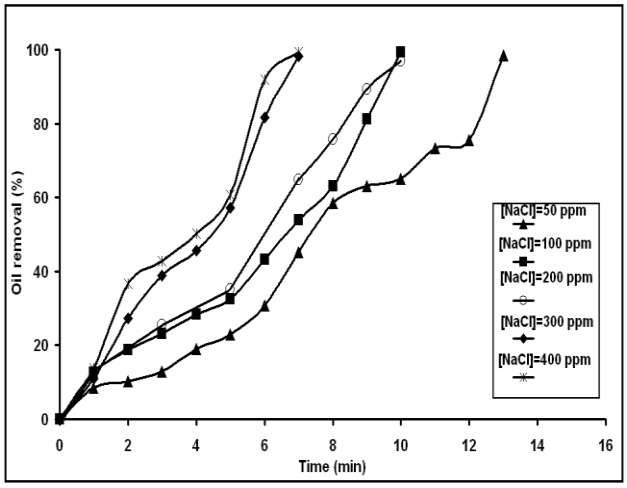


Fig.(9) Variation of oil removal efficiency with different NaCl concentration(vertical Al/Al electrodes, 35V,[oil] =500 ppm,gap=1cm, pH=7.5,60 ºC,AAA=182.47 cm^2).

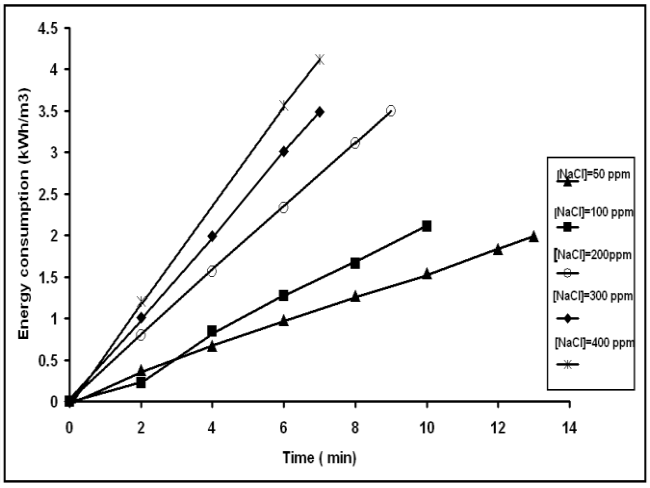


Fig.(10) Variation of energy consumption with different NaCl concentration(vertical Al/Al electrodes, 35V,[oil] =500 ppm,gap=1cm, pH=7.5,60 ºC,AAA=182.47 cm^2).

-Effect of electrodes surface area with different gap

Three electrodes types with two different sizes are used to investigate the effect of electrode size and gap between electrodes on the removal efficiency. The results are shown in fig.(11 and 12) for (Al/Al) electrodes. It can be observed that increasing the active area from (182.217 -273.12 cm^2) enhanced the removal efficiency and reducing the treatment time required.

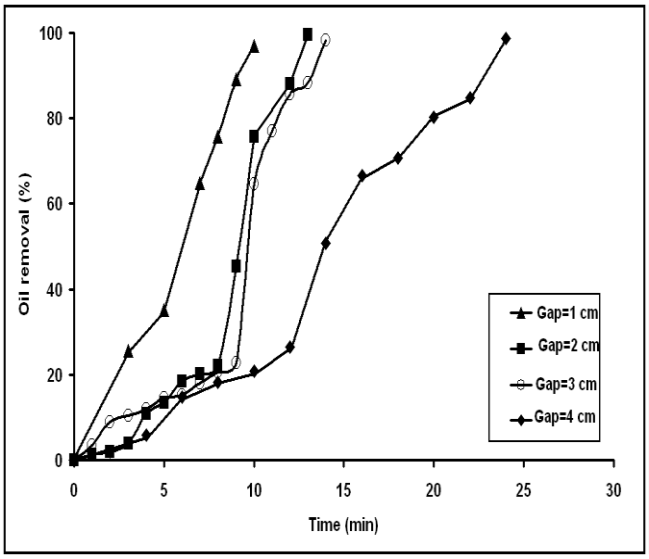


Fig.(11)Variation of oil removal efficiency with gap (Vertical Al/Al electrodes, 35V ,[oil] =500 ppm,[NaCl]=200 ppm,pH=7.5, 30ºC,AAA= 182.47 cm^2).

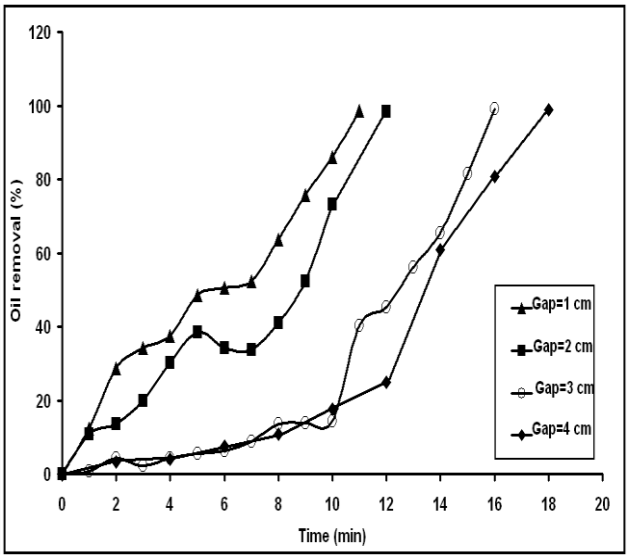


Fig.(12)Variation of oil removal efficiency with gap (Vertical Al/Al electrodes, 35V, [oil] =500 ppm,[NaCl]=200 ppm,pH=7.5, 30ºC,AAA=273.12 cm^2).

-Effect of electrodes material and current density

The influence of different electrodes type and current density on oil removal efficiency are shown in fig(13). The optimum electrode type is the vertical (Al/Al) electrode which achieved 99% removal efficiency in 10 minutes and current density (6.33 mA/cm^2).

This is ascribed to the fact that at Al/Al electrodes under higher applied potential the amount of aluminum oxidized increased, resulting in a greater amount aluminum hydroxide. In addition, it was demonstrated that bubble density increases, and their size decreases with increasing current density for same electrodes type, resulting in a greater upwards flux and a faster removal of oil. As the current density decreased, the time needed to achieve similar efficiencies increased and the results of this research confirm this fact.

The amount of metal released with different electrodes gap is shown in fig.(14),from this figure it can be seen that the amount of metal released depend on the gap and time. Cell with Al/Al electrodes release an amount much than that in the Al/St.St..

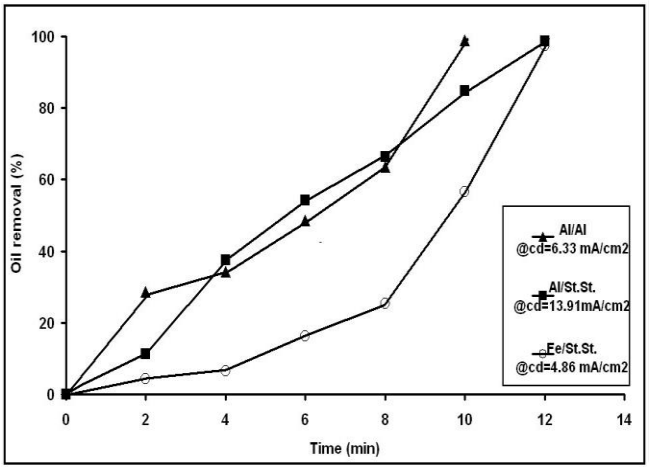


Fig.(13) Variation of oil removal efficiency with electrodes type(Vertical (Al/Al) 35V, 30 ºC, [oil] =500 ppm,gap=1cm,[NaCl]=200ppm,).

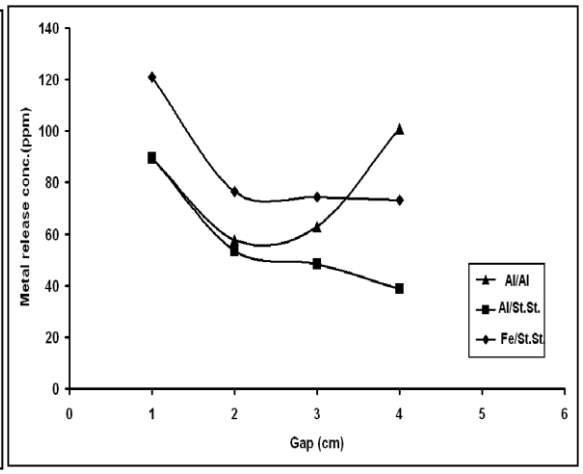


Fig.(14) Variation of oil removal efficiency with gap (Vertical (Al/Al) 35V, 30 ºC, [oil] =500 ppm,gap=1cm,[NaCl]=200ppm,).

CONTINUOUS EXPERIMENTS

Effect of liquid flow rate

The effect of liquid flow rate on the electrocoagulatin efficiency of emulsified oil has been investigated. The relationship between oil removal at different flow rates for (Al/Al), (Al/St.St.), and (Fe/St.St.) electrodes are plotted in fig.(15~17).from those figures it can be seen that the removal efficiency increased with increasing solution flow rate. This result could be explained by the fact that more steady convection allowed by higher flow rates are to improve the rates of transport and transfer phenomena of the various species in the electrochemical cell. In addition, higher flow velocity induce a greater number of collisions between the particle of $Al(OH)_{3(s)}$ and the destabilized oil droplets, thus improving the flocculation.

However, it is observed that the flow rate effect on the oil removal was more significant for the highest values when using (Al/Al) electrodes in (fig.15). This exhibit the effect of: (i) the anode and cathode dissolution, forming Al^{3+} and allowing coagulation, (ii) the transfer phenomena by convective diffusion, and (iii) the flocculation phenomena. However, the influence of the flow rate on the oil removal was more significant at high current densities. From these figures it was found that the highest removal efficiency (99%) after 11 min electrolysis time and 10.58 mA/cm^2 current density is achieved by higher flow rate (0.15 l/min),while for (Al/St.St.) electrodes fig.(16) the highest removal efficiency 98.6% after 16 min electrolysis time and 5.6 mA/cm^2 current density is achieved at higher flow rates(0.15 l/min),and for (Fe/St.St.) electrodes fig.(17) the highest removal efficiency 96.2 % after 26 min electrolysis time and 7.51 mA/cm^2 current density.

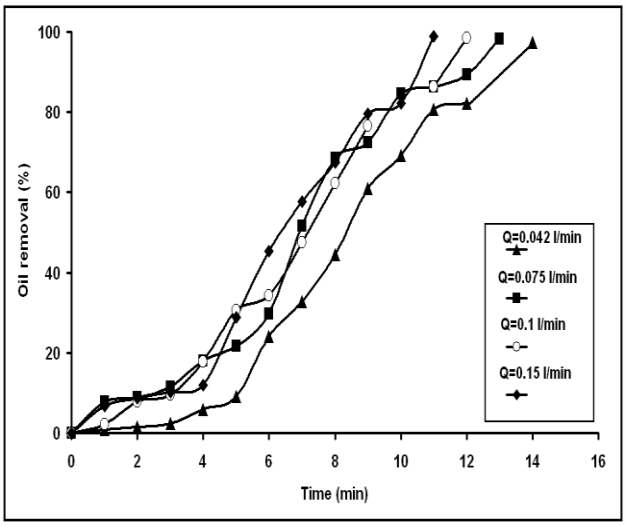


Fig.(15) Variation of oil removal efficiency different flow rate (Vertical Al/Al electrodes, 35V,[oil] =500 ppm,[NaCl]=200ppm, pH=7.5, 30 ºC, AAA=273.12 cm^2).

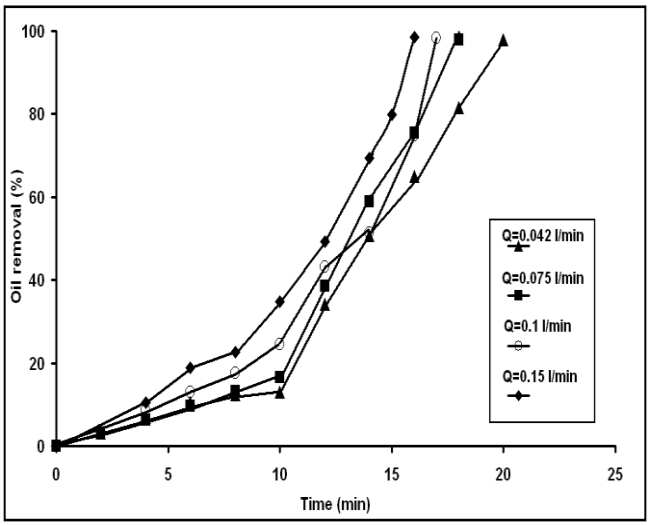


Fig.(15) Variation of oil removal efficiency with different flow rate(Vertical (Al/St.St. electrodes 35V,[oil] =500 ppm,[NaCl]=200ppm, pH=7.5, 30 ºC, AAA=273.12 cm^2).

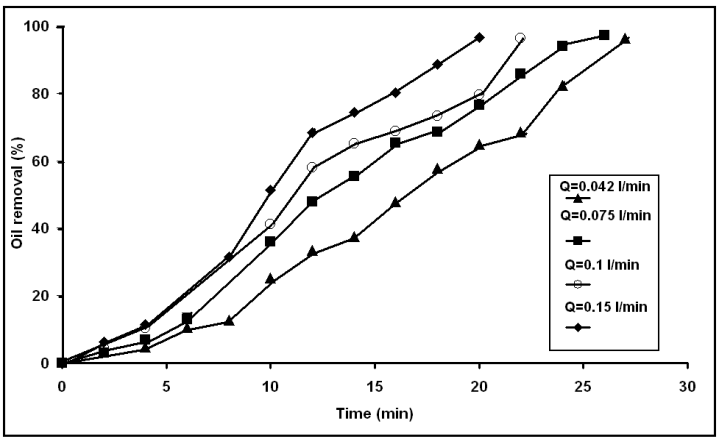


Fig.(17) Variation of oil removal efficiency with different flow rate (Vertical (Fe/St.St.) electrodes, 35V,[oil] =500 ppm,[NaCl]=200ppm, pH=8,30 ºC, AAA =271.44 cm^2).

CONCLUSIONS

The demulsification of oil in water was achieved by electrocoagulatin and the results can be summarized as follows;-

- An aluminum electrode was preferable for electrical demulsification due to higher oil removal efficiency (99%) than iron electrodes which reached a maximum removal efficiency of (97.4%).
- The effects of the current density on oil removal efficiencies, for aluminum and iron electrode materials. The current densities are in favor of both removal efficiencies for both electrode materials between (4.86-6.33 mA/cm^2).the higher efficiencies are obtained (99%) with aluminum and (97.4%) with iron electrodes.

- It is possible to achieved (99%) removal efficiency using a current density of (1.78 mA/cm^2) with an energy consumption of (6.37 kWh/m^3) with (Al/Al) electrodes.
- The optimum temperature was 60ºC for vertical Al/Al electrodes, the removal efficiency reached (98.8%) at 9 min.
- The smaller electrodes gap (1cm) resulted in higher removal efficiency at lowest time due to a higher consumed of the electrode.
- With regard to pH, the treatment time increased with decreasing the pH or increasing it above 7.5. The optimum pH was 7.5 for aluminum electrodes.
- With increasing volumetric flow rate (0.042 - 0.15 l/min) the removal efficiency increased in the range (96.3 - 99%).
- The presence of sodium chloride enhances the oil removal efficiency and
- decrease treatment time.
- The best equation which represents the experimental values is obtained
- with a correlation coefficient of 90.9% and variance 82.7%.

$$R\%=98.05211*X_1^{-0.00561}*X_2^{-0.00182}X_3^{-0.00182}*X_4^{0.00364}*X_5^{-0.00491}*X_6^{0.00247}$$

NOMENCLATURE

R%=percentage of oil removal efficiency.

X_1=pH.

X_2=gap(cm).

X_3=current density (mA/cm2).

X_4= temperature (ºC).

X_5= treatment time (min).

X_6=oil concentration (ppm).

ABBREVIATION

AAA=Active anode area
Al=Aluminum electrodes
Fe=Iron electrodes
St.St.=Stainless steel

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