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Electrocoagulation of cutting oil emulsions using aluminium plate electrodes

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Abstract

The treatment of very concentrated oil–water emulsions by electrocoagulation (EC) was experimentally investigated as a pre-treatment step prior to a membrane process. The oil–water emulsion was prepared from a cutting mineral oil B22 currently used for drilling and machining operations. The electrocoagulation progress was followed by the measurement of COD, turbidity and pH in a batch process with recirculation of the liquid. This study is mainly focused on the effects of operating parameters such as initial pH, current density, oil concentration and recirculation rate, on the de-emulsification efficiency. Kinetic curves showed that the EC process exhibits two phases: a "reactive phase" during which the COD and the turbidity removals increase with electrolysis, and a stationary phase for which further aluminium dissolution is useless in the pollution abatement. The results showed that the treatment efficiency increases with increasing current density, but decreases with oil concentration. It appears that treatment of the considered cutting oil is completed through dissolution of around 10 mg Al/g oil, with a slight positive effect of the liquid flow rate. Best results are also obtained with initial pH near 7.

Keywords: Electrocoagulation; Cutting oil emulsions; Aluminium plate electrodes; Very high COD

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1. Introduction

Cutting oils also called "soluble oils", are used in particular during mechanical operations of cutting and machining metals. They combine the properties of cooling and lubrication. A cutting fluid concentrate usually contains a mineral oil, a surfactant mixture, in some cases water and various additives, which are included to meet the specifications of commercial concentrates such as resistance to bacterial growth and low corrosion capacity [1]. During their use, cutting fluids loose their properties and effectiveness because of their thermal degradation and the production of suspended metal particles. The oils have therefore to be replaced periodically and the organic wastes generated have to be taken away and treated. This constitutes a danger to the environment because these effluents are highly charged in surface-active agents and other organic matters. Moreover, because of their great capacity of penetration in the ground, they constitute a very serious threat for groundwater. Sokovic and mijanovic [2] give an analysis of the ecological parameters of the cutting fluids. The significant developments of the formulations of cutting oils as well as the preparation of the synthetic or semi-synthetic emulsions complicate some more the issue of the purification of these effluents. Because of the stability of these emulsions, there is no universal solution for their treatment, and it can be necessary to combine one or two treatment processes for highly effective purification. Several techniques have been applied to treat these types of oily wastewater, e.g. chemical [3] and biological [4] destabilization, ultrafiltration [5] and nanofiltration [6]. Adsorption on mixed Ca and Mg oxides obtained by thermal decomposition of dolomite was studied to remove exhausted oils [7].

Coagulation makes it possible to destabilize the suspended particles which cannot settle naturally because of their submicrometric size. The destabilization of negatively charged particles

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takes place upon addition of positively charges species, which can be supplied either by addition of chemicals, e.g. iron chloride or aluminium sulphate, for conventional chemical treatment, or by the dissolution of anodes in the case of electrocoagulation (EC). Electrocoagulation is initiated by the oxidation of sacrificial anodes out of aluminium or iron yielding, respectively Al^{3+} and Fe^{2+} ions. The latter ions are rapidly oxidized to Fe^{3+} by air oxidation. The metal ions combine to the hydroxyl ions produced by the water electrolysis at the cathode, to form the corresponding metal hydroxides, which favor the formation of the flocs by destabilisation of the contaminants or particulate suspensions. The flocs formed can be recovered from the liquid surface by scraping – when the bubbles of hydrogen produced at the cathode allow flotation – or settle depending on their density.

The development of EC process has been hindered for years by the high investment costs and severe competition with the chemical processes. It started to regain importance with improvement of electrochemical processes and promulgation of more stringent environmental legislations on wastewater. This new rise of electrocoagulation has also been due to the relative reduction in the operation and investment costs. EC has the potential to be competitive with respect to both economical and environmental criteria for treatment of wastewater and other related water management issues [8]. This technique has been applied for treatment of waters containing suspended solids [9], oils and greases [10–13], dyes and textile wastewaters [14,15], or industrial wastes containing heavy metals [16] and phosphate [17]. EC was also applied for defluoridation of water [18] and urban wastewaters [19].

The electrochemical reaction occurring at the anode and involving metal M-aluminium in the present case, is written as

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

Hydrogen evolution occurs at the cathode depending on pH.

$$2H_2O_{(l)} + 2e^- \rightarrow H_{2(g)} + 2OH_{(aq)}{}^- \qquad (2)$$

$$2H_{(aq)}{}^+ + 2e^- \rightarrow H_{2(g)} \qquad (3)$$

The generated $Al_{(aq)}{}^{3+}$ ions combine with water and hydroxyl ions to form corresponding hydroxydes and/or polyhydroxides [20–22] as follow:

- monomeric species such as $Al(OH)^{2+}$, $Al(OH)_2{}^+$, and $Al(OH)_4{}^-$ by Eqs. (4), (5) and (7),
- polymeric species such as $Al_2(OH)_2{}^{4+}$ and $Al_2(OH)_5{}^+$,
- amorphous and less soluble species such as Al $(OH)_3$ by Eq. (6) and Al_2O_3.

$$Al^{3+} + H_2O \rightarrow Al(OH)^{2+} + H^+ \qquad (4)$$

$$Al(OH)^{2+} + H_2O \rightarrow Al(OH)_2{}^+ + H^+ \qquad (5)$$

$$Al(OH)_2{}^+ + H_2O \rightarrow Al(OH)_3{}^0 + H^+ \qquad (6)$$

$$Al(OH)_3{}^0 + H_2O \rightarrow Al(OH)_4{}^- + H^+ \qquad (7)$$

Considering only mononuclear speciation, the concentration of the various Al forms present in solution (α) was calculated by Holt et al. [26] depending on pH. Fig. 1 provides the speciation diagram obtained by the authors. Al complexes acting as coagulants are adsorbed on the particles and thus neutralise the colloidal charges, resulting in destabilization of the emulsion. This phenomenon is similar to the action of chemical coagulants in the conventional chemical treatment. Hydrogen bubbles formed at the cathode can adsorb on the flocculated species and induce their flotation. The bubbles formed also reduce fouling of the cathode surface which could occur by the formation of deposits.

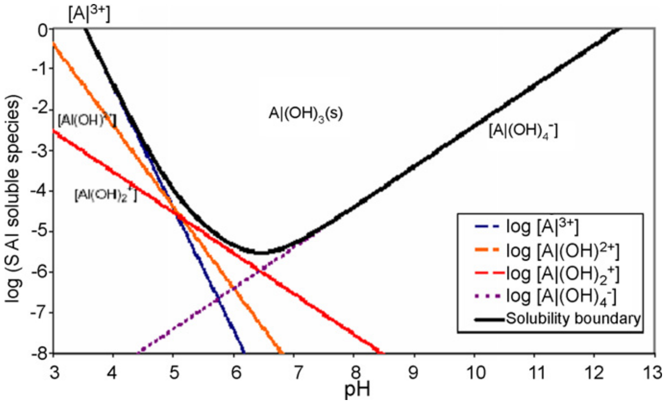

Fig. 1. Solubility diagram of aluminium hydroxide $Al(OH)_3(s)$ considering only mononuclear aluminium species. Data from [26].

NaCl is usually employed to increase the conductivity of the water or the wastes to be treated. The presence of the chloride ion in solution has been reported to decrease passivation of the Al surface and thereby increase the efficiency of electrocoagulation processes [23,24]. All authors attributed the effect of chloride ion to its role in the "pitting" corrosion of the metal surface. Mameri et al. [18] postulated a mechanism for the reduction in Al passivation by the oxide layer formed, and the overall equations can be written as

$$2Al + 6HCl \rightarrow 2AlCl_3 + 3H_2 \qquad (8)$$

$$AlCl_3 + 3H_2O \rightarrow Al(OH)_3 + 3HCl \qquad (9)$$

Because of EC complexity, previous researchers have adopted a largely empirical approach to understand the various processes involved [8,19,26]: the capability of removal the pollutant by their technology was clearly demonstrated and analysed. The published works have often resulted in various EC systems whose design and operations were developed to meet the specification of the specific pollutants investigated. However most of these contributions have failed to extract and to quantify the key underlying mechanisms of pollutant removal.

The efficiency of EC processes is to be controlled by several parameters, e.g. current density, pH, flow rate, electrode materials, and the amount of matter to be removed. In this study, treatment of cutting oil emulsions was considered as a pretreatment step prior to a membrane process. The progress of the treatment carried out in a batch system provided with recirculation of the flow, was followed by time variations of COD and turbidity.

Table 1
Characteristics of the emulsions used

Oil concentration	2%	4%	6%
COD (mg O_2/L)	60 282	108 208	116 128
Turbidity (NTU)	26 400	51 712	64 125
pH before NaCl addition	8.63	9.09	9.69
pH after NaCl addition	8.43	9	9.3
Conductivity before NaCl addition (μs/cm)	257	322	419
Conductivity after NaCl addition (μs/cm)	3 150	3 350	3 730

2. Materials and methods

2.1. Chemical and analytical techniques

The metal used for the cell electrodes was Al alloy AU4G (2017-Al). It is mainly produced by recovery of aluminium waste, which explains its very accessible cost. The alloy contains Cu at 4%, Fe, Mg and Mn each at 0.7%, Si at 0.5% and lower percentages of Zn and Cr.

Oil–water emulsions were prepared from a cutting mineral oil B22 supplied by Naphtal (Sonatrach, Algeria) and currently used for drilling and machining operations. The emulsions were diluted in deionized water to form very stable emulsion with mean diameter droplet and the zeta potential equal, respectively to 112 nm and −30 mV. The zeta potential was measured using a Malvern Zetasizer 3000HS. The diameter of the oil droplet was measured using Malvern Mastersizer nano S. In all cases, sodium chloride at 1.5 g L^{-1} concentration was added for sufficient electrical conductivity of the emulsion to be treated, as suggested by Sánchez-Calvo et al. [13] and Chen et al. [27]. This addition has negligible effect on the initial pH of the emulsion (Table 1). Carmona et al. [28] observed that the characteristics of these types of emulsions were not affected by the addition of supporting electrolyte such as NaCl at low/moderate concentrations. COD and turbidity levels of the dilute emulsions are very high (Table 1) and nearly proportional to their concentration, as expected. The progress of electrocoagulation in the batch process was followed by measurement of COD, turbidity, conductivity and pH. The COD levels of the samples were determined using the standardized colorimetric technique with an excess of hexavalent chromium and subsequent measurement of the optical density. Turbidity was measured by a Hanna Instrument LP 2000 turbidimeter which measures the quantity of light absorbed by the suspended particles in comparison with a standard solution. Accuracy of the two analytical techniques was better than 5%. The pH was measured using a Hanna Instrument pH 211 Microprocessor pH Meter. Conductivity was measured using a Hanna Instrument Conductivity Meter EC 214. The main features of the emulsions used are given in Table 1.

2.2. Set-up and protocol

The batch experimental set up is shown in Fig. 2. The system consisted of an EC cell, a cylindrical reactor, a peristaltic pump for the recirculation of the effluent and a sampling valve to collect the samples. The EC cell consisted of two polymeric halves which were bolted to form the rectangular channel cell. An aluminium electrode with dimensions of 100 mm × 50 mm × 12 mm, was embedded in each of the two valves: only one face was exposed to the solution and the effective electrode area was 5.0×10^{-3} m^2. The electrode gap was maintained constant at 20 mm. The electrodes were connected to a digital dc power supply (P. Fontaine, MC4020C, 40 V, 2 A). Two digital multimeters (Multimetrix X1000) were used to measure the current passing through the circuit and the applied potential, respectively. The tank reactor with a capacity of 1.5×10^{-3} m^3 was made in Plexiglass^{TM} and was provided with inlet and outlet fittings for recirculation of the liquid. Homogenisation of the effluent was ensured by magnetic stirring at approximately 250 rpm. The gentle agitation allowed the gases to be separated from the liquid, thus avoiding the formation of foam which could affect the occurrence of electrocoagulation.

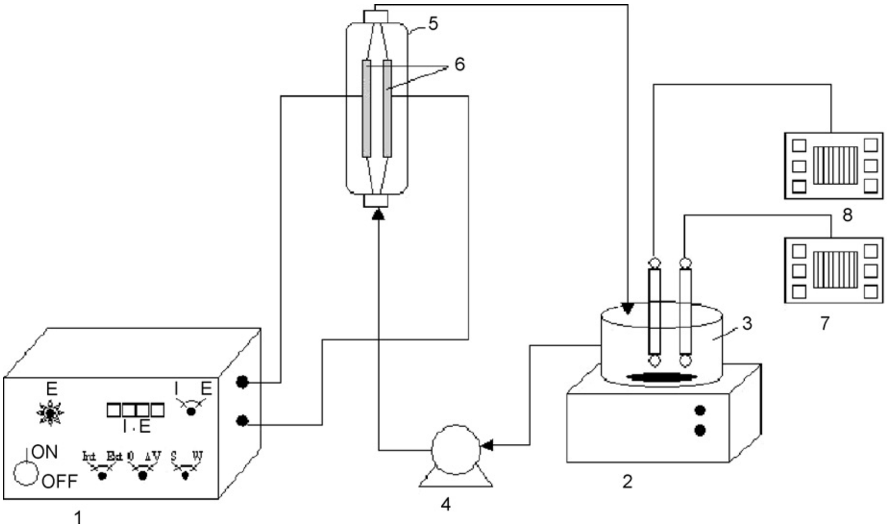

Fig. 2. Schematic diagram of experimental set-up. (1) dc power supply; (2) magnetic stirrer; (3) plexiglass reactor; (4) peristaltic pump; (5) electrochemical cell; (6) aluminium electrodes; (7) conductimeter; (8) pH meter.

To avoid passivation of the electrodes, the electrochemical cell was entirely cleaned after each experiment with detergent and acetone, as described by Kobya et al. [15]. All experiments were carried out at room temperature near 25 °C.

One litre of fresh emulsion was introduced into the tank, and after the NaCl was added, the selected current was applied to the cell. At regular intervals, samples were collected and pH was adjusted to 7 by the addition of aliquots of concentrated hydrochloric acid for optimal precipitation of aluminium hydroxide as reminded in Fig. 1 [13]. The collected samples were allowed to settle for 24 h, COD and turbidity were then determined from the clear fractions recovered. Contrary to previous observations with others suspensions [29] the only adjustment of pH did not result in visible COD abatement in the B22 oil suspensions. It has to be mentioned that accidental entrainment of a small sludge particle could occur drawing the clear liquid from the settling tube, causing an unexpected additional error in the analytical procedure. Replicates of most experiments were made to overcome this experimental issue. Analysis of the results of replicate experiments led to estimate that the global uncertainty in determination of COD and turbidity was near 5%.

3. Results and discussion

3.1. General aspects

This study is mainly focused on the electrocoagulation treatment of cutting oil emulsions with very high concentration for investigation of the effects of the main operating parameters, e.g. initial pH, current density, oil concentration and recirculation rate. Turbidity and COD values were used to evaluate the EC progress and the removal efficiency. For all cases, two phases were observed during the discontinuous treatment: (i) a "reactive" phase for which the abatement of COD and turbidity increases regularly with time and (ii) a steady phase for which further Al dissolution has no effect on the treatment efficiency. The intercept between the two periods can be considered as the minimum time required for the treatment, corresponding to the minimum required concentration of dissolved Al.

The progress of electrocoagulation is to be linked to the amount of dissolved aluminium. The theoretical concentration of dissolved Al, C_{Al} in g/L, can be expressed by Faraday's law as follows

$$C_{\mathrm{Al}} = \frac{M\Phi_{\mathrm{e}}It}{nFV} = \frac{M\Phi_{\mathrm{e}}Q}{nFV} \qquad (10)$$

where M, I, t, n, F and V, are, respectively the molecular weight of aluminium (g/mol), the current (A), the electrolysis time (s), metal valence (3 for Al), Faraday constant (96 500 C/mol) and the emulsion volume in the overall circuit. Q is the electrical charge passed during the galvanostatic runs and Φ_{e} is the current yield. For appreciable change in the volume of the liquid in the flow rig because of the repeated samplings, Eq. (10) had to be modified accordingly. The current efficiency for Al dissolution was previously found to be larger than unity [10,29], and the actual concentration of Al can attain 150% of the theoretical level, due to the chloride-induced dissolution mentioned above.

3.2. Effect of oil concentration

The experiences were followed at constant current density ($i = 100\ \mathrm{A/m^2}$), the initial pH of emulsion was adjusted to 7, and the liquid velocity in the cell was $5.33\ 10^{-3}$ m/s, corresponding to $320\ \mathrm{cm^3\ min^{-1}}$ through the $10\ \mathrm{cm^2}$ cross-sectional area. We observe in Fig. 3 that removal of COD and turbidity expressed in %, are lower with higher oil concentration. This is can be explained by the fact that higher amounts of Al ions are required for the treatment of more concentrated oil suspensions. For a 2 vol.% emulsion, the treatment does not progress any further after 35 min, the residual COD was equal to 1475 mg O_2/L, corresponding to 98% COD abatement. Final turbidity was near 507 NTU, corresponding to the same abatement. For higher oil concentrations, the stationary plateau of COD and turbidity was reached after more than 1 h. This result is not in agreement with previous work [29] also conducted with suspensions of machining oil, for which the abatement yield was observed to be nearly independent of the amount of suspended matter. However, the dependence of the concentration was shown to largely depend on the nature of the pollutant to be treated and latex suspensions had been shown to behave like the present cutting oil [29].

The above results were plotted on the basis of the specific electrical charge per gram oil, Q/m_{oil} (Fig. 4). Such a plot shows that the electrical charge required for the treatment of the investigated cutting oil – and the amount of dissolved aluminium – is directly proportional to the concentration of oil in the emul-

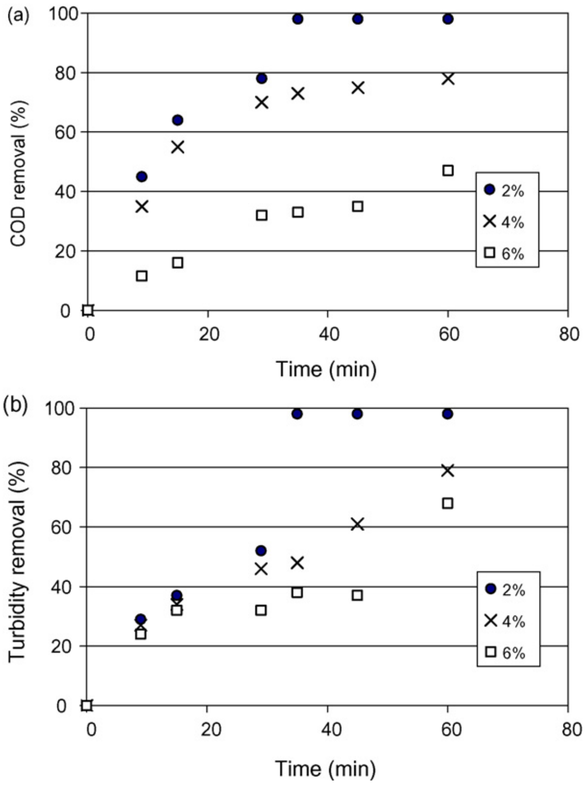

Fig. 3. Time variations of COD (a) and turbidity (b) removal depending on the oil concentration at $i = 100\ \mathrm{A/m^2}$, initial pH 7, liquid flow rate = $320\ \mathrm{cm^3/min}$. Oil concentrations: (●) 2%; (×) 4%; (□) 6%.

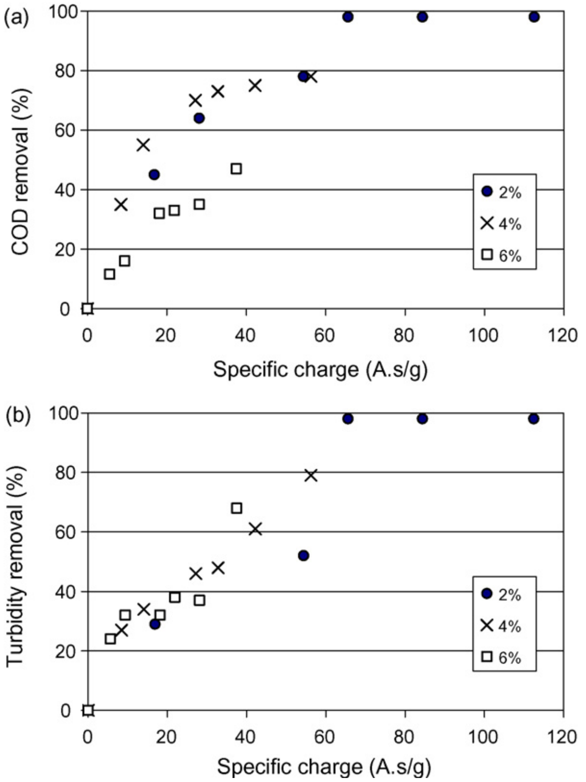

Fig. 4. Variations of COD (a) and turbidity (b) removal with the specific charge (Q/m_{oil}) depending on the oil concentration at $i = 100\,\mathrm{A/m^2}$, initial pH 7, liquid flow rate = 320 $\mathrm{cm^3/min}$. Oil concentrations: (●) 2%; (×) 4%; (□) 6%.

sion to be treated. Treatment conducted in the above conditions appears to be completed upon application of 70 As/g oil.

3.3. *Influence of the current density*

Three current densities 100, 150 and 200 $\mathrm{A\,m^{-2}}$ were tested for the treatment of 4 vol.% emulsions. The initial pH was the natural pH of the diluted emulsion, around 9, and the flow rate was 320 $\mathrm{cm^3\,min^{-1}}$. The results, expressed in the form of COD and turbidity reduction versus time, are shown in Fig. 5. During the reactive phase, the reduction rate of COD and turbidity were observed to increase with the current density. This can be explained by the fact that the amount of $\mathrm{Al^{3+}}$ species formed by dissolution of the anode, increases with the current density according to Faraday's law (Eq. (10)) and previous results [12,15,29]. Higher amounts of dissolved aluminium allowed higher coagulation efficiency and more significant destabilisation of the emulsion. Moreover as shown by Khemis et al. [29], higher production rates of hydrogen allowed by higher currents, favors the flotation of the flocculated matter.

This is not true in the second phase of the treatment: during this period the separation process is not controlled by coagulation but by other phenomena, e.g. formation of solid, liquid/solid aggregation and adsorption of destabilized particles onto solid $\mathrm{Al(OH)_3}$ formed. As done in Section 3.1, the data could be successfully plotted versus the specific charge (Q/m_{oil}), as shown in Fig. 6. For the initial pH considered, around 80 As/g are required for the treatment of the emulsion whatever the current density.

Fig. 7 represents the variation of the emulsion pH during the electrolytic run. The pH was observed to increase rapidly during the reactive phase, then to stabilise at pH close to 11. This progressive increase in pH is explained by the occurrence of water electrolysis resulting in hydrogen evolution and production of $\mathrm{OH^-}$ ions which are partly buffered by the various forms of Al-hydroxides. As can be expected from [20,21], and demonstrated in Fig. 1, at pH equal or higher than 11, aluminium hydroxide is mainly in the form of soluble forms, which is to limit the efficiency of the flocculation process. The relative stabilisation of the pH, for 80 As/g, corresponds to the end of the treatment.

3.4. *Effect of liquid flow rate*

The effect of the flow conditions on the electrocoagulation efficiency of the liquid waste has been scarcely investigated. We studied here the influence of this parameter for the treatment of 4 vol.% emulsions. As shown in Fig. 8, the EC progress observed by the removal of COD and turbidity, was slightly better at high flow rates. 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 is to induce a greater number of collisions between the particles of $\mathrm{Al(OH)_3}$ and the destabilized oil droplets, thus improving the flocculation.

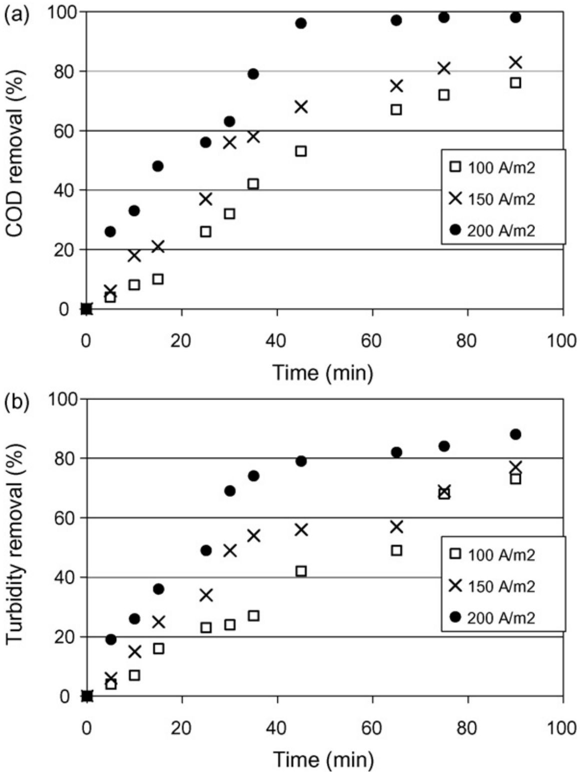

Fig. 5. Time variations of COD (a) and turbidity (b) removal depending on the current density; initial pH 9; oil concentration = 4%, liquid flow rate = 320 $\mathrm{cm^3/min}$. Current density: (□) 100 $\mathrm{A/m^2}$; (×) 150 $\mathrm{A/m^2}$; (●) 200 $\mathrm{A/m^2}$.

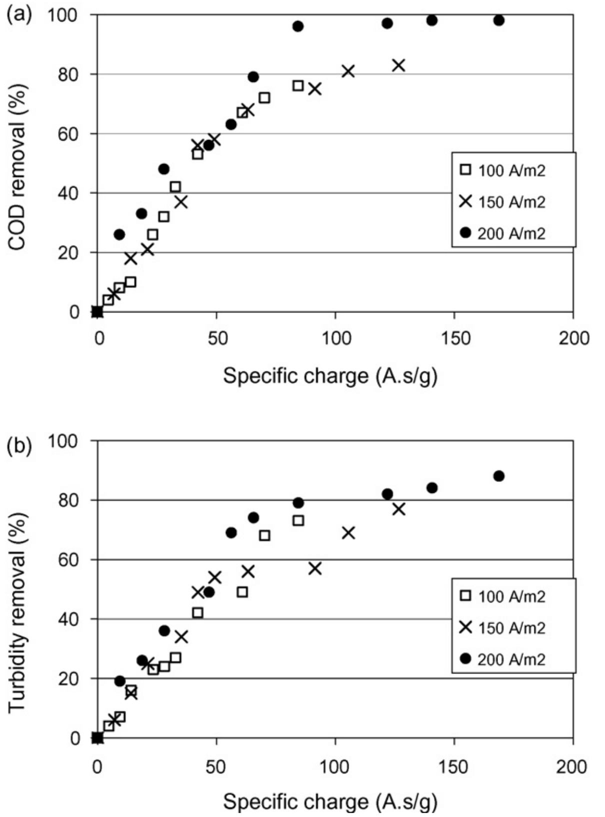

Fig. 6. Variations of COD (a) and turbidity (b) removal with the specific charge (Q/m_{oil}) depending on the current density; initial pH 9, oil concentration = 4%, liquid flow rate = 320 cm^3/min. Current density: (□) 100 A/m^2; (×) 150 A/m^2; (●) 200 A/m^2.

However, we observed that the flow rate effect on the turbidity abatement was more significant for the highest values of current density (Fig. 8). This reveals the synergetic effect of: (i) the anode 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 COD abatement was more significant at low current densities, and was hardly visible at 200 A m^{-2}. These observations are illustrated in Fig. 9 which gives the influence of the flow rate and the current density on the COD and turbidity abatement, after 90 min of electrolysis, corresponding to maximum treatment in all cases.

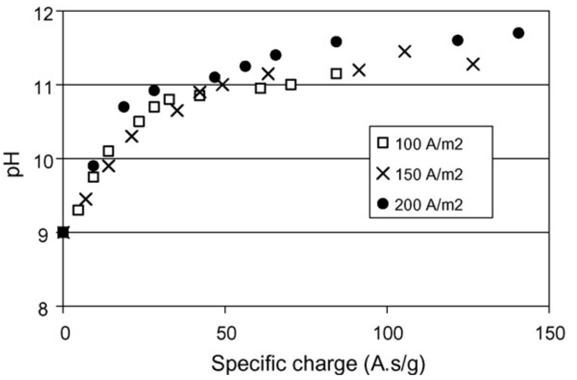

Fig. 7. Variation of the liquid pH with the specific charge (Q/m_{oil}) during the electrocoagulation according to current density; initial pH 9, oil concentration = 4%, liquid flow rate = 320 cm^3/min. Current density: (□) 100 A/m^2; (×) 150 A/m^2; (●) 200 A/m^2.

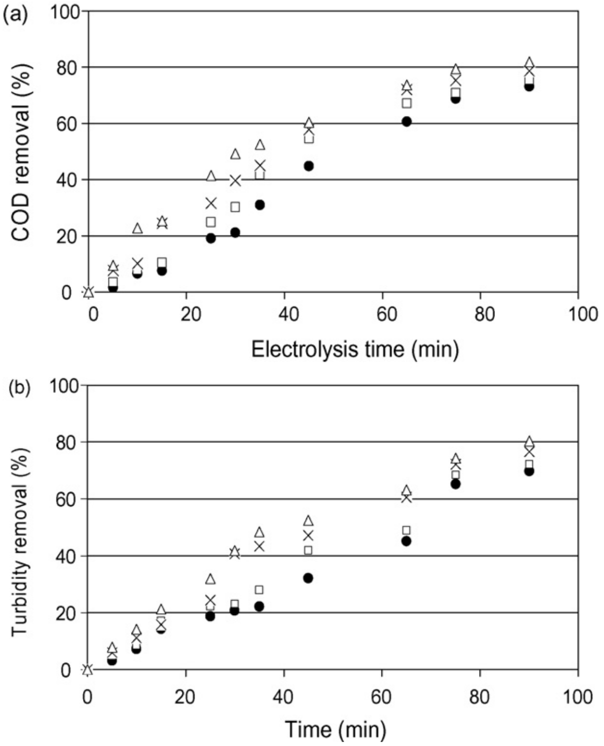

Fig. 8. Time variation of COD (a) and turbidity (b) removal depending on the flow recirculation, initial pH 9, i = 100 A/m^2, oil concentration = 4%. Liquid flow rate: (●) 130 cm^3/min; (□) 320 cm^3/min; (×) 520 cm^3/min; (Δ) 750 cm^3/min.

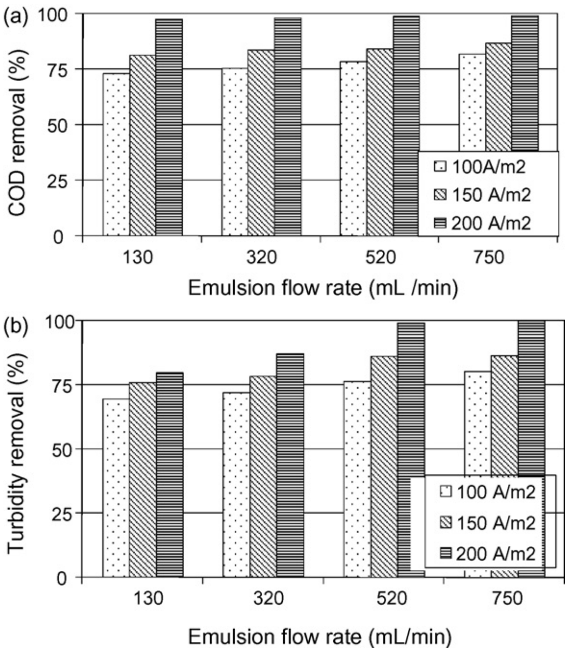

Fig. 9. Effect of the recirculation flow rate on removal efficiency, initial pH 9, oil concentration = 4% and electrolysis time = 90 min.

Table 2
Effect of the initial pH on efficiency removal after 60 min of electrolysis

Initial pH	Final COD (mg O_2/L)	Final turbidity (NTU)	COD removal (%)	Turbidity removal (%)	Final pH
3	10 036	24 587	91	52	6.8
5	9 856	20 083	91	61	7.8
6	8 652	630	92	99	8.0
7	10 058	610	91	99	8.4
9	28 516	19 024	74	63	10.5
11	43 251	23 581	60	54	10.6

Current density = 100 A/m^2; oil concentration = 4 vol.%, i.e. (Q/m_{oil}) = 56.25 As/g; liquid flow rate = 750 cm^3/min.

3.5. *Effect of initial pH of emulsions*

It has been established that the initial pH is an important operating factor influencing the performance of EC process [10,12,27]. To examine its effect, the pH of the emulsion to be treated was adjusted to the desired value by aliquots of sodium hydroxide or hydrochloric acid. The current density was fixed at 100 A m^{-2} and the oil concentration was 4 vol.%. Table 2 shows the removal yields of COD and turbidity after 60 min of electrolysis, depending on the initial pH. The maximum removals of COD, and turbidity were observed at neutral pH 6–7, and this is in agreement with many previous works related to EC using aluminium electrodes [11,13,15]. For neutral or acidic pH, the effect is less significant on the COD removal whose value is higher than 90%. The drop in turbidity removal, in acid or basic media, is in accordance with the amphoteric character of aluminium hydroxide $Al(OH)_3$ whose solubility increases as the solution becomes either more acidic or alkaline (Fig. 1). Solid precipitate of aluminium hydroxide formed at pH 6–7 is a precursor for oil removal by coagulation. Furthermore, as shown in Table 2, the more acid the initial liquid is, the higher is the increase in pH during the run. This phenomenon was ascribed by Vik et al. [25] to hydrogen evolution at cathode and according to reaction (2).

In alkaline medium (pH > 7), the final pH does not vary very much and a slight drop was recorded. However for initial pH over 10, ionic forms of Al-hydroxides predominate, which reduces the efficiency of the treatment by electrocoagulation.

4. Conclusion

Electrocoagulation using aluminium electrodes is a feasible process for treatment of very stable cutting oil emulsions, characterized by high COD and turbidity. Whatever the operating conditions, EC proceeds in two phases: a reactive phase in which the removal efficiency increases regularly with time – and the amount of dissolved Al – and a stationary phase for which further energy consumption for solubilisation of aluminium anode is useless. The reactive phase is as longer as the oil concentration is high, and can be reduced by using higher current density and, to a lower extent by higher liquid recirculation rate. The optimal initial pH was found equal to 6–7 and remain for which the COD and turbidity removal correspond, respectively to 92 and 99%.

In spite of very important removal efficiency, the final values of COD (8652 mg O_2/L) and turbidity (610 NTU) remain higher than standard rejections (120 mg O_2/L and 5 NTU). In this fact, the EC process would be appropriate perfectly as pretreatment step prior to a membrane process. Besides, treatment of the investigated cutting oil, although incomplete, consumes from 60 to 80 As/g oil, corresponding to 5.6–7.4 mg Al/g oil assuming a current efficiency at 100%. In reality, higher dissolution yields are to be expected, and the Al concentration required would be of the order of 10 mg/g oil. This value is a positive aspect of the technique, since the sludge to be produced by the treatment is to have an Al content of 1 wt.%, which is satisfactory in view to disposing the sludge in safe conditions.

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