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The optimization of the electroflotation process using DSA® electrodes for treating the simulated effluent of produced water from oil production

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ABSTRACT

One of the main problems faced by the oil industry is related to the amount of produced water generated during the oil extraction process, and therefore, the disposal of this effluent, which must undergo treatment processes to meet the specifications for disposal or reinjection into oil wells. Thus, this research aimed to study the oil–water separation through the electroflotation process, using a simulated wastewater prepared by mixing water, lubricating oil and NaCl. To this end, the effect of operational variables: effluent input rate, current density and NaCl concentration were studied in a pilot system. The performance of the process was determined by the removal of oil and grease (OG) content in the treated effluent. According to the results, the electroflotation process showed a good separation efficiency, presenting an OG removal of 94.96% for the input rate of the effluent from 62 $m^3 m^{-2} d^{-1}$, current density of 80 A m^2 and the NaCl concentration of 7,900 mg L^{-1}. The final concentration of OG was 13.15 mg L^{-1}. These results encourage the applicability of electroflotation as a treatment process for the petrochemical industry.

Keywords: Effluent treatment; Electroflotation; Experimental planning; Oil; Produced water

1. Introduction

In the exploration and production of oil and natural gas, waste and effluents are generated, of which the produced water stands out consisting of formation water (water naturally present in the geological formation of the oil reservoir) and water injection (water injected into the reservoir to increase production) [1].

This produced water contains contaminants, including hydrocarbons, heavy metals and chemical additives [2,3]. Among the more soluble and toxic species present in the produced water, aromatic compounds such as benzene, toluene, ethylbenzene, xylene isomers and phenols are prominent. Still containing high salinity, suspended solids, soluble and insoluble organic matter, oil in suspension and/or emulsified and sometimes radioactivity, making them a pollutant difficult to discard aggravated by the significant amount involved.

According to OGP [1], the amount of produced water in a mature field can exceed 10 times the volume of oil produced. In view of this magnitude, the final disposal of this water is important, either for operational or environmental reasons.

Produced water, regarded as an effluent to be discarded, will have to undergo effective treatments in order to fit into the current legislation with a vision for its final destination. If reinjected or reused in another way, it needs to be treated to meet the standards necessary for the process to be used.

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A well studied and promising process today is the electrochemical, where through the electron action itself, substances that are toxic and harmful to the environment are removed or transformed through oxidation–reduction reactions into less toxic substances.

The application of electrochemical technology has always been limited by the stability of the electrode material. However, a major breakthrough occurred in this area in 1970s with the development of the dimensionally stable anodes (DSA), which provides a broad application without deterioration of the electrodes [4]. DSA have excellent electrochemical properties which have encouraged studies on the application of these materials in wastewater treatment and industrial water [5–11].

This technology makes it possible to expand the treatment capacity of traditional physical–chemical systems because it uses the same basic fundamentals of coagulation–flocculation [12].

In this context, the present study evaluated the influence of operational variables: effluent input rate, current density and NaCl concentration, the efficiency of oil and grease (OG) content removal in the electroflotation process using stable electrodes.

2. Materials and methods

This research was developed at the Laboratory of Water Potabilization (LAPOA), located in the Department of Sanitary and Environmental Engineering, Federal University of Santa Catarina (UFSC), Florianópolis, SC, Brazil.

2.1. Produced water from oil

The produced water from oil used in the experiments consisted of a mixture of water, common lubricating oil (20W50 Ipiranga F1 Master Protection) and NaCl, which was labeled as "study water".

The NaCl was added to the study water in order to increase the effluent conductivity and thereby decrease the applied electric charge.

The study water was prepared in the input reservoir, with the following proportions: 250 L of water, 142.26 mL of lubricating oil (for an initial concentration of 500 mg L^{-1}) and the NaCl concentrations ranged from 3,000 to 13,000 mg L^{-1}. The mixing was performed by a centrifugal pump of ½ horse potence (HP), which promoted the recirculation of the effluent in the tank itself for 30 min. After mixing, the study water had a pH level at approximately 7.

2.2. Pilot system

The experimental system used in the study consisted of an electrochemical reactor; a stabilizing voltage source (INSTRUTEMP – ITFA 5020), used for the determination of the current density; a centrifugal pump of ½ HP (Schneider – BC-98), used for recirculating the effluent; a metering pump (Grabe – DDM-130-07 PP/TF-1), used to control the effluent from the input flow, hydraulic and electrical systems; and two reservoirs with 500 L capacity (input/output) as shown in Fig. 1.

2.2.1. Electrochemical reactor

The electrochemical reactor used in research has 115 mm internal diameter and a useful volume of 2.08 L.

The electrodes used contained titanium cathodes and dimensionally stable anodes DSA® type ("De Nora"), composed of $Ti/Ru_{0.3}Ti_{0.7}O_2$.

An arrangement of 10 electrodes in parallel were used, 5 cathodes and 5 anodes arranged interchangeably, with a total effective area of 785 cm^2 and a distance of about 0.8 cm between the electrodes, as shown in Fig. 2.

2.2.2. Operation of pilot system

After preparing the study water in the inlet reservoir, via a metering pump, suction of this water to the electrochemical reactor occurred, where the electroflotation process took place due to the potential difference applied to the electrodes by the power source. The samples of the study water before and after the treatment were conducted in the collected taps of raw water and treated water, as shown in Fig. 1. After treatment, the water was intended to the output reservoir.

The oil foam obtained during the electroflotation process was collected in a reservoir at the upper part of the pilot system.

2.3. Experimental procedure

In order to identify the best conditions of the reactor operational parameters (ROP), preliminary tests were conducted

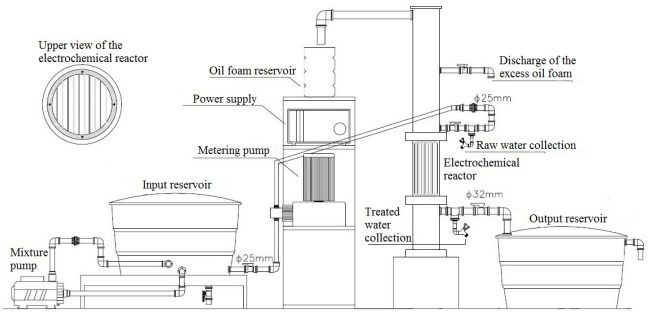

Fig. 1. Scheme of pilot system used in the research.

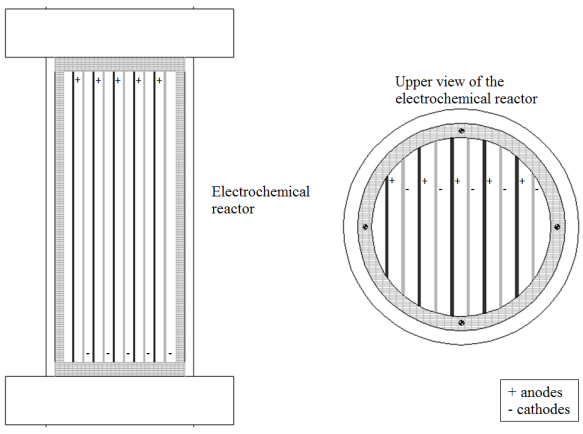

Fig. 2. Scheme of electrochemical reactor used in the research.

in which the study water was treated at ambient temperature (25°C) under different conditions by varying the input rate of the effluent, the current density and NaCl concentration.

After 180 min of each test, rates of treated effluent were collected and the OG analyzes were conducted.

To determine the optimal conditions of ROP and identify the maximum efficiency of the electroflotation process, an experimental design using Statistica® software was carried out.

2.4. Experimental planning and statistical analysis

The central composite rotational design (CCRD) was conducted in triplicate at the center point, with a total of 17 trials. The levels of the variables in the optimization of electroflotation process can be seen in Table 1.

The independent variables of the electroflotation process were the effluent input rate, the current density and the NaCl concentration.

The dependent variable or response was the OG removal rate.

The matrix CCRD with the coded values of the independent variables for the study water is shown in Table 2.

The results obtained in the experimental design were statistically analyzed, and this analysis comprised the analysis of variance (ANOVA) statistical model to forecast and contour plots. To obtain these responses, the Statistica® software was used.

The operational conditions of higher performance of the studied treatment system were obtained in this way.

2.5. Optimization of the electrolysis time in function of the removal of OG from the study water

Experiments were carried out using the optimum conditions of ROP obtained by the experimental design, taking samples of 100 mL, with times of 0, 30, 60, 90, 120, 150 and 180 min for analyzing the removal of OG.

The analysis of the OG was performed according to the procedures described in Method 5520B Standard methods for the examination of water and wastewater [13].

3. Results and discussion

3.1. Experimental planning and statistical analysis of the electroflotation process results

The experimental design used for the interaction among all variables and levels, and the results obtained in the OG removal of the study water through the electroflotation process are presented in Table 3.

According to the data in Table 3, the OG removal rate ranged from 38% to 90%, considering all the effects of the independent variables and their interactions within the experimental range studied in this research.

The results were analyzed statistically combining the actions of the variables using the model shown in Eq. (1).

$$R(\%) = 83.84 - 16.43 * X_1 + 5.29 * X_2 - 0.55 * X_3 - 6.56 * X_1^2 - 9.32 * X_2^2 - 8.44 * X_3^2 \quad (1)$$

As R (%) is the percentage oil removal; 83.84 is a constant; X_1 is the input rate ($m^3\ m^{-2}\ d^{-1}$); X_2 is the current density ($A\ m^{-2}$); X_3 is the NaCl concentration ($mg\ L^{-1}$); X_1^2 is the input rate; X_2^2 is the current density and X_3^2 is the NaCl concentration such as quadratic terms of the equation.

The equation fits the experimental data very well, as shown in Fig. 3.

The values obtained by the mathematical model and those observed in experiments showed little dispersion, with r^2 0.974 (Fig. 3), which proved a good fit of the model for the experimental data, because the dispersion was directly related to a beneficial correlation between the provided data and the experimental data.

Table 2
Matrix of experimental design

Test	Experimental conditions		
	q_1	q_2	q_3
1	–1	–1	–1
2	+1	–1	–1
3	–1	+1	–1
4	+1	+1	–1
5	–1	–1	+1
6	+1	–1	+1
7	–1	+1	+1
8	+1	+1	+1
9	–1.68	0	0
10	+1.68	0	0
11	0	–1.68	0
12	0	+1.68	0
13	0	0	–1.68
14	0	0	+1.68
15	0	0	0
16	0	0	0
17	0	0	0

Table 1
Levels of the independent variables investigated in the electroflotation process

Independent variables	Variables	Levels				
		–1.68	–1	0	+1	+1.68
Input rate ($m^3\ m^{-2}\ d^{-1}$)	q_1	50.0	70.0	100.0	130.0	150.0
Current density ($A\ m^{-2}$)	q_2	17.0	38.0	70.0	100.0	126.0
NaCl ($mg\ L^{-1}$)	q_3	3,000	5,000	8,000	11,000	13,000

Table 3
Experimental conditions and results of the CCRD in triplicate with the central point for OG removal (%) of the study water using the electroflotation process for 180 min of electrolysis

Test	Input rate ($m^3 m^{-2} d^{-1}$)	Current density ($A m^{-2}$)	NaCl ($mg L^{-1}$)	OG removal (%)
1	70.0	38.0	5,000	75 ± 1
2	130.0	38.0	5,000	40 ± 1
3	70.0	100.0	5,000	79 ± 1
4	130.0	100.0	5,000	42 ± 1
5	70.0	38.0	11,000	71 ± 1
6	130.0	38.0	11,000	38 ± 1
7	70.0	100.0	11,000	81 ± 1
8	130.0	100.0	11,000	45 ± 1
9	50.0	70.0	8,000	90 ± 1
10	150.0	70.0	8,000	40 ± 1
11	100.0	17.0	8,000	42 ± 1
12	100.0	126.0	8,000	72 ± 1
13	100.0	70.0	3,000	61 ± 1
14	100.0	70.0	13,000	58 ± 1
15	100.0	70.0	8,000	83 ± 1
16	100.0	70.0	8,000	83 ± 1
17	100.0	70.0	8,000	84 ± 1

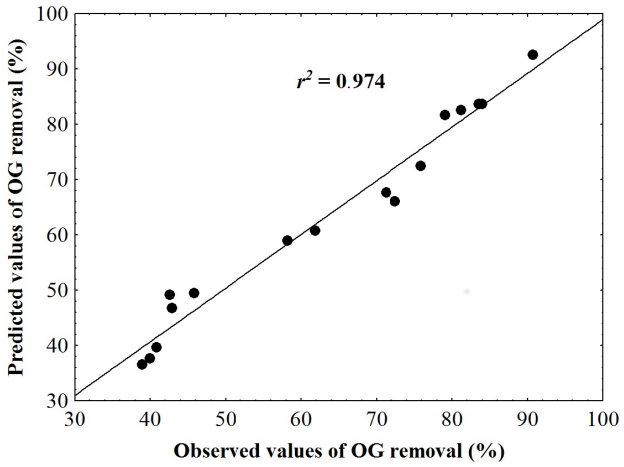

Fig. 3. The correlation between the values observed in OG removal and their corresponding values provided by the statistical model proposed for treating the study water through the electroflotation process.

An ANOVA was performed in order to validate the adjustment of the provided model through the obtained results. The ANOVA results are presented in Table 4.

ANOVA showed that the model provided (Eq. (1)) was valid at a 95% confidence interval, as shown in Table 4. As the $F_{calculated}$ (29.53) > $F_{tabulated}$ (3.68), it can be said that the values of the experimental responses (R) resulted in a useful reproduction of the data.

According to the statistical analysis and interpretation of the results, the highest efficiency of the electroflotation process of OG removal was achieved in the following operating conditions of the electrochemical reactor: 62 $m^3 m^{-2} d^{-1}$, 80 A m^{-2} and 7,900 mg L^{-1} which are effluent input rate, current density and NaCl concentration, respectively.

These results are best seen in Fig. 4, when on the basis of responses (R%) obtained in the experimental design, contour plots were constructed using the proposed statistical model.

Fig. 4 shows that all operational parameters significantly influenced the efficiency of the electroflotation process. The OG removal rate rose considerably with the increase of the current density and the concentration of NaCl values were near the center point of the experimental design.

Increased OG removal rate with the current density occurred due to the increased amount of gas bubbles which were generated in the electroflotation process, allowing greater separation of oil–water efficiency.

Decreased OG removal rate with increasing NaCl concentration and current density for values above the midpoint of the experimental design was probably due to the large amount of chlorine bubbles produced, since excess chlorine bubbles reduced the removal rate efficiency due to the coalescence of the bubbles [14].

OG removal increases when the effluent from the input rate is a value near the lower levels of the experimental design, this is due to the fact that the lower the input rate is, greater is the residence time, i.e., the effluent has more contact time with the electroflotation process and the turbulence of the feed stream into the reactor is lower.

Nahui et al. [15] used DSA® electrodes in the electroflotation process for treating oily water. The variables studied were the current density, oil concentration and NaCl concentration. A removal of 99.71% of the OG was observed for a current density of 233.76 A m^{-2}, initial concentration of oil of 1,050 ppm and NaCl concentration of 35,000 ppm. As in the present research, the authors concluded that increases in current density and salinity improved oil removal.

Gargouri et al. [16] studied the application of electrochemical technology for produced water treatment using lead dioxide (Ta/PbO_2) and boron-doped diamond (BDD) electrodes. Experiments were conducted at different current

Table 4
The analysis of variance test of the model provided for the OG removal values of the study water through the electroflotation process, at a confidence level of 95% ($p < 0.05$)

Parameter	Source of variance	Sum of squares	Degrees of freedom	Mean squares	F		Significance level (%)
					Calculated	Tabulated	
OG	Regression	5,548.58	9	616.51	29.53	3.68	<0.01
	Residual	146.19	7	20.88			
	Total	5,694.77	16				

densities (30, 50 and 100 mA cm^{-2}). The chemical oxygen demand (COD) removal was approximately 85% and 96% using PbO_2 and BDD reached after 11 and 7 h, respectively, at a current density of 100 mA cm^{-2}. This time difference in relation to the present research can be attributed to the use of DSA® anode that has better electrocatalytic properties than the lead anode [15].

Dermentzis et al. [17] studied the application of electrochemical process for oily wastewater treatment using platinized titanium (Ti/Pt) and BDD electrodes. The electro-oxidation process with Ti/Pt and BDD electrodes at the applied current density of 20 mA cm^{-2} reduces COD to 15% and 36% at 25° and to 28% and >98% at 60°C, respectively, in 120 min electrolysis. In a similar study, Rocha et al. [18] also concluded that an increase of temperature favors organic oxidation due to an increase of the indirect reaction of organics with electrogenerated oxidizing agents from electrolyte solution.

Dos Santos et al. [9] studied the electrochemical oxidation system for the treatment of produced water using platinized titanium (Ti/Pt) and BDD anodes. At a current density of 15 and 30 mA cm^{-2} using Pt electrode, 33.8% and 46.5% of COD removals were achieved, respectively. Under similar conditions, at BDD anode, 50.3% and 57.5% of COD elimination were obtained after 10 h of treatment. Afterward, the authors studied the application of a current density of 40 mA cm^{-2}, varying the temperature (25°C, 40°C and 60°C) and observed a reduction in treatment time. The COD removals after 5 h of treatment were 83.2%, 87.4%, and 92.1% at Ti/Pt, while for BDD, the influence on temperature contributes with a modest increase on oxidation rate, and COD removals of 81.9%, 91.8% and 94.5% were achieved.

As noted in the studies cited previously, the application of DSA® electrodes appears to be an interesting alternative compared with others stable electrodes, because according to the results presented in this research, has a good removal efficiency of organic contaminants and requires a shorter treatment time without the need to change the temperature effluent.

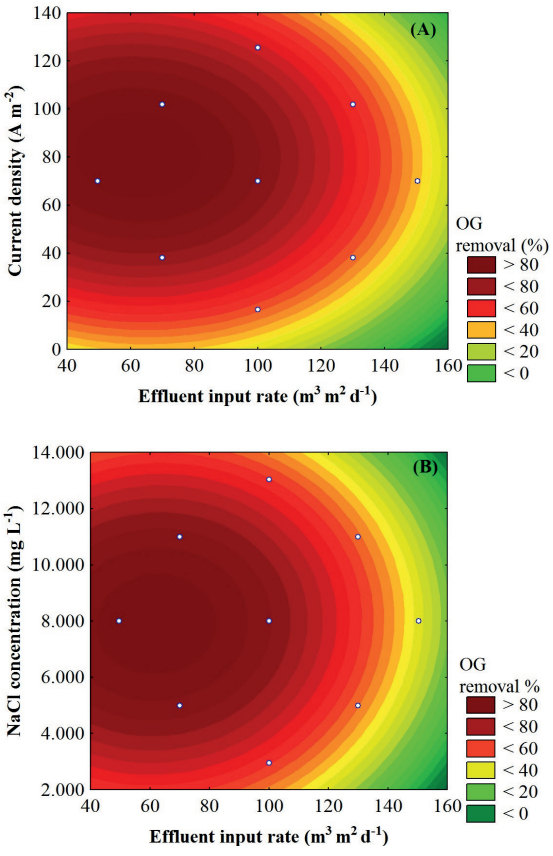

Fig. 4. Contour plots for evaluation of the OG removal according to the effluent input rate and current density (A) and according to the effluent input rate and the concentration of NaCl (B).

3.2. Results of the optimization of the electrolysis time in function of the removal of OG from the study water

For the electroflotation process under optimal operating conditions: effluent input rate of 62 $m^3 m^{-2} d^{-1}$, current density of 80 A m^{-2} and the concentration of NaCl of 7,900 mg L^{-1}, satisfactory results were obtained by the OG removal of the study water.

The OG values of the study water after 180 min of treatment through the electroflotation process can be observed in Fig. 5.

The influence of time on the electroflotation process, which was characterized by two regions, is observed in Fig. 5. The first region (0–30 min) describes the electroflotation of the oil emulsion, where 89% removal of the initial OG concentration occurred. And the second region (30–180 min) in which the electrochemical destruction of water-soluble organic compounds occurred or the removal of oil emulsions smaller than 2.5 μm which are difficult to remove through the electrochemical process. Therefore, from 30 min of electrolysis, a linear tendency of the experimental points of removal is observed. With 180 min of electrolysis, there was a removal greater than 94% of the initial OG.

Mansour and Chalbi [19] reported that, in general, emulsified oil droplets have a range of sizes, and since the larger droplets are removed, the efficiency process was slower. Consequently, in theory, the smaller droplets need a longer time to be removed. Markhasin et al. [20] stated that oil particles smaller than 2.5 μm cannot be removed from the effluent by electroflotation, unless such particles become larger.

According to the results observed in the optimization of the electrolysis time in function of the removal of OG from the study water, it was observed that with only 30 min of electrolysis, 1/6 of the total electrolysis time studied, the OG concentration reached 28.69 mg L^{-1}, an amount lower than the monthly average permitted by Brazilian law [21], which

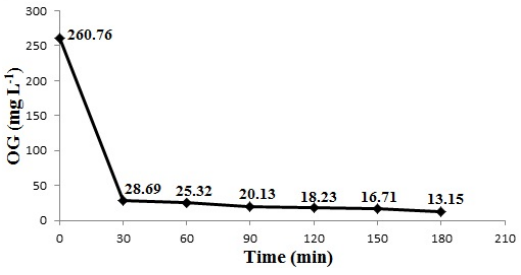

Fig. 5. OG reduction of the study water due to the electrolysis time.

is 29 mg L^{-1}, for the disposal of treated effluent in saline waters, such as the ocean.

Thus, when the final destination of effluent is disposed in the ocean, the treatment of the effluent can be done in just 30 min, with the electrolytic reactor operating under the experimental conditions of greater efficiency ("good" values obtained from the experimental design). With shorter treatment time, therefore, costs are reduced and it becomes possible to treat larger volumes of effluent per day, with the reactor operating in a "good" condition.

Finally, it was observed that all results suggest that application of electroflotation process with the use of DSA® type electrodes proved to be an attractive option, as well as providing a wide application without the deterioration of electrodes [4], promoting a good removal of organic pollutants (OG) present in the petrochemical industrial effluents and significantly reduces treatment time.

4. Conclusions

Using the experimental design as a tool to assess the effects of the independent variables and their interactions in the OG removal rate for produced water from simulated oil, it was possible to establish "optimized" operating conditions in order to have a higher rate of OG removal.

From the evaluation of the "optimized" operating conditions, there may be a study of the OG removal of produced water from simulated oil as a function of electrolysis time, thus optimizing the reaction time, which makes it possible to treat larger volumes of effluent per day when applied to the reality of a petrochemical industry.

According to the statistical analysis, the higher efficiency of the electroflotation process of OG removal is achieved in the following operating conditions of the electrochemical reactor: the effluent input rate of 62 $m^3\ m^{-2}\ d^{-1}$, current density 80 A m^{-2} and the concentration of NaCl of 7,900 mg L^{-1}.

Under these conditions, it was observed that with 30 min of electrolysis, it gave a removal of 89% of the initial OG, bringing the effluent to a concentration of 28.69 mg L^{-1}. With 180 min of electrolysis, there was a removal of over 94% (13.15 mg L^{-1}) of the initial OG.

It can be concluded, therefore, that treatment of produced water from simulated oil through the electroflotation process using DSA® type electrodes was effective in OG removal, leading to acceptable values by legislation, demonstrating and encouraging the applicability of the electrochemical process in treatment of produced water from oil.

Acknowledgment

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