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Optimization of electrocoagulation operating parameters and reactor design for zinc removal: application to industrial Tunisian wastewater

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ABSTRACT

This study was carried out to investigate the effect of the electrocoagulation reactor design parameters in the removal of zinc from water. In order to optimize experimental electrocoagulation reactor parameters such as inter electrode distance (d_{ie}), electrode connection mode, surface-area-to-volume ratio (S/V), and the initial temperature of the solution (T), many electrocoagulation tests were performed using aluminum electrodes. The obtained experimental results showed that optimal zinc removal was achieved with a distance between electrodes of 0.5 cm, a bipolar connection mode, the surface-area-to-volume ratio (S/V) of 13.6 m^{-1}, and for an initial temperature (T) of 50°C. The application of these results on the treatment of a sulfuric acid and superphosphate manufacturing industry wastewater made it possible to achieve a total zinc removal in five minutes of electrolysis time.

Keywords: Wastewater treatment; Electrocoagulation; Zinc removal; Reactor design

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1. Introduction

Generally, the discharge of industrial effluents to the ecosystem has a harmful effect on the environmental living organisms [1]. Zinc is one of the most essential elements in humans' lives naturally present in water. Zinc is not classified among heavy metals detrimental to health and its potential effect is considered to be merely limited to imparting undesirable taste to drinking water. However, according to the World Health Organization, if the intake of zinc exceeds 5 mg L^{-1} [2], it becomes harmful and can also damage human health as it causes health problems such as stomach cramps, cell damage, nausea, and vomiting [3]. With environmental regulations in Tunisia becoming stricter, there is an urgent need to install an efficient treatment equipment to treat and remove heavy metals from industrial wastewater. Several techniques has been studied to remove heavy metals from industrial effluents, the most widely used

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ones are adsorption [4,5], photo catalysis [6], electrodialysis/electrode ionization [7], chemical precipitation [8], ion exchange [9], reverse osmosis [10], coagulation–flocculation [11], treatment by electrocoagulation [12], etc.

Electrocoagulation is a process that generates metallic ions by electrochemical dissolution of a soluble sacrificial anode. The application of the electrocoagulation process on the treatment of wastewater does not require any addition of chemical salts [13]. This process promotes high removal efficiency for many types of pollutants in shorter treatment time with a comparatively very low operating cost. Electrocoagulation has been tested on many types of effluents. Existing studies show that the electrocoagulation process is very effective for the treatment of many types of water such as: the treatment of wastewater charged with heavy metals [14], the defloration of natural water [15], the treatment of water flowing from tobacco factory [16], suspended solids, and oil and fat in restaurant [17], and surface water [18].

Despite the fact that electrocoagulation technology has been utilized for decades, the existing literature has at best paid little attention to the need to provide a systematic approach to the optimization of the electrocoagulation reactor design parameters. The present study was designed to address this gap in the literature on the implementation of electrocoagulation in the treatment of industrial wastewater. The aim of this work is to optimize the electrocoagulation reactor design parameters in order to take the electrocoagulation efficiency to the highest level possible. Optimization of the electrocoagulation reactor design parameter using aluminum electrodes was carried out in synthetic water using a current density of 3.68 mA cm^{-2}, an initial pH of 7, and a sodium chloride dose of 2 g L^{-1} corresponding to a conductivity of 4.28 mS cm^{-1}. Different parameters such as the surface-area-to-volume ratio, the distance between electrodes, the state of the aluminum plates, the stirring speed, the electrodes configuration mode, and the initial temperature of water were studied in detail. This study also investigated the application of electrocoagulation to a sulfuric acid and superphosphate manufacturing wastewater industry at the optimized variable.

2. Experimental

2.1. Materials

All the chemicals used in the study were analytically pure. The electrolyte used in the study was sodium chloride (NaCl). The pH was adjusted using hydrochloric acid and/or sodium hydroxide solutions of 0.1 M concentration before the electrolysis started. These two solutions were prepared using hydrochloric acid (37%) and sodium hydroxide (98%). Aluminum electrodes, 99% of which was pure, were used. The wastewater under investigation was collected from a Tunisian sulfuric acid and superphosphate manufacturing industry. Sample filtration, collection, and preservation were carried out immediately in the industry laboratory of quality control of TCG, Gafsa, Tunisia. The characterization and the treatment of the effluent were performed at the URTDE, Tunis, Tunisia, and university laboratory.

2.2. Experimental procedure

The electrochemical reactor (glass beaker) having working volume of 1 L was used to treat the synthetic water and the industrial wastewater with aluminum sheets (250 mm × 80 mm × 2 mm) as an electrode. The effective surface area of each electrode was 58 cm^2 corresponding to an immersed aluminum sheet of (85 mm × 80 mm × 2 mm) as an electrode. The anode–cathode distance (d_{ie}) was varied from 0.5 to 2 cm. The assembly was connected to a regulated direct current (DC) AFX 2930 SB DC power supply to fix the required current density. The current intensity between the electrodes and the resulting voltage was measured and controlled using the generator. Distilled–dematerialized water was used to prepare the synthetic water under study then, if required, the pH was adjusted using hydrochloric acid and/or sodium hydroxide solutions of 0.1 M concentration before the electrolysis started. The stirring speed was fixed at the required value using a digital magnetic stirrer. The temperature is measured at the beginning and as a function of time for each experiment using the pH meter pH Cyber Scan 510 (WDW, Germany). A schematic diagram of the electrocoagulation reactor is shown in Fig. 1.

In each test 1 L of synthetic water or wastewater was put into the reactor and all the tests were performed for an electrolysis time of 60 min. Every five minutes, a sample was extracted from the solution. The collected samples were filtered and analyzed in order determine zinc concentration. The percentage of zinc removal efficiency (P) was calculated by Eq. (1).

$$P = \frac{C_i - C_o}{C_o} \times 100 \tag{1}$$

C_i and C_o, are the initial and final concentrations (mg L^{-1}) of zinc in the solution, respectively.

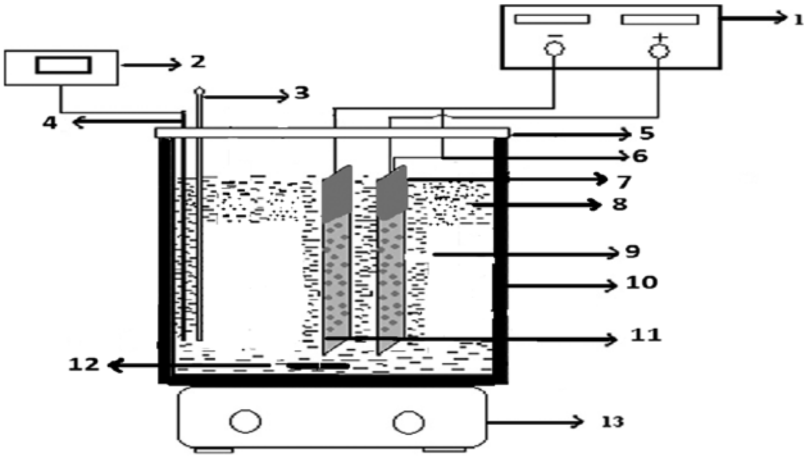

Fig. 1. Laboratory scale cell assembly. (1) DC power supply; (2) pH meter; (3) thermostat; (4) pH sensor; (5) support of electrochemical cell; (6) conductive cable; (7) anode; (8) flocculent; (9) electrolyte solution; (10) baker glass (Pyrex); (11) cathodes; (12) magnet bar; and (13) magnetic stirrer.

2.3. *Analytical techniques*

The conductivity and pH of the solution were measured during the experiments using a conductivity meter Jenway 4510 (Ω Metrohm) and a pH meter pH Cyber Scan 510 (WDW, Germany). An atomic absorption spectroscopy with flame (AASF) method with background correction was used to determine zinc concentration. This method was the most feasible one on the higher concentration measurement of zinc ions [19]. For this reason, AASF Analytik Jena Nova 400 was used.

3. Results and discussion

3.1. *Effect of the variation of the surface-area-to-volume ratio*

The surface-area-to-volume ratio of the treated solution (S/V) is an important reactor design parameter in electrocoagulation. It has been reported that an increase in the S/V ratio causes a decrease in the energy consumption [20]. Hence, it is made necessary to take into account this parameter and determine its effect on the performance of the electrocoagulation process. In order to study the effect of the S/V ratio on the removal efficiency of zinc, several electrocoagulation tests were performed by varying the S/V ratio values from 3.4 to 13.6 m^{-1} keeping constant the volume of the solution of 1 L, the initial temperature at 18℃, the distance between electrodes at 2 cm, the stirring speed at 200 rpm, and using two aluminum electrodes. Fig. 2 shows a schematic view of the electrodes covers.

The results obtained during testing the electrocoagulation studied for values of ratio (S/V) are summarized in Fig. 3. The effect of the variation of the S/V ratio on the energy consumptions is shown in Table 1.

As it is clearly shown in Fig. 3, the higher the rate of surface-to-volume ratio, the more efficient is the

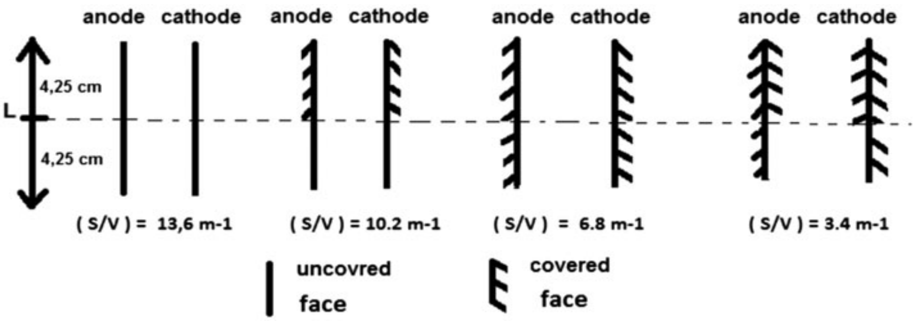

Fig. 2. Schematic view of different electrodes covers in the EC process.

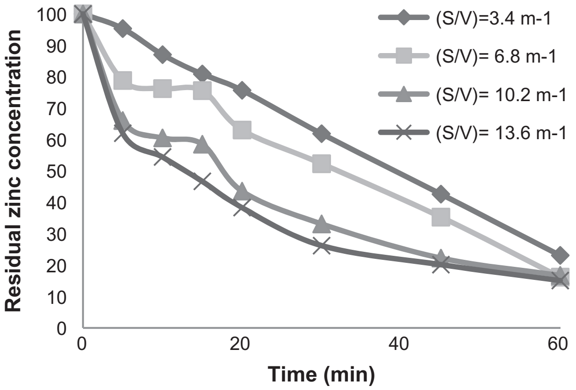

Fig. 3. Variation of zinc concentration as a function of electrocoagulation time for different S/V values ($pH_i = 7$, $J = 3.68\ mA\ cm^{-2}$, $d = 2\ cm$, $NaCl = 0.4\ g\ L^{-1}$ stirring speed = 200 rpm).

Table 1
Effect of the variation of the S/V ratio in the removal efficiency and the energy consumption

S/V (m^{-1})	3.40	6.80	10.20	13.60
% Zn removed	38.13	47.69	66.84	73.76
W ($kWh\ m^{-3}$)	5.12	3.12	2.85	0.87

treatment of water by electrocoagulation. An increase in the S/V ratio from 3.4 to $13.6\ m^{-1}$ allowed an increase in zinc removal efficiency ranging from 38.13 to 73.76% with a large increase in energy consumption ($4.25\ kWh\ m^{-3}$). Thus, an S/V value of $13.6\ m^{-1}$ would be chosen for the tests to minimize the subsequent electrolysis time and achieve maximum removal.

3.2. Choice of electrode configuration

The electrocoagulation efficiency is strongly related to the dissolution of the electrodes which produces a large amount of metal ions [21,22]. This dissolution is reinforced by the increase in the number of aluminum plates which provides a higher active surface of the anodes and causes a significant ohmic drop. According to the literature [23–25], there are two types of electrode configuration (monopolar and bipolar) where the number of the electrodes is varying between 4 and 8. As shown in Fig. 4, with the bipolar configuration, only the electrodes at the extremities are connected to the generator; one as anode and the other as cathode. All intermediate electrodes are used for both anode and cathode but are not directly connected. For the monopolar configuration all the electrodes are connected to the generator as anodes and cathodes one by one.

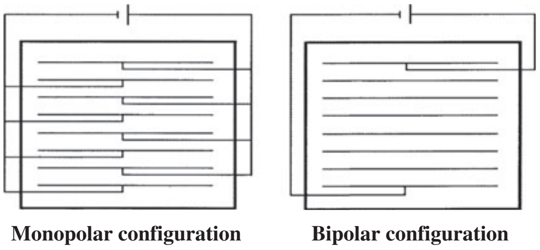

Fig. 4. Electrodes configuration mode.

In order to choose one of the two configurations, two tests were performed using four electrodes, the first with a bipolar configuration and the second with a monopolar configuration. This study was carried out with an initial temperature at 18°C, a distance between electrodes of 2 cm, an initial pH of 7, a stirring speed of 200 rpm, and an S/V ratio of $13.6\ m^{-1}$. The temperature, the pH, the difference in potential between the electrodes, and the final zinc concentration was measured for each sample. Also, energy consumption (W) was calculated for each sample. The results are shown in Table 2.

The obtained results (Fig. 5) were consistent with the hypothesis that increasing the number of electrodes results in removal efficiency. In fact, increasing the number of electrodes from two to four allowed the reduction of zinc concentration to become faster and reach 88% in only 30 min, while 45 min of electrolysis time was required to obtain the same performance in the case of only two electrodes. Using bipolar configuration, the elimination of zinc was more efficient but it was done with high energy consumption, and the difference in removal efficiency was not significant compared to the difference in energy consumption and the treatment cost. The final pH depended on the type of electrodes configuration. As shown in Table 2, after 60 min of electrolysis time, with bipolar configuration the pH increased up to 10.60, whereas with the monopolar configuration the pH increased up to 8.99. This result is in agreement with the hypothesis that the use of soluble anodes causes a change in the pH of the solution during electrocoagulation [26,27]. Then, the monopolar configuration was chosen as a mode of electrode configuration because it presented high removal efficiency with low energy consumption. These results are in agreement with those found in other works [28,29].

Table 2
Variation of residual zinc concentration as a function of time for different electrode configurations ($pH_i = 7$, $d = 2$ cm, NaCl = 0.4 g L^{-1}, stirring speed = 200 rpm)

Configuration type	Initial zinc concentration (mg L^{-1})	Time (min)	U (V)	pH	Final zinc concentration (mg L^{-1})	W (kWh m^{-3})
Bipolar	100	5	9.2	7.00	80.56	0.38
	100	10	8.4	8.10	74.38	0.70
	100	15	8.4	11.73	68.16	1.05
	100	20	8.4	11.40	54.13	1.40
	100	30	8.4	10.95	17.71	2.10
	100	45	8.4	10.80	16.56	3.15
	100	60	8.4	10.60	12.21	4.20
Monopolar	100	5	2.1	7.00	79.41	0.08
	100	10	2.0	8.32	75.1	0.16
	100	15	2.0	8.14	74.88	0.25
	100	20	2.0	8.08	65.46	0.33
	100	30	2.0	8.23	22.66	0.50
	100	45	2.0	8.66	15.13	0.75
	100	60	2.0	8.99	13.06	1.00

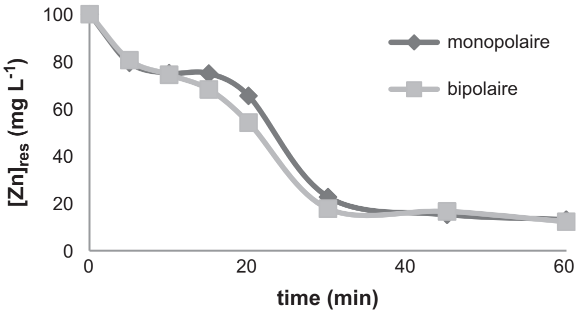

Fig. 5. Variation of zinc concentration as a function of electrocoagulation time ($pH_i = 7$, $J = 3.68$ mA cm^{-2}, $d = 2$ cm, NaCl = 0.4 g L^{-1}, $S/V = 13.6$ m^{-1}, stirring speed = 200 rpm).

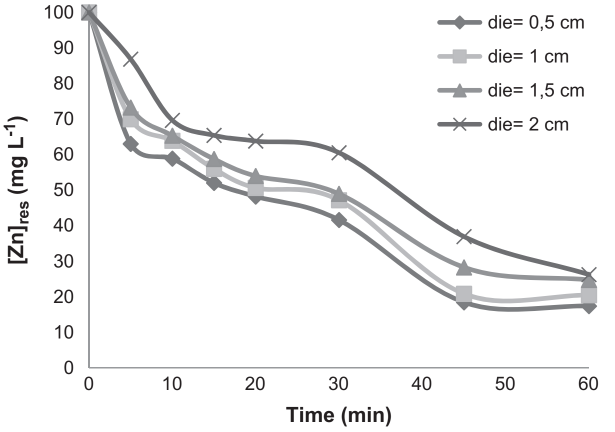

Fig. 6. Variation of the residual zinc concentration as a function of time for different interelectrode distances ($pH_i = 7$, $J = 3.68$ mA cm^{-2}, NaCl = 0.4 g L^{-1}, $S/V = 13.6$ m^{-1}, stirring speed = 200 rpm, $T = 18$℃).

3.3. *Effect of inter electrode distance*

To highlight the effect of the inter electrodes distance (cathode–anode distance), various electrocoagulation tests were performed by varying the distance between electrodes in the range 0.5–2 cm and keeping constant the initial temperature at 18℃, the S/V ratio at 13.6 m^{-1}, the stirring speed at 200 rpm, and using two aluminum electrodes. The variation of the residual zinc concentration as a function of time for different inter electrode distances is shown in Fig. 6.

As illustrated in Fig. 6, the percentages of zinc removal decreased with the increase in inter electrodes distance. It has been agreed that the anodic oxidation starts with the application of the potential to electrodes. As the electrolysis time progressed, a fine gelatinous film on the active surface of the anode was formed. The formation of this film increased with the increase in the inter electrodes distance. As a result, the resistance between the electrodes increased with the increase in the inter electrode distance, the resistance to mass transfer became larger, the kinetics of charge transfer and the rate of oxidation of the aluminum became slower and consequently a lower removal efficiency at higher inter electrode distance [30]. Hence, it was beneficial to use a short inter electrode distance of 0.5 cm to minimize energy consumption and to increase the zinc removal efficiency.

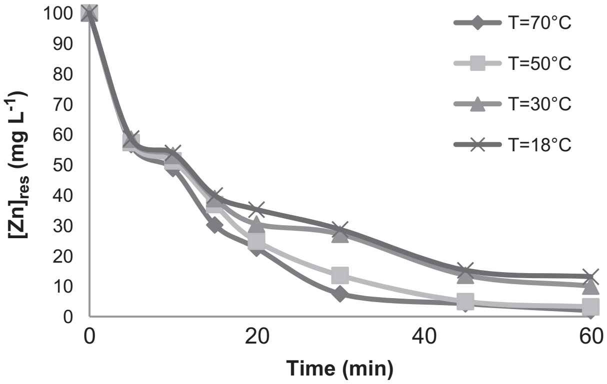

Fig. 7. Variation of zinc concentration as a function of electrocoagulation time for different initial temperatures ($pH_i = 7$, $J = 3.68$ mA cm^{-2}, $d = 2$ cm, $S/V = 13.6$ m^{-1}, NaCl = 0.4 g L^{-1}, $S/V = 13.6$ m^{-1}, stirring speed = 200 rpm).

3.4. Effect of the initial temperature of the water

Initial water temperature is an important parameter influencing the zinc removal efficiency by electrocoagulation [31,32]. To optimize this parameter in order to remove zinc from water by electrocoagulation process using two aluminum electrodes, several electrocoagulation tests were performed by varying the initial temperature from 18 to 70℃ and keeping constant distance between electrodes of 2 cm, S/V ratio of 13.6 m^{-1}, and stirring speed of 200 rpm. The results are presented in Fig. 7.

The performance of zinc removal by electrocoagulation was significantly improved with the increase in temperature. In fact, due to the increase in the initial temperature, there was an increase in the mass transfers and improved the kinetics of particle collision. But, with too high temperature, a change in the

Table 3
Variation of zinc removal efficiency as a function of time for different stirring speeds

Stirring speed (RPM)	$Zn_{initial}$ (mg L^{-1})	t(EC) (min)	U (V)	pH	I (A)	Zn_{final} (mg L^{-1})	W (KWh m^{-3})	Cost of treatment (DTN m^{-3})
600	100	5	3.5	6.03	0.5	100	0.145	0.019
	100	10	3.5	6.12	0.5	58.88	0.291	0.039
	100	15	3.5	6.29	0.5	47.86	0.437	0.058
	100	20	3.5	6.55	0.5	42.04	0.583	0.078
	100	30	3.5	6.03	0.5	32.03	0.875	0.117
	100	45	3.5	6.46	0.5	22.85	1.312	0.175
	100	60	3.5	7.20	0.5	18.74	1.750	0.234
450	100	5	3.5	5.94	0.5	100	0.145	0.019
	100	10	3.5	5.97	0.5	15.32	0.291	0.039
	100	15	3.5	6.00	0.5	11.04	0.4375	0.058
	100	20	3.5	6.26	0.5	8.92	0.583	0.078
	100	30	3.5	6.43	0.5	6.62	0.875	0.117
	100	45	3.5	6.90	0.5	5.02	1.312	0.176
	100	60	3.5	7.75	0.5	3.95	1.750	0.234
300	100	5	3.5	6.39	0.5	100	0.145	0.019
	100	10	3.5	6.69	0.5	10.92	0.291	0.039
	100	15	3.5	6.73	0.5	6.76	0.437	0.058
	100	20	3.5	6.78	0.5	6.15	0.583	0.078
	100	30	3.5	6.94	0.5	5.54	0.875	0.117
	100	45	3.5	7.83	0.5	4.24	1.312	0.175
	100	60	3.5	7.69	0.5	0.58	1.750	0.234
0	100	5	3.2	6.03	0.5	100	0.133	0.017
	100	10	3.3	6.12	0.5	20.63	0.275	0.036
	100	15	3.4	6.29	0.5	18.44	0.425	0.057
	100	20	3.4	6.55	0.5	15.92	0.566	0.075
	100	30	3.4	7.05	0.5	10.62	0.850	0.114
	100	45	3.4	7.46	0.5	8.023	1.275	0.171
	100	60	3.4	7.70	0.5	6.754	1.700	0.228

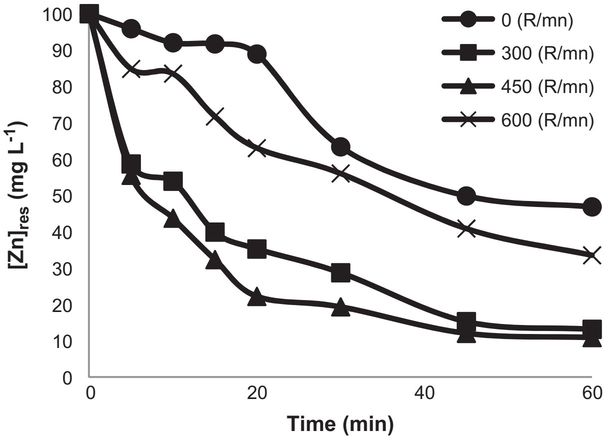

Fig. 8. Variation of zinc concentration as a function of electrocoagulation time for different agitation speeds (pH$_i$ = 7, J = 3.68 mA cm^{-2}, d = 2 cm, S/V = 13.6 m^{-1}, NaCl = 0.4 g L^{-1}, S/V = 13.6 m^{-1}, T = 17℃).

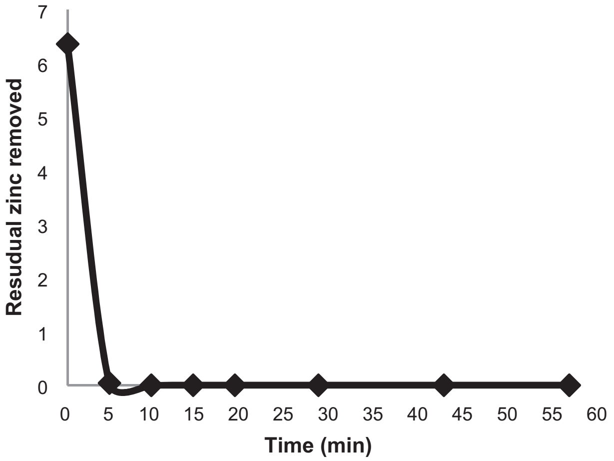

Fig. 9. Variation of residual zinc concentration as a function of electrocoagulation time (pH$_i$ = 7, J = 3.68 mA cm^{-2}, d = 0,5 cm, S/V = 13.6 m^{-1}, NaCl = 0.4 g L^{-1}, S/V = 13.6 m^{-1}, stirring speed = 200 rpm, T = 50℃).

interface gas–liquid occurred, which was not desirable for the removal of pollutants [33]. In addition, a medium temperature implied a production of larger hydrogen bubbles, which reduced the adhesion of suspended particles [34]. Accordingly, the solution had to be heated with a medium temperature just before the electrocoagulation test started. As shown in Fig. 7, a maximum percentage of zinc elimination of 93.44% was achieved for a medium temperature of 50℃ and electrolysis time of 30 min. Then, the temperature of 50℃ was chosen as an optimum initial electrocoagulation temperature because it presented high removal efficiency and a short treatment time.

3.5. Effect of stirring speed

The effect of stirring speed was investigated to improve the influence of this parameter on the removal of zinc by electrocoagulation. In order to study the effect of the stirring speed, several electrocoagulation tests were performed at different stirring speed of 0, 300, 450, and 600 rpm and keeping constant the distance between electrode at 2 cm, the S/V ratio at 13.6 m^{-1}, the initial temperature at 18℃, and using two aluminum electrodes. The temperature, the difference in potential between the two electrodes, the pH, and the final concentration of zinc was measured for each sample. The energy consumption and the cost of treatment were calculated for each sample. The results are shown in Table 3.

As shown in Fig. 8, the elimination of zinc was much faster for a high agitation speed. Without agitation the removal of zinc was low and the percentage of removal did not exceed 70% for an electrolysis time of 30 min. For an agitation speed of 450 rpm, the elimination percentage reached 95% after only 30 min. However, there was a slight decrease in removal efficiency when the agitation reached 600 rpm. This could be explained by the breaking of coagulant electrocoagulation under the effect of strong agitation. It is important to note that the amount of sludge decanted increased by increasing the stirring speed. With a high agitation speed, a greater amount of flocs was formed in the electrocoagulation cell reactor and the treated solution became turbid. For a stirring speed of zero, the solution appeared homogeneous and not turbid.

Table 4
Characteristics of the effluent before and after the electrocoagulation treatment

	pH	Conductivity (mS cm^{-1})	P_2O_5 (%)	MS (%)	Zn^{2+} (%)	Ca^{2+} (%)	Mg^{2+} (%)	k^+ (%)	Na^+ (%)
Before the treatment	2. 08	1. 176	0.49	0.025	0. 621	0.17	0.05	0.024	0.57
After the treatment	10.9	1. 158	0.13	0.034	0	0.07	0	0	0.13

The flocs were very stable at top of the reactor and the zinc residual concentration decreased gradually with electrocoagulation time.

4. Treatment of the case-study Tunisian Chemical Group wastewater

The Tunisian Chemical Group in Gafsa, Tunisia, is a sulfuric acid and superphosphate manufacturing industry. This industry is one of the most water-dependent industries which consume 400 m^3 of water per day.

The electrocoagulation process was applied in the treatment of sulfuric acid and superphosphate manufacturing industry wastewater. The characteristics of the effluent before and after the electrocoagulation treatment are shown in Table 4.

Before starting the treatment of the industrial effluent, the pH of the sample was adjusted to 7 using NaOH solution. Also, the wastewater solution was filtrated using a double filter paper in order to remove the salt precipitation formed with the adjustment of pH. The treatment of the industrial wastewater was carried out using the optimal conditions found with synthetic solution such as an inter electrode distance of 0.5 cm, a bipolar connection mode, a surface-area-to-volume ratio (S/V) of 13.6 m^{-1}, an initial pH of 7, and an initial temperature of 50°C. The variation of the residual zinc concentrations as a function of time is shown in Fig. 9.

The testing of zinc removal from industrial wastewater showed that the removal by electrocoagulation using aluminum electrodes was very effective; it reached 100% at the first 5 min of electrolysis with a low power consumption of only 1.6 kWh m^{-3}. The treated wastewater solution became very clean.

5. Conclusion

This study was carried out in order to optimize the effect of electrocoagulation reactor design parameters such as inter electrode distance (d_{ie}), electrode connection mode, surface-area-to-volume ratio (S/V), and the initial temperature of the solution (T) in the removal of zinc from water. The obtained high Zn(II) removal from aqueous synthetic solutions was achieved for an inter electrode distance of 0.5 cm, a bipolar connection mode, a surface-area-to-volume ratio (S/V) of 13.6 m^{-1}, and an initial temperature of 50°C. The experimental results showed that an electrocoagulation time of 30 min was sufficient to achieve a high removal percentage up to 93.66%. These operating conditions could simultaneously achieve a good flotation, stability of flocs and thus efficient removal in a relatively short reaction time. The application of this result on the treatment of the wastewater discharged by Tunisian Chemical Group in the region of Gafsa was carried out. This application proved that the treatment of the industrial wastewater by electrocoagulation using aluminum electrodes was highly effective; a total removal of zinc was achieved in only 5 min.

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