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Full Paper

Parametric and Kinetic Studies for The Defluoridation of Synthetic Fluoride Water by Electrocoagulation using Iron and Copper Electrodes

Yacouba Sanou,[1,2,*] Moudassirou Sedou,[2] Seyf-Laye Alfa-Sika Mande,[2,3,4] and Samuel Paré[1]

[1]*Laboratory of Analytical, Environmental and Bio-Organic Chemistry, University Joseph KI-ZERBO, UFR/SEA, 03 BP 7021 Ouagadougou 03, Burkina Faso*
[2]*Laboratory of Water Resources and Environmental Engineering, University of Kara, Faculty of Sciences and Technics, B.P. 404, Kara-Togo*
[3]*Laboratory of Applied Hydrology and Environment, University of Lomé, BP. 1515, Togo*
[4]*Beijing Key Laboratory of Water Resources and Environmental Engineering, China University of Geosciences (Beijing), Beijing 100083, P.R. China*

*Corresponding Author, Tel.: +22672191530
E-Mail: prosper_yacson@yahoo.fr

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Abstract- The present work has been focused on the removal of fluoride from aqueous solutions through the electrocoagulation process. This work aimed to assess the efficiency of electrodes and electrocoagulation process in fluoride removal from aqueous solutions. Batch experiments were carried out using monopolar Iron and Copper electrodes as anode and cathode. Fluoride removal were influenced by operating variables such as current density, nature of electrodes, initial fluoride concentration and NaCl amount which have significant effects on removal efficiency. Indeed, the increase of current density from 0.18 to 27.8 mA/cm^2, fluoride removal efficiency increased from 61 to 98% using Fe electrodes while 57 to 63% using Cu electrodes during the electrolysis time indicating the high capacity of Fe electrodes for fluoride removal. The addition of NaCl solution increased the fluoride removal efficiency from 65% to 70% using Fe electrodes. Kinetics modelling revealed that Fluoride was removed on the surface of Fe electrodes following a pseudo-second order kinetic while pseudo-first order kinetic using Cu electrodes. Maximum energy consumption was 70 kWh/m^3 with water containing 0.5 g/L of NaCl while an energy of 45 kWh/m^3 for water without NaCl. Consequently, electrocoagulation technology is a promising method for defluoridation of water using iron electrodes.

Keywords- Electrocoagulation; Fluoride; Removal efficiency; Water treatment

1. INTRODUCTION

Nowadays, the contamination of Fluoride in water is a worldwide issue because of health effects such as dental fluorosis, skeletal fluorosis due to high fluoride concentration in drinking water [1]. High fluoride in water could be due to volcanic ash, fluoride rich minerals, fly ash and fertilizer plant wastewater, etc. [2]. Literature revealed that a high consumption of fluoride can lead to diseases such as asosteoporosis, thyroid disorder, neurological damage, and fluorosis of skeleton, cancer, infertility in women, Alzheimer's syndrome [3-5]. To prevent the fluoride pollution, World Health Organization (WHO) fixed to 1.5 mg/L the maximum value of fluoride concentration in drinking water [6]. Various technologies like adsorption, chemical precipitation, reverse osmosis, electrocoagulation, and ion exchange have been developed for fluoride removal from contaminated water [7]. Among them, Electrocoagulation (EC) is an electrochemical approach for defluoridation regarding its advantages such as operational favorability of less sludge generation, high efficiency, fastcoagulation and simple operation [8]. Implementation of EC unit consists of an electrochemical cell which can be a batch or continuous reactor with metal electrodes connected to a Current density (DC) power supply.

Literature indicated that EC technology contributes to a cleaner wastewater, leachate, and groundwater and covers a very broad range of technologies in water treatment [9-11]. The principle of EC is based on the in-situ production of metal ions by progressive dissolution of anode in EC cell receptor under the influence of an electric current. According to Chibani *et al.* [12], the oxidation half-reaction which causes the dissolution of the anode is given in following reactions (1-3):

Anode: $M \rightarrow M^{z+} + z\,e^-$ (1) with M = metal

By combining with the OH^- ions from the cathodic reduction of water (2):

Cathode: $(z-1)\,H_2O + (z-1)\,e^- \rightarrow (z-1)\,OH^- + \frac{z-1}{2}\,H_2$ (2)

$M^{z+} + z\,H_2O \rightarrow M(OH)_z + z\,H^+$ (3)

At the anode, the generated metal cations are due to hydroxo-metal complexes which act as coagulant and promote the formation of flocs by destabilizing the polluting matter and suspended particles [13]. Several metals can be used as electrodes as among them Iron (Fe) and Copper (Cu) are interesting because of their low cost and high valences of their generated cations. This work aims to investigate the removal of fluoride in water using electrocoagulation by optimizing experimental conditions. The effects of operating parameters such as electrolysis time, current density, initial fluoride concentration, nature of electrodes and NaCl amount on fluoride removal efficiency have been studied. In addition, we investigated the kinetic of EC process and energy consumption in fluoride removing.

2. MATERIALS AND METHODS

2.1. Experimental design of electrocoagulation

The experimental setup of electrocoagulation (Figure 1) was given as described Elsewhere [14]. The electrocoagulation unit consisted of 0.4 L electrochemical reactor with Fe/Fe electrodes in the first step and Cu/Cu electrodes in the second step. The distance between electrodes set was 1 cm. The electrochemical reactor (14.7 cm of height) was made of Plexiglas. The electrodes were dimensioned as follow: Iron electrode denoted Fe (15 cm × 4.7 cm × 1 mm), and Copper electrode denoted Cu (15 cm × 4.7 cm × 2 mm) were used as cathode and anode for fluoride removal.

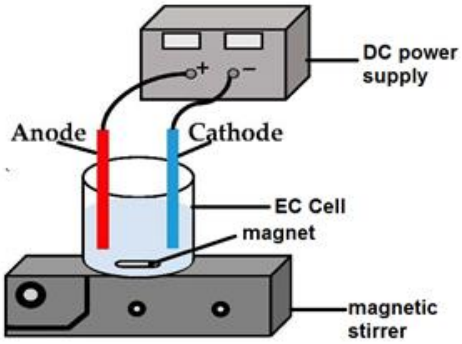

Figure 1. Schematic diagram of EC reactor

2.2. Fluoride removal experiments

Desired concentrations of fluoride solutions were obtained by dissolving a weight of sodium fluoride NaF into distilled water. The conductivity of fluoride solutions was improved by adding sodium chloride (NaCl) solution as electrolyte.

In the electrochemical reactor, two electrodes (anode and cathode) were positioned vertically and parallel to each other. At the beginning of each run, 350 mL of synthetic fluoride solution (11.7 cm of height) was fed in the reactor and 0.5 g/L NaCl solution was added to increase the conductivity of solution. The current intensity was adjusted from 0.75 A, 1 A to 1.5 A, corresponding to a current density varying from 14 to 27.8 mA/cm^2 using a digital DC power supply. Fluoride removal experiments were carried out with iron electrodes and copper electrodes as anode and cathode. The electrolysis time was performed during 90 min and the treated solution was collected each 10 minutes to be analyzed using UV-Vis spectrophotometer (DIV-UV5600). Fluoride concentration was determined using a calibration curve drawn with fluoride standard solutions compared to a blank (distilled solution). Kinetic of fluoride removal was studied by varying the electrolysis time between 5 and 90 min. Kinetic models (pseudo-first and pseudo-second orders) were applied using experimental data (final fluoride concentration, time and adsorption capacity). Experiments were repeated three times and average values were used for calculations in this work.

Removal efficiency (%) was evaluated using the following relation:

$$\text{Removal Efficiency (\%)} = \frac{C_0 - C_f}{C_0} \times 100 \qquad (4)$$

Where C_0 and C_f represent respectively the initial and final fluoride concentrations (mg/L).

Energy consumption (E) expressed in kWh/m^3 was calculated as follow:

$$E = \frac{\mathrm{I \, x \, U \, x \, t}}{V_{water}} \qquad (5)$$

Where **I** is the applied current intensity (A), **U** is the applied voltage (V), **t** is the electrolysis time (h), and V is the volume of treated water (m^3).

2.3. Analysis

All analyses were carried out according to APHA standard methods [15]. Fluoride was analyzed using molecular absorption spectrometry with zirconium and Erichrome-cyanine R at the wavelength of 540 nm.

3. RESULTS AND DISCUSSION

3.1. Effect of electrode nature

The nature of electrodes is an influent parameter which affect significantly the treatment of fluoride contaminated water. In this study, electrodes in iron and copper were tested as indicated by Figure 2 during the overtime of electrolysis (90 min). Most of studies using different electrodes investigate the effect of electrode type [16,17]. Results showed that all treatment yields obtained with Fe electrodes were up to those obtained with Cu electrodes indicating the high efficiency of Fe electrodes (Figure 2). Indeed, Fe and Cu electrodes have achieved the performances (75% and 31%) in fluoride removal using 14 mA/cm^2 during 90 min (Figure 2a). Those low percentages of fluoride removal should be due to the low current density (DC) applying to the EC reactor and probably the lack of sufficient electrolysis time for maximum treatment. When the DC was increased up to 27.8 mA/cm^2, Fe electrodes removed up to 98% of fluoride while Cu electrodes removed 63% during 90 min of treatment (Figure 2b). However, the efficiency of each type of electrodes increases with DC value. The low efficiency of Cu electrodes would be due to their high density comparatively to Fe electrodes. Consequently, Fe electrodes were more efficient to remove fluoride compared to Cu electrodes. This result is in agreement with literature data where Fe and Cu electrodes were used as anode and cathode, respectively and results concluded high efficiency of Fe electrode [16,17].

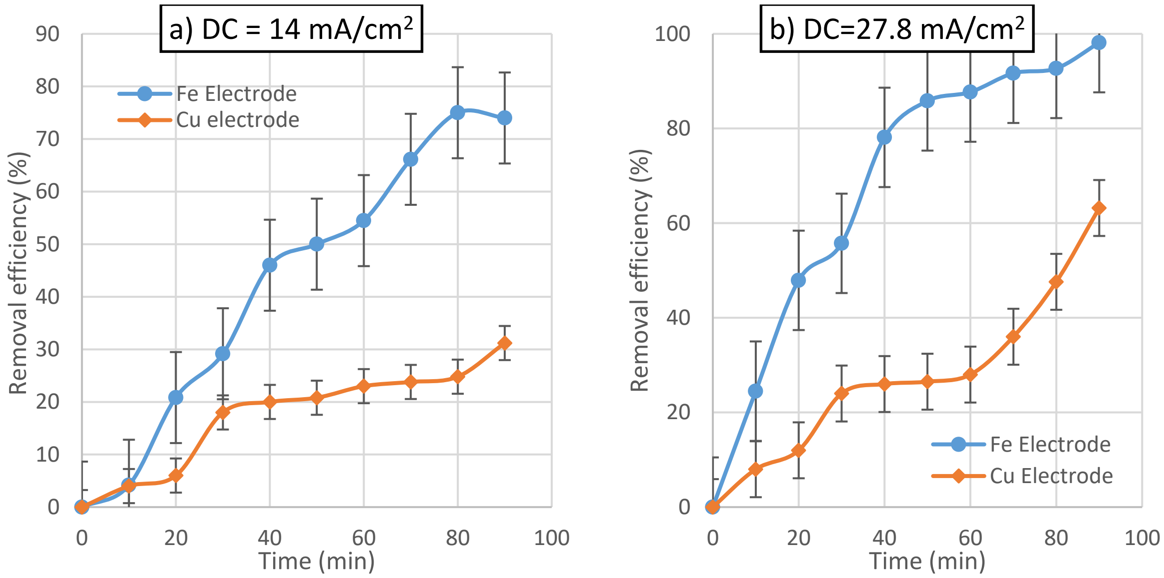

Figure 2. Effect of electrode type on Fluoride removal using Fe and Cu electrodes with C_0 =10 mg/L; pH= 7.68; [NaCl] = 0.5 g/L, and V= 350 mL

3.2. Effect of current density

The current density in electrochemical process is directly linked to the imposed current intensity and the active surface of the electrodes used. In addition, this parameter determines the level of coagulant dosage, the rate of bubble production, size and growth of the floc which may affect the electrocoagulation efficiency [18]. We investigated the influence of current density on fluoride removal process and experiments were performed using three current intensities 0.75A, 1A and 1.5A corresponding to current densities of 14, 18.5 and 27.8 mA/cm^2. Obtained results were represented by Figures 3a and 3b, respectively using Fe and Cu electrodes.

Using Fe electrodes as shown in Figure 3a, fluoride removal efficiency increased up to 74%, 85% and 98% with the increase of current density of 14, 18.5 and 27.8 mA/cm^2 respectively. The increasing of EC efficiency can be explained by the dosage of many coagulants in water due to high current supplied to electrodes. Besides, the increase of current density causes the quick dissolution of the anode which increases the amount of the coagulant in water. All these results are in agreement with literature data [18,19]. Faraday's law indicates that a high current density leads to a good removal of fluoride in water. When Cu electrodes were used during 90 min of electrolysis, 31%, 48% and 63% of fluoride was removed using current densities of 14, 18.5 and 27.8 mA/cm^2, respectively (Figure 3b). We noticed an increase of removal efficiency, probably due to the presence of many flocs in solutions. That is confirmed by a previous study concluding that the more formed flocs increase indicating a good removal by flotation and an increase of current density cause an increase of electrodes consumption which may increase the EC efficiency [20,21].

The fluoride removal efficiency has been beneficial using higher current density of 27.8 mA/cm^2 giving 98.14% of fluoride removed during 90 min of electrolysis. Experimental results showed that a current density 14 mA/cm^2 is not sufficient to treat a water containing initially 10 mg/L of fluoride for 90 min despite the low energy consumption. By comparing EC efficiency in both Figures (3a and 3b), we concluded a high performance of iron electrodes (Fe) for fluoride removal by electrocoagulation.

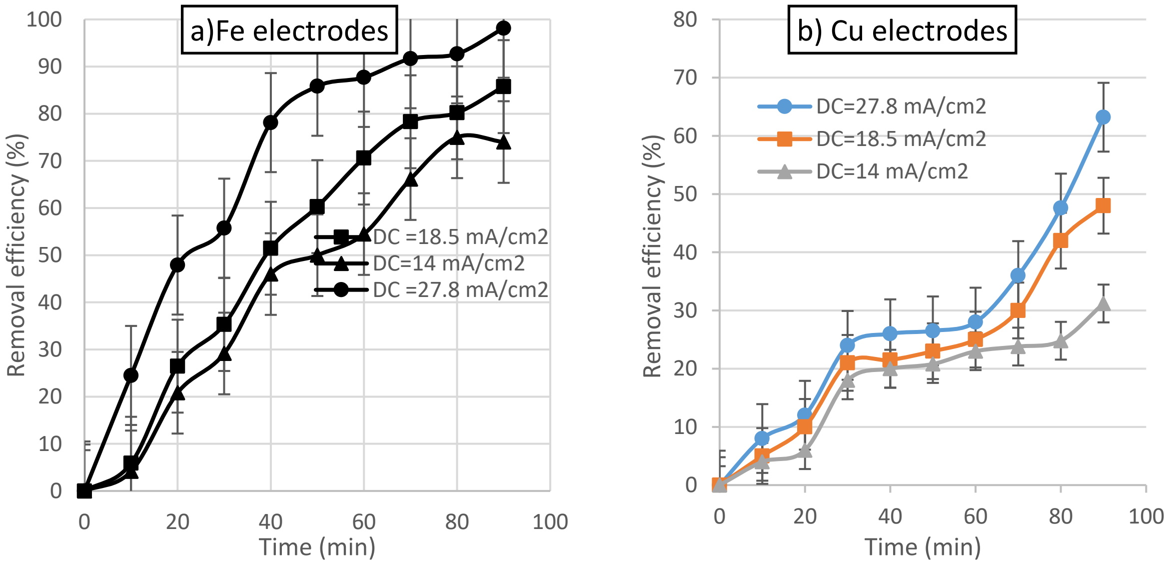

Figure 3. Effect of current density on Fluoride removal by Fe electrodes using pH= 7.68; C_0=10 mg/L 0.5 g/L NaCl, and V=350 mL

3.3. Effect of initial fluoride concentration

In order to study the influence of initial fluoride concentration on EC, initial concentrations of 5, 10 and 15 mg/L were tested by keeping constant other parameters such as initial pH, current density and NaCl concentration. The Figure 4 shows fluoride removal vs current intensity using different initial fluoride amount. The efficiency of electrocoagulation decreases with the increase in the initial fluoride concentration (Figure 4a) indicating the better fluoride removal using low initial fluoride concentrations closed to 5 mg/L. The same trend of curve was observed using different intensities using Cu electrodes (Figure 4b). This result could be due to the capacity of EC reactor, applied DC and available surface of electrodes for fluoride ions removal. Indeed, using low initial concentration, the fluoride removal was more rapid and efficient up 90.5% during 90 min of treatment and the standard limit was quick reached by the first 60 min of electrolysis. While, the standard limit was not reached for initial concentrations 10 and 15 mg/L until up to 90 min. This result can be explained by the theory of dilute solution. Indeed, the diffusion layer at the vicinity of the electrode causes a slower reaction rate in diluted solution, while this diffusion layer has no effect on the rate of diffusion or migration of metal ions in concentrated solution [22-24]. The EC process was more efficient using Fe electrodes comparatively to Cu electrodes as indicated in Figures 4a and 4b.

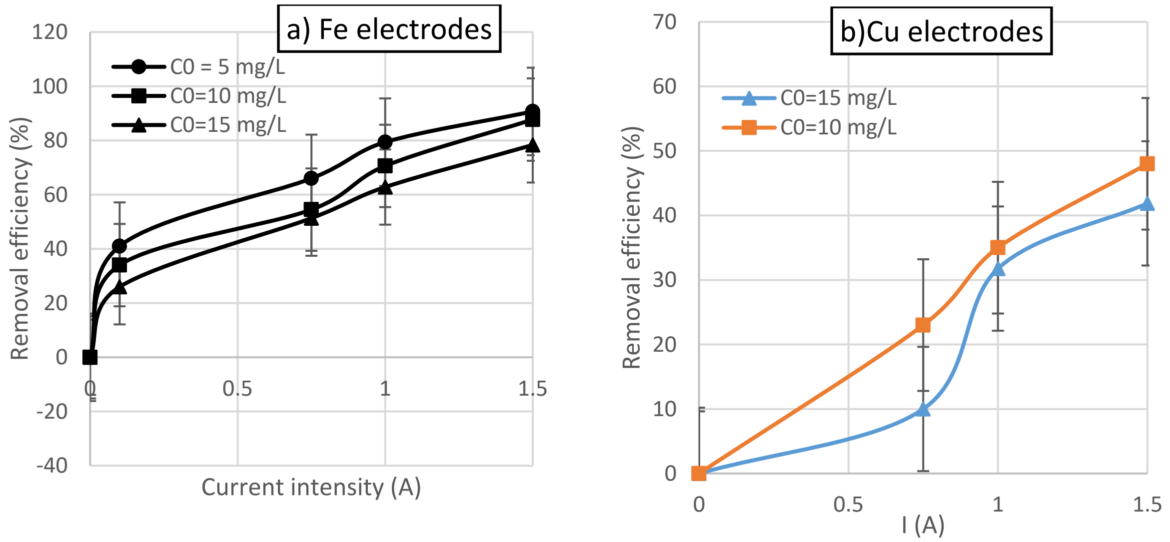

Figure 4. Effect of initial fluoride concentration on removal efficiency using Fe and Cu electrodes with I = 0.01-1.5 A; t = 60 min, pH= 7.68; 0.5 g/L NaCl, and V=350 mL

To confirm the increase of fluoride removal efficiency with initial amount, EC process was applied over 90 min of treatment and the efficiency was calculated each 10 min (Figure 5). It did see clearly the decrease of removal efficiency from 90.5 to 75% when initial fluoride was increased between 5 and 15 mg/L. This decrease of EC efficiency with the increase in fluoride concentrations should be due to the limited surface of Fe electrodes used and small number of produced iron hydroxides $Fe(OH)_2$ which remove fluoride ions by adsorption or precipitation.

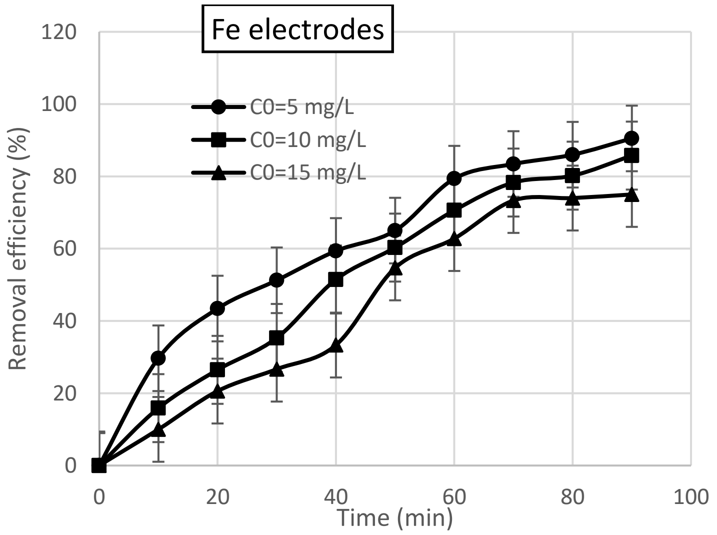

Figure 5. Effect of initial fluoride concentration on EC efficiency using Fe electrodes with DC=18.5 mA/cm^2, pH =7.68; 0.5 g/L NaCl, and V=350 mL

3.4. Effect of NaCl solution

In EC process, the addition of an electrolyte increases the electrical conductivity of the medium and reduces its resistance to ensure a better dissolution of anode. Literature indicated that NaCl is the most effective electrolyte for EC compared to others electrolytes [25,26]. Indeed, Chloride ions significantly reduce the negative effects of other coexisting ions such as CO_3^{2-} and SO_4^{2-} which can form an insulating layer on the electrode surface and increase the resistance of the electrochemical cell [27]. In this study, we added 0.5 g/L of NaCl to the synthetic fluoride solution in order to assess its effect on removal efficiency.

From Figure 6, it was observed an increase of treatment yields when 0.5 g/L NaCl solution is added to the water comparatively to the water without NaCl. High fluoride removals at any time with 0.5 g/L NaCl added can be explained by the corrosion and oxidation. This result was due to the effect of chloride ions on iron electrodes inducing the formation of flocs and inhibiting the formation of a passive film on the surface. According to literature [26,28], the corrosion phenomenon is summarized in the following reactions:

$$Fe + 3\,HCl \rightarrow FeCl_3 + \frac{3}{2}H_2 \qquad (6)$$

$$FeCl_3 + H_2O \rightarrow Fe(OH)_3 + 3\,HCl \qquad (7)$$

Besides, the addition of NaCl increases the flow of current in the EC cell, which leads to an over consumption of electrodes under the corrosion effect and when the chloride concentration increases, defluoridation is inhibited slightly. However, this low influence of can be due to the low NaCl concentration added to water and probably, the increase of NaCl amount will increase rapidly the EC efficiency. All these results are in agreement with literature data [29,30].

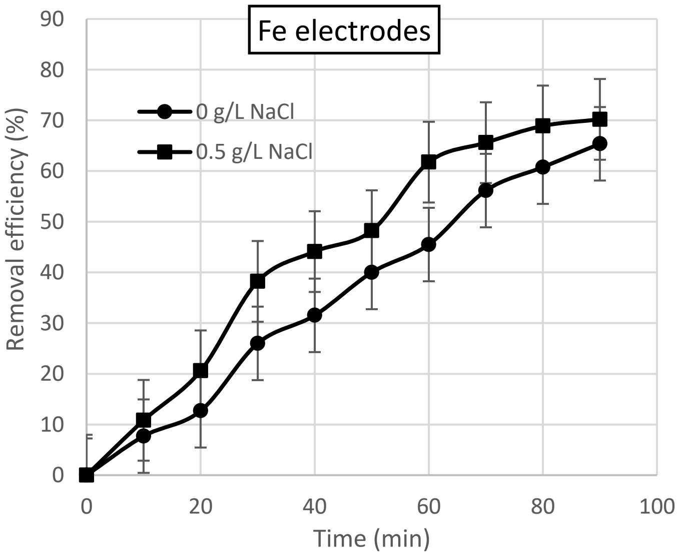

Figure 6. Effect of NaCl concentration on removal efficiency using Fe electrodes with C_0 = 10 mg/L, I=10 mA; pH= 7.68; and V = 350 mL

3.5. Energy consumption

The energy consumption depends on electrolysis time and current intensity or voltage because the increase in electrolysis time leads to increase of energy consumption [14]. Three values of intensities were used for assessment of energy consumption and removal efficiency over the electrolysis time. Experimental results using Fe electrodes with C_0=15 mg/L and 0.5 g/L of NaCl during 90 min of electrolysis are listed in Table 1.

Table 1. Evolution of energy consumption using iron electrodes

Treatment time (min)	Energetically efficiency (kwh/m^3)			Removal efficiency (%)		
	0.75 A	1 A	1.5 A	0.75 A	1 A	1.5 A
10	3	5.33	9.71	10	20	26.67
20	5.43	10.19	17.43	16.67	30	43.33
30	8	15	24.43	26.67	33.66	56.67
40	10.57	18.66	28.85	35	43.33	60
50	13	22.38	36.78	49.33	54.67	68
60	15.43	26.57	42.43	51.33	68	78.33
70	17.74	30.66	55.5	57.33	73.33	90.93
80	20	34.66	62.85	65.33	81.6	98.4
90	21.53	38.57	70	73.33	95	99.99

From Table 1, it was noted an increase of energy consumption and removal efficiency with electrolysis time in the whole time of experiment. Moreover, the energetically efficiency increased with the current intensity from 0.75 to 1.5 A. the same remark is made to EC efficiency which increased with current density. Optimum removal of fluoride (99.99%) was obtaining by applying 27.8 mA/cm^2 (1.5 A) during 90 min of electrochemical treatment.

When the initial fluoride was decreased to 10 mg/L in the water, experimental data were obtained using Fe and Cu electrodes (Figure 7). We remark the energy consumption increased with increasing current intensity and electrolysis time up to 75, 48 and 37 kWh/m^3 when current intensities of 1.5 A, 1 A and 0.75 A were respectively applied for 90 min (Figure 7a). The figure 7b shows that the energy consumption increased with current intensity increasing but the values of energy consumption are lower comparatively to those obtained in Figure 7a, indicating the less efficiency of Cu electrodes. Looking at both two Figures 7a and 7b, energy consumption increases with the increase of treatment time and current intensities or densities indicating that both two parameters affect strongly the energetically efficiency of EC process.

The increase of energy consumption with electrolysis time and current intensity was confirmed by a previous work using the same EC design with Al electrodes [14]. Regarding both two figures (7a and 7b), high energy was consummated using higher current intensity (1.5 A). This result confirms the theoretical hypothesis which predicted the increase of energy consumption with the increase of electrolysis time.

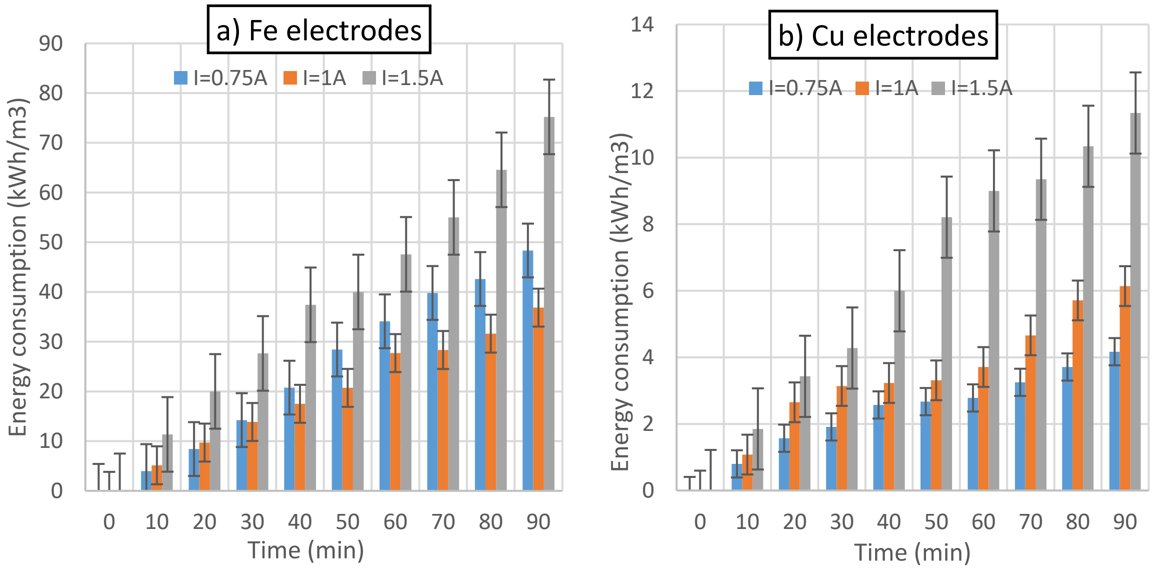

Figure 7. Effect of current intensity and initial fluoride concentration on energy consumption using Fe and Cu electrodes with C_0 =10 mg/L; pH= 7.68; 0.5 g/L NaCl, and V=350 mL

3.6. Kinetic of fluoride removal

3.6.1. Effect of electrolysis time

The electrolysis time is a critical parameter in the defluoridation process by electrocoagulation because its increase causes an increase in formed coagulant species and ultimately more fluoride removal depending to the rate of anodic dissolution [31,32]. Using two electrodes, obtained results (Figure 8a and 8b) indicated a decrease of final fluoride concentration after treatment when the electrolysis time was increased using all initial Fluoride amounts. With Fe electrodes (Figure 8a), only an initial fluoride concentration of 5 mg/L could achieve the WHO guideline value (1.5 mg/L) in drinking water during 70 min of electrolysis. In the whole experiment up to 90 min, the acceptable limit (red line) was not reached by increasing the duration of EC process using Cu electrodes (Figure 8b). That indicates that Copper electrodes are not suitable and efficient for water defluoridation using electrocoagulation process.

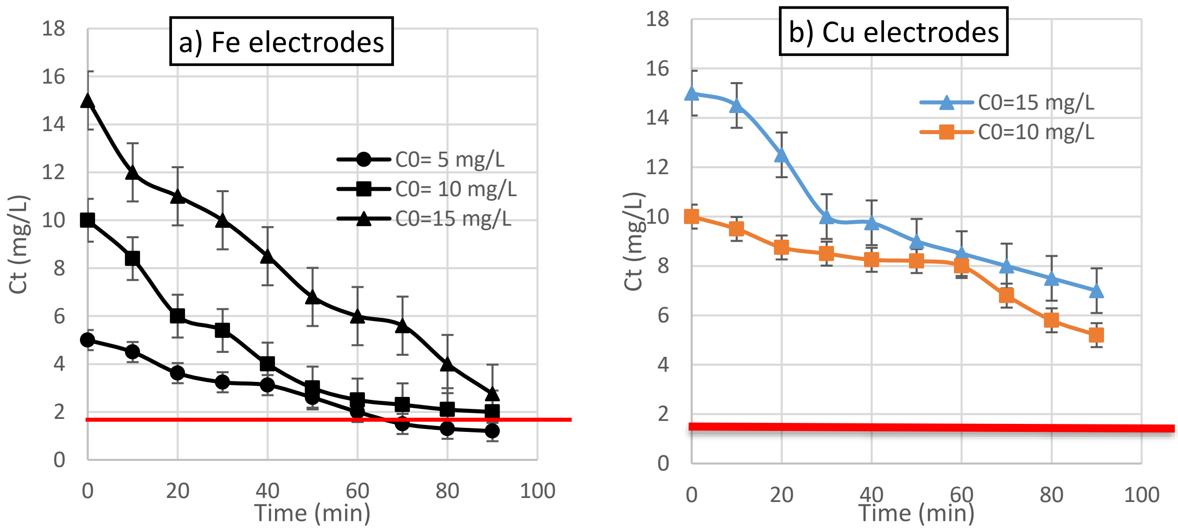

Figure 8. Effect of treatment time on fluoride residual concentration using Fe and Cu electrodes with different initial concentrations, DC=18.5 mA/cm^2 and V=350 mL

Using the EC efficiency data (Figure 9a and 9b), experimental results showed a rapid increase in removal efficiency within the duration of electrolysis using different initial fluorides. Maximum removals of fluoride such as 80.5%, 85.5% and 90.5% were achieved up to 90 min using initial fluoride of 15, 10 and 5 mg/L respectively. This increase of EC efficiency with time can be due to the high amount of adsorbing coagulants in solution as indicated by Drouiche *et al*. [32]. This result is in agreement with conclusions of Ashoori *et al.* [16] which found 93% of fluoride removal efficiency during 45 min of EC treatment. According to Faraday's law, the increase of time was leaded to an increase of the dissolved coagulants from the iron electrodes causing EC efficacy.

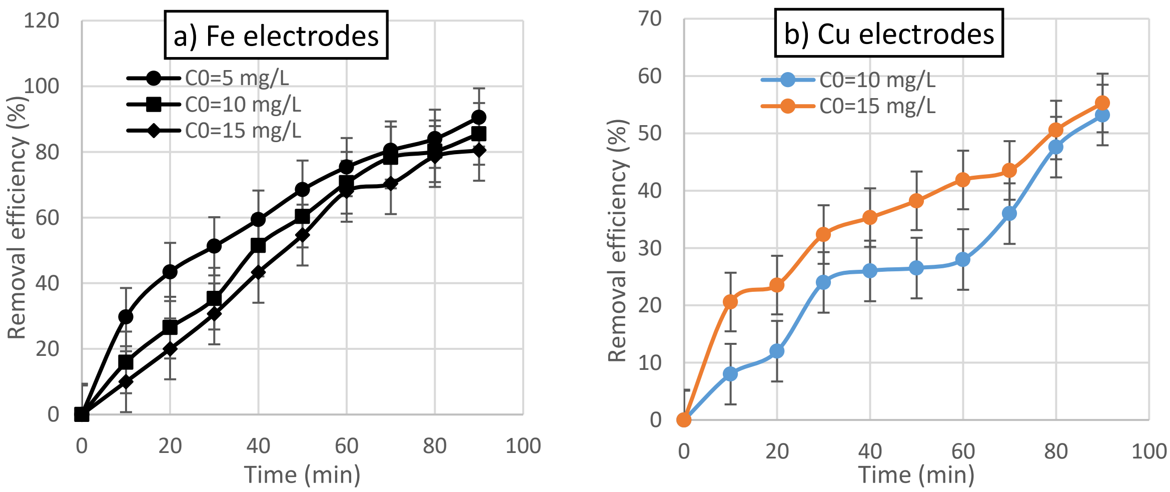

Figure 9. effect of electrolysis time on fluoride removal efficiency using Fe and Cu electrodes with different initial concentrations (5, 10 and 15 mg/L), I=1 A and V=350 mL

High fluoride removal using EC process was obtained under optimal conditions including an electrolysis of 90 min, an initial fluoride concentration of 5 mg/L, 0.5 g/L of NaCl, pH=7.6 and DC= 18.5 mA/cm^2.

3.6.2. Kinetic models

It has been demonstrated that the current density in EC process controls the kinetics of the anodic oxidation reaction and the production of hydrogen bubbles at the cathode [33]. In order to understand the kinetic of fluoride removal, the experimental data were applied to pseudo-first order and pseudo-second order kinetic models. According to literature [34,35], the pseudo-first order model is given by following equation (8):

$$\ln\left(\frac{C_t}{C_0}\right) = -k.t \qquad (8)$$

The plot $ln(Ct/C0) = f(t)$ has given the representation of pseudo-first order (Figure 10) and slopes of the lines indicate the kinetic constants of pseudo-first order model.
The pseudo-second order model is described by Equations 9 and 10.

$$-\frac{dC_t}{dt} = k\,C_t^2 \qquad (9)$$

By integrating this equation, equation 10 was obtained:

$$\frac{1}{Ct} - \frac{1}{C_0} = kt \qquad (10)$$

The plot of $1/Ct = f(t)$ has given the following graph (figure 11) for pseudo-second order model. The values of kinetic constants for two models are listed in Table 2 using Cu electrodes and Table 3 with Fe electrodes.

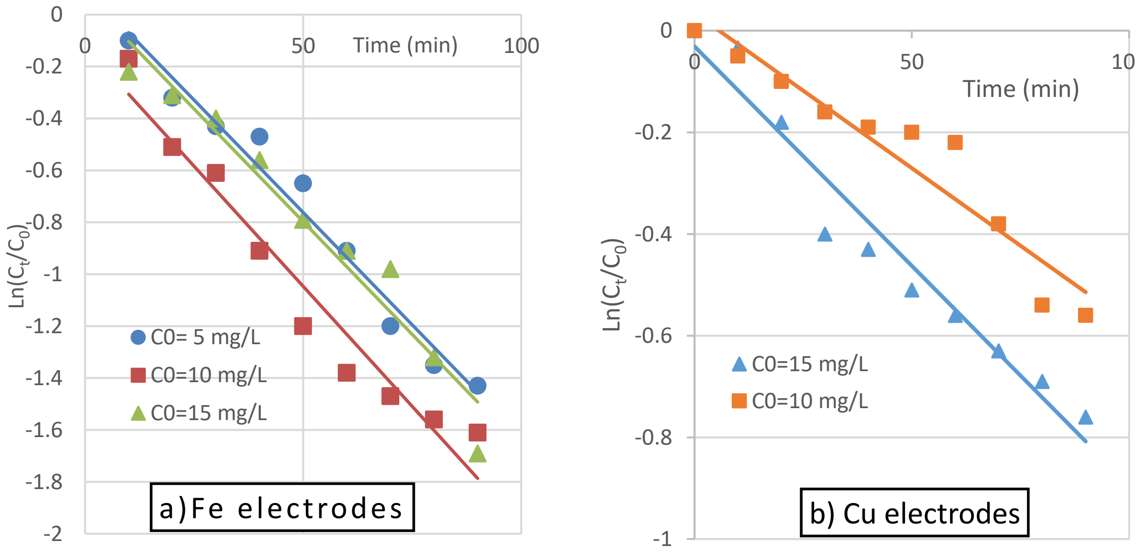

Figure 10. Plots of pseudo-first order kinetic model with I=1 A, C_0= 5-15 mg/L and V=350 mL

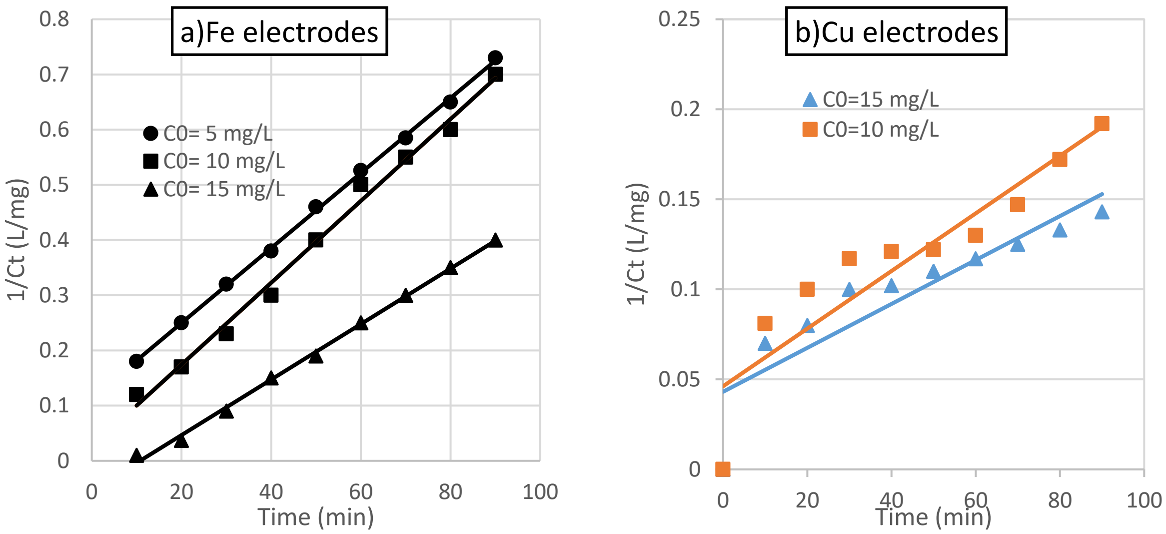

Figure 11. Plots for pseudo-second order kinetic with I =1 A, C_0= 5-15 mg/L and V=350 mL

Table 2. Constants of kinetic models with Cu electrodes

	Pseudo-first order		Pseudo-second order		
Initial fluoride	k (min^{-1})	R^2	k (min^{-1})	C_{0theo} (mg/L)	R^2
5 mg/L	-	-	-	-	-
10 mg/L	6.1 10^{-3}	0.912	1.6 10^{-3}	21.64	0.844
15 mg/L	8.6 10^{-3}	0.954	1.2 10^{-3}	23.2	0.806

(-): not experimentally studied

From Table 2, the values of correlation coefficient obtained with first order model (0.91 and 0.95) were higher than those obtained with second order model. In addition, the kinetic constants increased with initial fluoride amount of 10 and 15 mg/L for first order reaction. We concluded that the fluoride could be removed through EC process by pseudo-first order kinetic.

Table 3. Constants of kinetic models using Fe electrodes

	Pseudo-first order		Pseudo-second order		
Initial fluoride	k (min^{-1})	R^2	k (min^{-1})	C_{0theo} (mg/L)	R^2
5 mg/L	1.73 10^{-2}	0.97	6.8 10^{-3}	8.78	0.999
10 mg/L	1.85 10^{-2}	0.95	7.4 10^{-3}	18.75	0.992
15 mg/L	1.74 10^{-2}	0.95	5 10^{-3}	18.48	0.997

By comparing the correlation coefficients in Table 3, it was clear that higher values of correlation coefficient (R^2) were obtained with pseudo-second order model. Those values closed to unit (1) indicates the applicability of the pseudo-second order model.

We could express that defluoridation using Fe electrode has been occurred following a second order kinetic model as clearly indicated in Figure 11. This result is in agreement with previous studies [35,36]. Inside of electrolysis cell, iron hydroxides $Fe(OH)_2$ produced on the electrodes surface would be responsible of fluoride removal through either adsorption reaction, precipitation or complexation processes.

4. CONCLUSION

Electrocoagulation using iron electrodes has been an efficient process in defluoridation. Results revealed that the fluoride removal efficiency depends on the electrolysis time, current density, initial fluoride concentration, NaCl amount and the type of electrodes used. The increase of electrolysis time and initial fluoride concentration caused an increase of energy consumption in EC reactor. Low concentrations closed to 5 mg/L were recommended for better and less energetically treatment of fluoride using EC process. In this study, iron electrode has been more efficient (98% for 60 min) compared to copper electrodes which were not indicated for Fluoride treatment. Fluoride has been removed following a pseudo-second kinetic using Fe electrodes while the pseudo-first kinetic is fitted using Cu electrodes. The mechanism of fluoride removal should be mainly by adsorption or / and precipitation on iron hydroxides due to the addition of NaCl. In perspectives, Aluminum electrodes will be tested comparatively to iron electrodes in fluoride removal from water using electrocoagulation process in batch or continuous reactor.

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Consent for publication and Competing interests

This manuscript has not been published and is not being considered for publication elsewhere. The submission is approved by all the authors. Authors declare that there is no competing interest.

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