Desalination and Water Treatment
www.deswater.com

doi: 10.5004/dwt.2019.24071

154 (2019) 320–327
June

Electrochemical treatment and reclamation of water used during the rinsing of silicon wafers

B. Palahouane[a], A. Keffous[a], M.W. Naceur[b], M. Hecini[a], O. Bouchelaghem[a], A. Lami[a], N. Drouiche[a,*]

[a]*Centre de Recherche en Technologie des Semi-conducteurs pour l'Energétique (CRTSE), 2, Bd Dr. Frantz Fanon P.O. Box 140, Algiers-7 merveilles, Algeria, emails: nadjibdrouiche@yahoo.fr (N. Drouiche), palahouanebaya@crtse.dz (B. Palahouane), keffousaissa@crtse.dz (A. Keffous), hecinimouna@crtse.dz (M. Hecini), bouchelaghemouahiba@crtse.dz (O. Bouchelaghem), abdoulami@gmail.com (A. Lami)*

[b]*Laboratoire de génie des procédés université Blida, 1. Alger, Algeria, email: wnaceur2002@yahoo.fr*

Received 26 November 2018; Accepted 3 March 2019

ABSTRACT

This study investigates the effect of electrocoagulation process on fluoride removal from cleaning bath. Authors investigated changes in the efficiency and energy consumption of such system with different effective operational parameters, such as current density (13.88–37.03 A/m^2), electrodes number (4, 5 and 6), initial pH (4, 6, 7 and 9) and electrolysis time. The best performing operating conditions were obtained at pH: 7, current density of 27.77 A/m^2, electrodes number of 3. After 50 min of electrocoagulation, 88% of fluoride from an initial concentration of 100 mg/L was removed. Under these conditions, the power requirement was 5.5 kWh/kg fluoride removed. Residual sludge was characterized by using scanning electromicroscopy, ATR-FTIR analyses. According to the results, this process is suggested as an effective and efficient method for removing fluoride deoxidation of silicon wafers.

Keywords: Photovoltaic; Cleanng bath; Hydrofluoric (HF); Electrocoagulation process

1. Introduction

The production of silicon-based photovoltaic solar cells involves a multitude of successive steps that consume a lot of energy, water and toxic chemicals. A 6-inch wafer manufacturing plant that produces 40,000 wafers per month consumes 7.57–11.35 million L of water per month, 18–27 L of water per cm^2 of silicon [1] as a result, generates an equally large amount of effluent that needs to be treated. All products used in the process of developing wafers and cells are found in these wastewater: hydrogen peroxide, nitric acid, sulfuric, hydrofluoric, sludge polishing and even metal residues. Owing to the stringent environmental regulations, this wastewater must be treated and recycled. The wastewater generated during the chemical mechanical polishing steps of the wafers for the production of solar cells are generally classified into two groups of wastewater, those resulting from rinsing and those of concentrated acids. Rinse water has a much lower concentration of chemicals, is treated in situ in the wastewater treatment facility. While concentrated acids are usually collected for external discharge. From the point of view of composition, the level of fluoride content is one of the most important parameter because of its relatively strict discharge limit.

The various studies and researches have demonstrated incontestably that waters rich in fluorides can cause sometimes irreversible diseases in humans such as dental fluorosis or osteofluorosis [2], increasing the risk of fractures, hypothyroidism, acute nephritis and various lesions in the liver and heart (Table 1). The regulation in Algerian law

* Corresponding author.

Table 1
Relationship between fluoride dosage, route of entry and biological effects [3]

Fluoride concentration (mg/L)	Exposure medium	Duration of exposure	Biological manifestation
0.002–1.00	Air and water		Injury to vegetation
			Reduction in dental caries
1.0 mg/L	Water	Long term	Dental fluorosis
2 or more	Water	Long term	Mottled enamel
3–7	Water	Long term	Skeletal fluorosis
7–8	Water	7 years	Osteosclerosis (10%)
Up to 50	Food and water	Long term	Thyroid changes
60–100	Food and water	Long term	Growth retardation
100–120	Food and water	Long term	Kidney changes

sets the maximum concentration of fluoride for industrial effluents as 15 mg/L and for drinking water as 2 mg/L.

The steps involved in the manufacture of solar cells and which generate the largest flows and the most concentrated wastewater are illustrated in the following Table 2.

In general, the silicon wafer cleaning station at the laboratories of the Research Center in Semi-conductor Technology for the Energetic (CRTSE) requires a huge volume of ultrapure water to remove organic and metal impurities from the surface of silicon wafers. The high price of water and its treatment makes it necessary to reduce consumption and the treatment and reclamation of water bearing fluoride are significant and important and can be transformed with inexpensive means in utilitarian waters and according to this fact to meet the water quality requirements for reuse.

To reduce fluoride content in water to a value that meets water standards for specific purposes, various treatment techniques have been implemented such as membrane techniques [4], adsorption [5,6], ion exchange, precipitation–coagulation [7], electrochemical processes [8], electrodialysis [9,10] where each technique has its advantages and disadvantages.

The aim of this work is to treat HF rinsing water resulting from deoxidation of silicon wafers by an electrocoagulation process using aluminum as sacrificial anode in a bipolar reactor. Several parameters governing the process have been optimized. A characterization of the sludge produced was also studied.

Table 2
Solar cell manufacturing steps

Steps	Discharges
Saw damage etch	HF/HNO_3 baths
Texturing	Rinsing baths HF/HNO_3
	HF rinse bath
	Concentrated HF bath
Elaboration of $POCl_3$ emitter	HF rinse bath
HF deoxidation	
PSG-etching	Concentrated HF bath
	HF rinse bath

1.1. Electrocoagulation

Electrocoagulation (EC) is a simple and efficient method and has been used for the treatment of many types of wastewaters such as industrial effluent [11], photovoltaic wastewater [12], olive oil mill wastewater [13], real dairy wastewater [14] and textile wastewater [15]. EC process involves oxidation and reduction reaction in which destabilization of contaminants (suspended, emulsified or dissolved) occurs due to the application of electric current to the electrolytic solution. EC unit consists of an electrolytic cell and metal (Al or Fe) electrodes connected to an external power supply [16]. In the EC process, anodic dissolution generates in situ coagulants along with hydroxyl ions and hydrogen gas at the cathode [16]. In an electrocoagulation (EC) process, no addition of chemicals is required; Small amount of sludge is produced, compared with the amount generated in classical chemical process, which can be easily removed by decantation [17]. EC requires only low currents and can be operated using green technologies such as solar or wind power.

2. Materials and methods

2.1. Chemicals

The fluorinated discharges are collected after each deoxidation operation of the silicon wafers, their characteristics are shown in Table 3.

In order to simulate photovoltaic wastewater, the desired concentrations of F^- solution were prepared by dissolving appropriate amount of sodium fluoride in water. Sodium chloride was used as a supporting electrolyte and the pH was adjusted by adding sodium hydroxide (1 N) or sulfuric acid (1 N). All chemicals were obtained from Prolabo, Paris, France.

Table 3
Operationg conditions

Parameters	Values
Fluoride (mg/L)	100
pH	3
Conductivity (mS/cm)	5

2.2. Electrocoagulation experiment

The study was conducted using a pilot plant with three aluminum electrodes. Aluminum electrodes were used as the anode and cathode. The electrodes used are all planes in square shape, of a dimension 10x10 cm and 2 mm thick. The total anodic area was 100 cm^2. The interelectrodic gap was 1 cm and the volume of the electrocoagulator was 1.5 L. For most runs, aluminum electrodes were arranged in bipolar mode. The purity of the aluminum electrodes used was about 99.8%. Before and at the end of each run, electrodes were washed thoroughly with water, dipped in HCl solution (5% vol/vol) for at least. The reactor consists of a parallelepiped shaped Plexiglas cell. The current input of the DC power supply was maintained constant; by means of a precision DC power supply (P.Fontaine MC 303°C). The efficiency of the process also depends on the sample pH. The pH in the reactor was maintained at 7. Sample solution of 10 mL from each run were collected at contact times of 10 min to 120 min and tested for parametric analysis. The sample supernatant was filtered and analyzed. Experiments were conducted with temperature of around 25°C. The experimental setup is schematically shown in Fig. 1.

2.3. Chemical analysis

A selective ion sensor electrode (PF4L from Tacussel [Lyon, France]) was used to determine the fluoride concentration, according to the standard method given by American Public Health Association Greenberg et al. (1992). To prevent the interference from other ions (Al^{3+}, Fe^{3+}, Cu^{2+} and Ca^{2+}), TISAB II buffer solution-containing CDTA was added to the samples [18].

The pH values were determined by using Sension 1 pH meter (HACH, USA).

Environmental scanning electromicroscopy (SEM) Quanta (type FC250) was used to characterize sludge. SEM pictures were taken at 15 kV at various magnifications.

The ATR-FTIR analysis in the flocs was carried out in a PerkinElmer (Turku, Finland) Spectrum GX FTIR spectrometer using an EasiDiff diffuse reflectance accessory. Analysis was carried out with PerkinElmer (Turku, Finland) paragon 1000 spectrum RX and the results were obtained with OMNIC software. The flocs samples were prepared using potassium bromide.

3. Results and discussion

3.1. Effects of operational parameters on EC process

In order to have maximum efficiency of the electrocoagulation process, it is necessary to study the effect of various important parameters that govern the process.

3.2. Effect of current density

The current density is the key operational parameter that affects the system's response time and also influences the dominant pollutant separation mode [19]. To examine the effect of current density on fluoride removal efficiency, a series of experiments were carried out with the current density variation from 13.88 to 37.03 A/m^2. The results are depicted in Fig. 2. It can be seen that the fluoride removal efficiency increases with the increase of current density. The fluoride removal increased significantly and reaches standard limits in 40 and 50 min for applied current density of 37.03

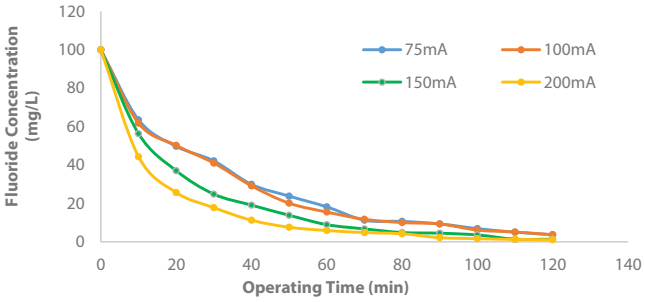


Fig. 2. Effect of current intensity on the removal of fluoride.

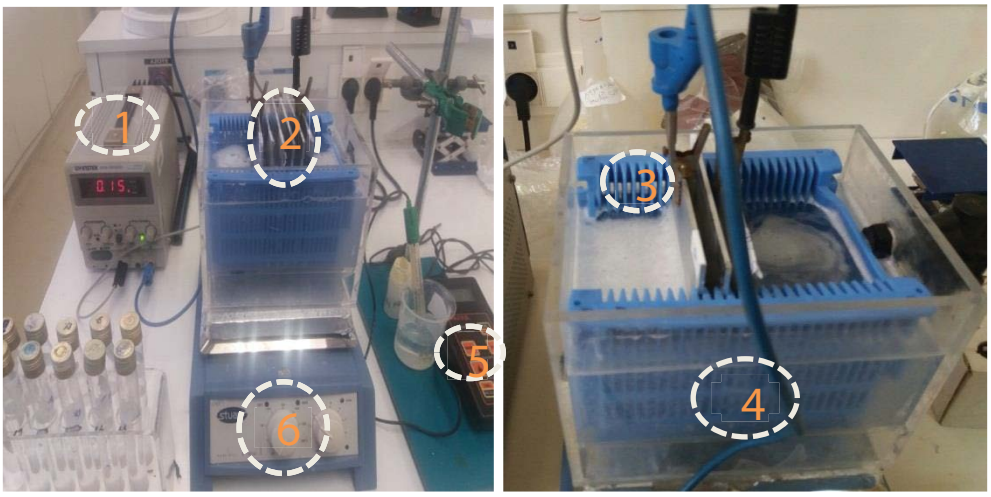


Fig. 1. Laboratory-scale electrocoagulation system: (1) DC power supply; (2) aluminum electrodes; (3) electrodes support; (4) electrocoagulator; (5) pH meter; (6) magnetic stirrer.

and 27.77.51 A/m², respectively; whereas for 13.88 A/m² more time is needed to achieve the same results. This is mainly due to insufficient amount of electric power at lower voltages to produce aluminum coagulant species and gas bubbles responsible for defluoridation. According to Faraday's law, increasing the current density allows to a higher coagulant dosage per time unit [18].

The results obtained were confirmed by studying the effect of the current density on the specific electrical energy consumption (EESC) and the reduction rate of the fluoride ions for a time equal to 60 min. EESC is defined as the amount of electrical energy consumed per unit mass of pollutant (fluorine in our case). EESC was calculated using Eq. (1):

$$\text{EESC} = \frac{IUT}{\left(X_i - X_f\right)V} \tag{1}$$

where U is the voltage across the electrodes (V), I is the intensity (A), t is the time (h) and V is the volume (L), X_i is the initial concentration of fluoride ions (g/L), X_f is the final concentration of fluoride ions (g/L).

The effect of the current density on EESC and the rate of fluoride ion abatement is shown in Fig. 3, we found that when the current density increased from 13.88 to 27.77 A/m², the rate of the fluoride abatement increased from 81.77% to 91.03% while the corresponding EESC increased slightly. However, when the current density increased from 27.77 to 37.03 A/m², the EESC increased significantly. From these results, we can deduce that the current density of 27.77 A/m² provides the optimal conditions for removal efficiency of fluoride ions (91.03% as removal rate and 5.5 kWh/g F of EESC.

3.3. Effect of electrodes number

There is a direct relation between removal efficiency and the total number of electrodes. So when the area of electrodes increases, the distribution of the coagulation agents density will be more effective [20]. The effect of electrodes number was studied. Fig. 4 shows that with increase in electrode number, the fluoride removal efficiency increases. It can be due to more consumption of energy and then production of more flocs in shorter time [4]. Almost 95% removal efficiency is obtained within 50 min for electrodes number 4, 5 and 6. Fig. 4 shows the relationship between operation cost and number of electrode. As observed from Fig. 2 energy consumption and costs increased with increased number of electrodes. However the number of electrodes was chosen as 3 for the rest of the work.

3.4. Effect of initial pH on the efficiency of fluoride removal

It has been established that pH is an important parameter influencing the performance of the electrochemical process [21]. The maximum pollutant removal efficiency is obtained at an optimum solution pH for a particular pollutant. The precipitation of a pollutant begins at a particular pH. The pollutant removal efficiency decreases by either increasing or decreasing the pH of the solution from the optimum pH [22]. It also has influence on the superficial charge of the aluminum hydroxide precipitates (caused by the adsorption

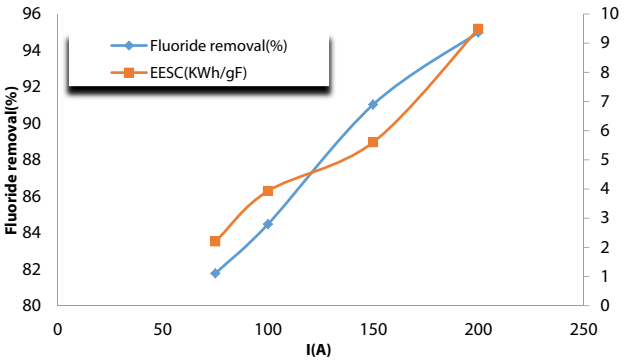


Fig. 3. Effect of the current density on EESC and the rate of fluoride ion abatement.

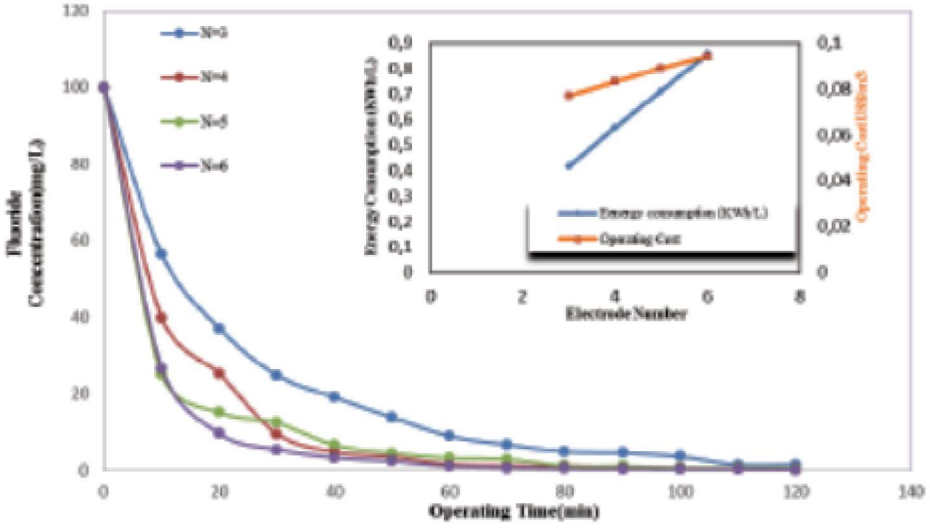


Fig. 4. Effect of the number of electrodes on the removal of fluoride ions (pH = 7. i = 27.77 A/m²).

of ionic species) [23]. During the time course of coagulation and EC processes, the pH changes in an opposite direction and this affects significantly the coagulant species formed, and hence the efficiencies of pollutants removal obtained [23]. To evaluate the effect of pH on the removal of fluoride ions, we have studied the elimination of these for various solutions at pH ranging from 4 to 9. This range has been chosen, because the pH of the species is generally in this range. Fig. 5 shows the removal of fluoride after 120 min as a function of pH. The results obtained showed that the influence of pH is not negligible on the elimination of the pollutant. However, it can be observed that the optimal results were obtained for pH values of 4 and 7. In fact, aluminum is mainly in the form of aluminum hydroxide in this pH range, the latter is suitable for the elimination of fluoride. Emamjomeh and Sivakumar [24] found that the pH of 7 gives the fastest elimination kinetics in CE with aluminum electrodes.

3.5. Consumption of electrode material and energy

The energy consumption and consumption of consumable electrodes are very important economic parameters in the electrocoagulation process. Like all other electrolytic processes, they are taken into account in the calculation of the operating cost, that is, per liter of treated effluent or $ by quantity of fluoride ions removed [25,26]. Therefore, main operating cost of the EC process is due to two components as explained in the equation:

$$\text{Operating cost} = xC_{\text{Energy}} + yC_{\text{Electrode}} \quad (2)$$

where C_{Energy}, $C_{\text{Electrode}}$ are the amount of electrical energy and electrode consumed per liter of wastewater treated, which is experimentally achieved and the variables x and y are obtained via the Algerian market price of October 2018, and it is as follows:

- x: price of electric energy US$ 0.039/kWh,
- y: electrode material price 2.17 US$/kg for aluminum.

The relationship between current density and dissolved electrode materials for electrochemical processes is commonly correlated using Faraday's Law. Electrode consumption and energy consumption are theoretically calculated by the following equations [27]:

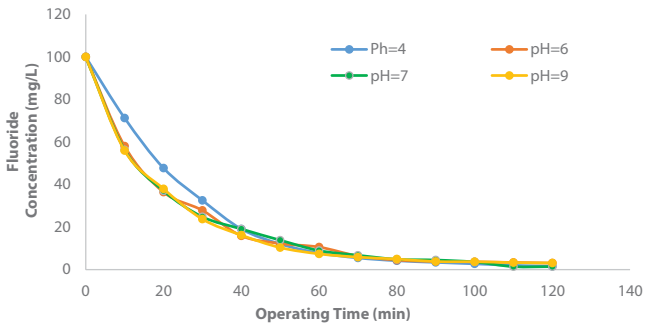


Fig. 5. Effect of pH on the fluoride removal during electrocoagulation process (Nb = 3, i = 27.77 A/m^2).

$$C_{\text{Electrode}} = \frac{ItM_{\text{Electrode}}}{nFV} \quad (3)$$

where C = the amount of dissolved electrode material (g/L); I = current intensity (A); t = time (s); $M_{\text{Electrode}}$ = molecular weight of electrode material (g/mol); n = valence of metal ions (e^-/mol); F = Faraday constant (96,500 Coulomb/mol); V = volume of treated solution (L).

$$C_{\text{Energy}} = \frac{UI \times t_{EC}}{V} \quad (5)$$

C_{Energy} = Consumed energy (kWh/L); I = current intensity (A); U = volt (V); t = time (h); V = volume of treated solution (L).

In this study, the discharge standards have been achieved in a time of 50 min; therefore, the calculations were all made for that value. The electrode and energy consumptions in relation with current density are given in Fig. 7. The results showed that the energy and electrode consumptions are, respectively, 0.144 kWh/L and 0.28 g/L for a current density of 27.77 A/m^2. Meanwhile the cost increased from 0.076 US$/m^3 and 0.108 US$/m^3 for a current density of 27.77 and 37.07, respectively, as can be seen in the inset graph in Fig. 6. The electrode and energy costs increase with the increased in the density of current. Following these results, we can conclude that EC has been performed with lower energy consumption and cost effective manner.

3.6. Effect of electrolysis time

Residence time is an important factor that determines the amount of produced Al^{3+} and OH^-. As the residence time increases, the amount of electro-generated Al^{3+} increases, resulting in the increment of the amount of flocs, which is made up of insoluble monomeric and polymeric aluminum hydroxides [28]. The pollutant removal efficiency increases with an increase in the electrolysis time. But beyond the optimum electrolysis time, the pollutant removal efficiency becomes constant and does not increase with an increase in the electrolysis time. Electrolysis time has a negative impact on cost of treatment due to increase in energy and electrode consumption at longer electrolysis time [29] (Fig. 8). As a result, it plays an important role in the choice of the operating conditions of the process. The removal efficiencies of F^- under different residence times are shown in Fig. 7.

3.7. Effect of initial fluoride concentration

To understand the effect of initial fluoride concentration on fluoride removal, the experiments were performed for three initial fluoride concentrations. The experiments were conducted by changing initial fluoride concentration from 30 to 100 mg/L at the same current densities and electrolysis time. It can be observed from Fig. 9 that when the initial fluoride concentration was increased from 30 to 100 mg/L at a constant electrolysis time of 20 min and constant applied current of 27.77 A/m^2, the values for residual fluoride concentration increases from 3.83 to 37.06 mg/L. This is possibly due to formation of insufficient aluminum hydroxide complexes. It was also observed that the higher initial fluoride concentration requires a higher value of applied current or longer

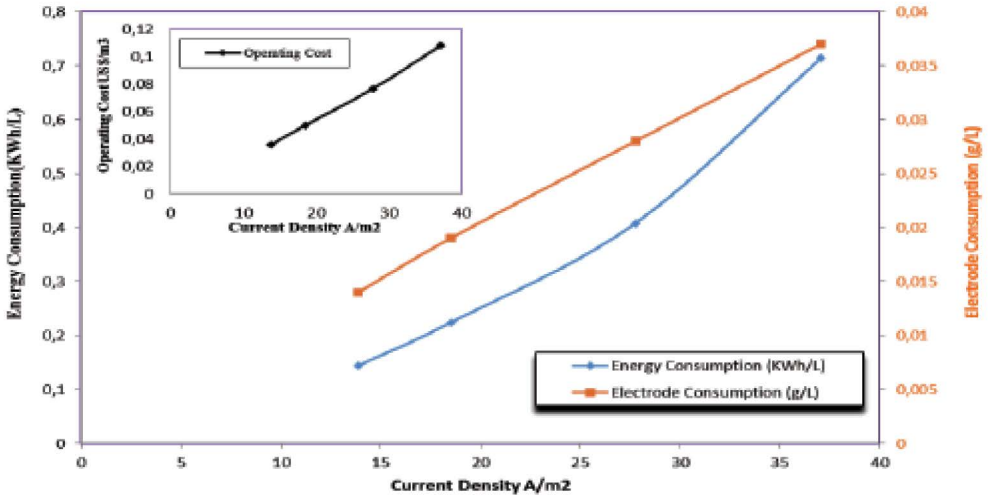


Fig. 6. Effect of current density on the energy and electrode consumption (pH = 7, t = 50 min).

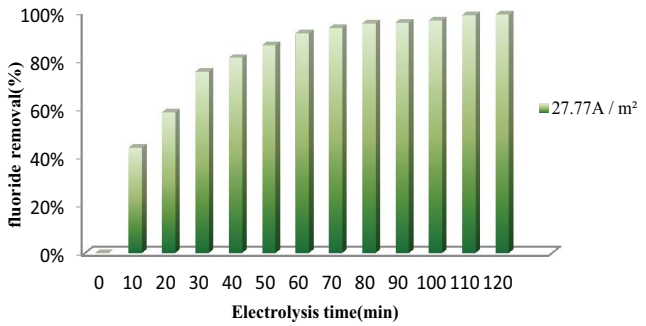


Fig. 7. Effect of initial fluoride concentration on fluoride removal (pH = 7, i = 27.77 A/m²).

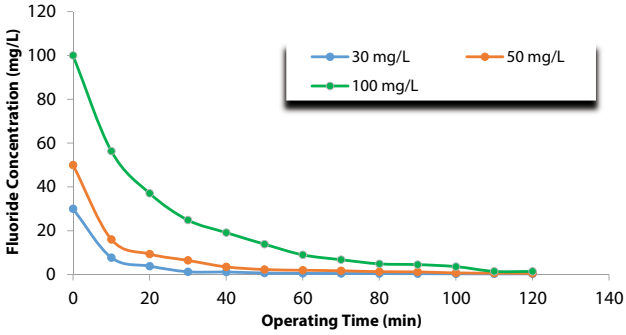


Fig. 8. Effect of initial fluoride concentration on removal efficiency (pH = 7, i = 27.77 A/m²).

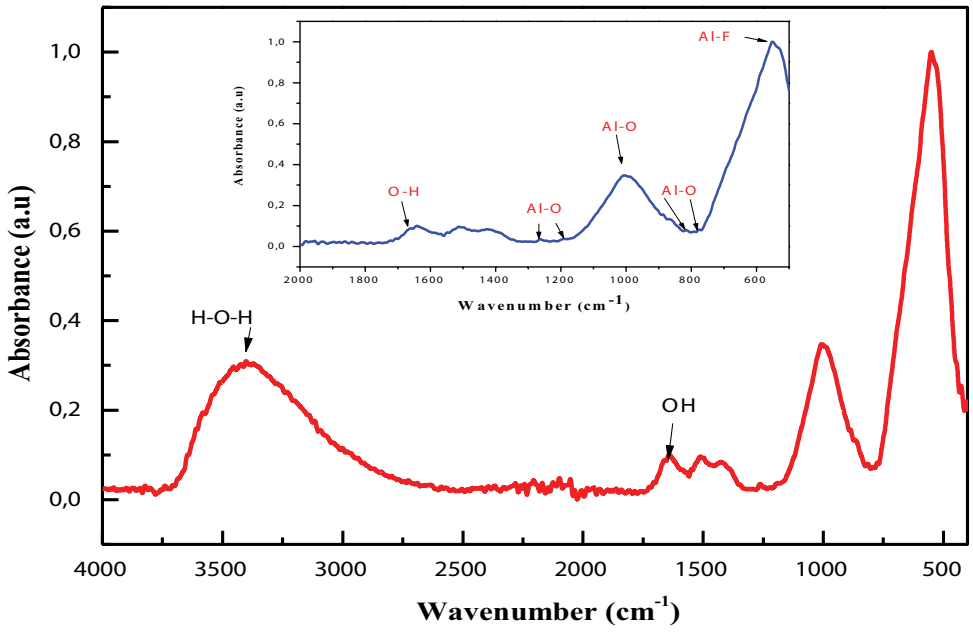
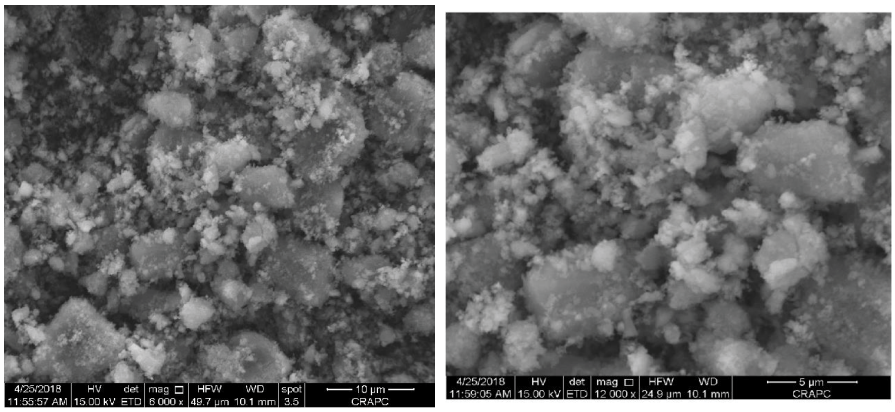


Fig. 9. ATR-FTIR spectrum of the sludge produced in the electrocoagulation process.



(a) (b)

Fig. 10. SEM visualization of sludge after 90 min of EC at two different magnifications (a) 6,000× and (b) 12,000×.

electrolysis time for bringing down the residual fluoride below the permissible limit of 15 mg/L. Therefore, we can conclude that higher initial fluoride concentration needs higher current for enhancing the defluoridation efficiency.

4. Characterization of the by-products

4.1. ATR-FTIR analysis

The electro co-precipitated sludge generated during electrocoagulation was analyzed using FTIR spectrometer. A 10–15 mg of sample was dispersed in 200 mg of spectroscopic grade KBr to record the spectra. Scans were collected at a resolution of 4 cm^{-1}. The wave numbers ranged from 4,000 to 400 cm^{-1}. The infrared spectrum analysis of the defluoridation process showed that the peaks in the 700–1,300 cm^{-1} region corresponded to the stretching and bending modes of Al–O [30]. The peaks located at 551 cm^{-1} is attributed to the Al–F bonding. A wide band between 3,600 and 3,000 cm^{-1} attributed to the vibration of the hydroxyl group (O–H). The band at 1,642 cm^{-1} corresponds to the O–H deformation. From this analysis, it was confirmed that fluoride was linked with aluminum hydroxide complexes and precipitated at the bottom of EC.

4.2. Characterization by SEM

To evaluate the microstructural characteristics of the precipitate generated by the electrochemical process, SEM experiments were conducted. Figs. 10a and b show the morphology of the precipitate produced by the electrocoagulation process. The aggregates formed do not have a well-defined structure. The SEM images show agglomerated grains of different sizes and the presence of an ultrafine structure in the nanometer range.

The FTIR and SEM analyses of the EC by-products thus lead us to conclude that the chemical speciation of this amorphous phase can be aluminum hydroxides and aluminum oxyhydroxides.

5. Conclusion

The aim of this study was to treat the rinsing water contaminated by the fluoride ions generated from the photovoltaic process by an electrochemical process. The influence of several important parameters governing the process of electrocoagulation has been studied in this work. The effectiveness of the treatment depended on the initial pH, current density, electrolysis time and active surface. It was observed that these variables significantly affected the fluoride removal. The optimum fluoride removal (88%) was obtained with typical operating conditions: current density, 27.77 A/m^2; number of electrodes of 3, and initial pH of 7. The best EC tests in terms of energy consumption were obtained at 27.77 A/m^2 with a value equal to 5.5 kWh/gF. The ATR-FTIR analysis of the precipitate obtained after treatment, allowed us to conclude that the by-products of the EC are mainly oxyhydroxides and aluminum hydroxide. The SEM image showed that the precipitate formed flocs and aggregates thus ensuring the adsorption of fluoride ions on in situ generated coagulant species. Electrocoagulation using aluminum electrodes is a compact and efficient process for the reduction of fluorine rinsing water from surface treatment of silicon wafers. The generated sludge was neutral and non-toxic.

Acknowledgements

The authors would like to thank the General Directorate for Scientific Research (DGRSDT) and the Semiconductor Research Center for Energy (CRTSE) for their financial support.

References

[1] L. Peters, Semiconductor International, March 2003, pp. 50–54, Available at: http://www.reed-electronics.com/semiconductor/archive/1998.html (Accessed September).
[2] N. Drouiche, P. Cuellar, F. Kerkar, S. Medjahed, N. Boutouchent-Guerfi, M. Ould Hamou, Recovery of solar grade silicon from kerf loss slurry waste, Renew. Sustain. Energy Rev., 32 (2014) 936–943.
[3] S. Aoudj, A. Khelifa, N. Drouiche, M. Hecini, Removal of fluoride and turbidity from semiconductor industry wastewater by combined coagulation and electroflotation, Desal. Wat. Treat., 57 (2016) 18398–18405.
[4] A. Mazighi, H. Lounici, N. Drouiche, R. Leenaerts, N. Abdi, H. Grib, N. Mameri, Economic study of groundwater defluoridation of the North African Sahara, Desal. Wat. Treat., 54 (2015) 2681–2691.

[5] Y. Çengeloğlu, Y.E. Kir, M. Ersöz, Removal of fluoride from aqueous solution by using red mud, Sep. Purif. Technol., 28 (2002) 81–86.
[6] S. Aoudj, A. Khelifa, N. Drouiche, M. Hecini, Development of an integrated electrocoagulation–flotation for semiconductor wastewater treatment, Desal. Wat. Treat., 55 (2015) 1422–1432.
[7] E.J. Reardon, Y. Wang, A limestone reactor for fluoride removal from wastewaters, Environ. Sci. Technol., 34 (2000) 3247–3253.
[8] N. Drouiche, B. Palahouane, S. Aoudj, M. Hecini, O. Bouchelaghem, T. Ouslimane, Defluoridation of post treated photovoltaic wastewater using aluminum electrodes: optimization of operating parameters and sludge characteristics, J. Sol. Chem., 45 (2016) 1571–1579.
[9] M. Zeni, R. Riveros, K. Melo, R. Primieri, S. Lorenzini, Study on fluoride reduction in artesian well water from electrodialysis process, Desalination, 185 (2005) 241–244.
[10] N. Drouiche, F.Djouadi-Belkada, T. Ouslimane, A. Kefafi, J. Fathi, E. Ahmetovic, Photovoltaic solar cells industry wastewater treatment, Desal. Wat. Treat., 51 (2013) 5965–5973.
[11] N. Adhoum, L. Monser, N. Bellakhal, J.E. Belgaied, Treatment of electroplating, wastewater containing Cu^{2+}, $Zn^{2+}$and Cr^{6+} by electrocoagulation, J. Hazard. Mater. B, 112 (2004) 207–213.
[12] N. Drouiche, S. Aoudj, M. Hecini, N. Ghaffour, H. Lounici, N. Mameri, Study on the treatment of photovoltaic wastewater using electrocoagulation: fluoride removal with aluminium electrodes – characteristics of products, J. Hazard. Mater., 169 (2009) 65–69.
[13] A.R. Yazdanbakhsh, M.R. Massoudinejad, K. Arman, E. Aghayani, Investigating the potential of electrocoagulation-flotation (ECF) process for pollutants removal from olive oil mill wastewater, J. Appl. Environ. Biol. Sci., 3 (2013) 22–28.
[14] F. Belkessam, H. Lounici, N. Drouiche, M. Aidene, R. Soni, Removal of toxic metal Cr (VI) from aqueous solutions by electrocoagulation using aluminum electrodes, Desal. Wat. Treat., 26 (2017) 221–230.
[15] E. Bazrafshan, A.H. Mahvi, M.A. Zazouli, Textile wastewater treatment by electrocoagulation process using aluminum electrodes, Iran J. Health Sci., 2 (2014) 16–29.
[16] M. Bharath, B.M. Krishna, B. Manoj Kumar, A review of electrocoagulation process for wastewater treatment, Int. J. Chem. Technol. Res., 11 (2018) 289–302.
[17] K. Missaoui, W. Bouguerra, C. Hannachi, B. Hamrouni, Boron removal by electrocoagulation using full factorial design, J. Water Res. Protect., 5 (2013) 867–875.
[18] S. Aoudj, A. Khelifa, N. Drouiche, R. Belkada, D. Miroud, Simultaneous removal of chromium (VI) and fluoride by electrocoagulation-electroflotation: application of a hybrid Fe-Al anode, Chem. Eng. J., 267 (2015) 153–162.
[19] H. Liu, X. Zhao, J. Qu, Electrocoagulation in Water Treatment, C. Comninellis, G. Chen Eds., Electrochemistry for the Environment, Springer, 21 September 2009, pp. 245–262.
[20] B. Palahouane, N. Drouiche, S. Aoudj, K. Bensadok, Cost-effective electrocoagulation process for the remediation of fluoride from pretreated photovoltaic wastewater, J. Ind. Eng. Chem., 22 (2015) 127–131.
[21] N. Boudjema, N. Drouiche, N. Abdi, H. Grib, H. Lounici, A. Pauss, N. Mameri, Treatment of Oued El Harrach River Water by electrocoagulation noting the effect of the electric field on microorganisms, J. Taiwan Inst. Chem. Eng., 45 (2014) 1564–1570.
[22] E. Bazrafshan, L. Mohammadi, A. Ansari-Moghaddam, A.H. Mahvi, Heavy metals removal from aqueous environments by electrocoagulation process-a systematic review, J. Environ. Health Sci. Eng., 13 (2015) 74.
[23] P. Canizares, C. Jiménez, F. Martínez, M. Rodrigo, C. Saez, The pH as a key parameter in the choice between coagulation and electrocoagulation for the treatment of wastewaters, J. Hazard. Mater., 163 (2009) 158–164.
[24] M.M. Emamjomeh, M. Sivakumar, Fluoride removal by a continuous flow electrocoagulation reactor, J. Environ. Manage., 90 (2009) 1204–1210.
[25] N. Daneshvar, A. Oladegaragoze, N. Djafarzadeh, Decolorization of basic dye solutions by electrocoagulation: an investigation of the effect of operational parameters, J. Hazard. Mater., 129 (2005) 116–122.
[26] N. Drouiche, N. Ghaffour, H. Lounici, N. Mameri, A. Maallemi, H. Mahmoudi, Electrochemical treatment of chemical mechanical polishing wastewater: removal of fluoride-sludge characteristics-operating cost, Desalination, 223 (2008) 134–142.
[27] A. Abdi Mohamud, Y. Çalışkan, N. Bektaş, H. Cengiz Yatmaz, Investigation of shipyard wastewater treatment using electrocoagulation process with Al electrodes, Sep. Sci. Technol., 53 (2018) 2468–2475.
[27] J. Lu, Y. Li, M. Yin, X. Ma, S. Lin, Removing heavy metal ions with continuous aluminum electrocoagulation: a study on back mixing and utilization rate of electro-generated Al ions, Chem. Eng. J., 267 (2015) 86–92.
[29] M. Bharath, B.M. Krishna, B.A. Manoj Kumar, Review of electrocoagulation process for wastewater treatment, Int. J. Chem. Technol. Res., 11 (2018) 289–302.
[30] Inorganic library of FTIR Spectra Minerals, Nicodom, Vol. 1, 1998.