

## Simultaneous removal of fluoride and sodium dodecyl sulfate from synthetic photovoltaic wastewater by electrocoagulation: evaluation of operating parameters

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### ABSTRACT

The electrocoagulation (EC) method was applied for the treatment of a complex synthetic photovoltaic wastewater. In photovoltaic (PV) process, HF acid treatment is the most essential step in wafers manufacturing. In addition, repetitive cleaning of the wafers are achieved by use of surfactants. Consequently, PV wastewater is currently rich of fluorides ions and organic surfactants such as sodium dodecyl sulfate (SDS). In this study, fluoride and SDS are used as model pollutants. Previous studies deal with fluoride and SDS individually using one electrode type. So, this research targets to evaluate the performance of the electrocoagulation process on the removal of fluoride and SDS with hybrid electrode composed of aluminum and iron plates. The treatment was carried out in a discontinuous system equipped with aluminium and iron electrodes. Five influencing variables were investigated: anodic EC material, current intensity, initial pH, electrolysis time and initial pollutant concentration. The best conditions for simultaneous removal F<sup>-</sup> and SDS with initial concentrations of 100 and 60 mg/L, respectively are: initial pH of 7, a current density of 37.03 A/m<sup>2</sup> and a treatment time of the 80 min. The corresponding removal efficiencies were 89.07% for fluorides and 93.3% for SDS. The results showed that iron is more adequate to remove SDS while aluminium electrode is better to remove F<sup>-</sup>. EC method with hybrid Al-Fe electrodes gives better results for simultaneous removal of fluorides and SDS than one type electrodes. Hybrid Al-Fe makes a good compromise.

*Keywords:* Synthetic photovoltaic wastewater; Fluoride; Sodium dodecyl sulfate; Al-Fe electrodes

### 1. Introduction

Photovoltaic (PV) industry has known a fast growth in the past few years because of the increasing depletion and

the negative environmental impact of fossil fuels [1]. The demand for solar cells is going to increase with the wider expansion of solar energy as a renewable power source. Solar wafers manufacturing process requires consumption of a variety of chemicals and large volumes of ultra-pure water.

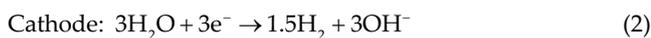
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Several processes are used in the surface treatment of silicon wafers. In order to have a very clean silicon wafer surface, acidic solutions such as hydrofluoric acid are widely used. Furthermore, in order to ensure the removal of stains and metal impurities on the wafer surface, surfactants such as sodium dodecyl sulfate (SDS) are added to these acidic solutions [2,3].

Consequently, PV industry effluents contain various inorganic and organic pollutants, such as fluoride ions and SDS. These contaminants are found at high concentrations which represents a major environmental threat [2,4]. Many studies have demonstrated that the discharge of effluent with high content of fluoride is harmful for health and environment [5–8]. In addition, skeletal fluorosis occurs if excessive fluoride is consumed by humans [9]. Environmental authorities limit fluoride discharge levels to 15 mg/L [5]. SDS is not very toxic for humans but its toxicity has been established for aquatic media [10,11]. Environmental regulations set strict standards for anionic surfactants which vary from 1 to 4 mg/L for wastewater [12,13]. In order to meet environmental requirements, the PV effluents must be properly treated before being discharged. In addition, because of the huge amounts of processing water, water recycling has become an important issue in photovoltaic industries [14]. Generally, the semiconductor industry reuses its treated wastewater for cooling and other facility applications which can significantly reduce water consumption [9].

Conventionally, several methods have been used in PV effluents treatment and they have been more and less performant in reducing pollution impact and water recyclability. Electrocoagulation (EC) technology has attracted growing interest and has been successfully developed as an alternative of conventional methods for wastewater treatment [4,15]. The EC has many advantages such as environmental compatibility, versatility, automation, cost-effectiveness, energy efficiency and safety [4,15]. Most importantly, electrocoagulation has been demonstrated to be effective for organic and inorganic pollutant treatment. The principle of EC is based on the *in-situ* generation of coagulants by electro-dissolution of a sacrificial anode [15]. The most used electrodes are iron or aluminium. The EC mechanism can be summarized as follows [15–17]:

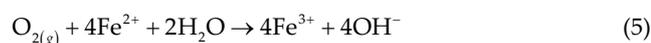
The main reactions in the EC process with aluminum as a sacrificial anode are:



The main reactions in the EC process with iron as a sacrificial anode are:



In the bulk of the solution:



The anodic dissolution of electrodes results in the formation of metal cations ( $\text{Fe}^{2+}/\text{Fe}^{3+}$  or  $\text{Al}^{3+}$ ) which react with hydroxide ions  $\text{OH}^-$  to form the metal hydroxides  $\text{Al}(\text{OH})_3$ ,  $\text{Fe}(\text{OH})_3$ , and  $\text{Fe}(\text{OH})_2$ . These metallic hydroxides possess high specific surface area which allows the removal of pollutants by adsorption and trapping on their surface. Furthermore, the removal of organic matter by EC process may take place in three ways: (i) neutralization of negative charge and double layer compression of colloidal particles, (ii) co-precipitation with coagulants, and (iii) complexation and electrostatic attraction with coagulants [15].

Electrocoagulation using aluminum anodes was successfully used for fluoride removal from acid waste [18–22]. Additionally, several authors have studied removal of SDS from wastewater [2,10,21]. Electrochemical treatments are also applied to wastewaters which contain SDS [10]. Electrocoagulation with iron electrodes showed to be efficient for SDS removal [2,16,17].

Herein, the present study aimed to investigate the feasibility of synthetic PV industry effluent treatment by EC process. As this kind of wastewater is currently a complex mixture of organics and inorganics, fluoride and SDS are used as model pollutants. Previous studies deal with fluoride and SDS individually using one electrode type. So, this research targets to evaluate the performance of the electrocoagulation process on the removal of fluoride and SDS using Al/Fe hybrid electrodes. The effect of various parameters such as electrode material, initial pH, current density, electrolysis time and initial pollutant concentration on the efficiency of the electrocoagulation process were explored. Energy and electrode consumption during EC were also estimated.

## 2. Materials and methods

### 2.1. Electrolytic cell

The characteristics of EC reactor are given in Table 1. All electrolysis runs were carried out at a constant current provided by a P.Fontaine MC-303 generator. The current density was fixed in the range 27.77–55.55 A/m<sup>2</sup> and applied under regular magnetic stirring. Aluminum and iron electrode were used as the anode and cathode (Fig. 1).

### 2.2. Experimental procedure

In order to ensure surface reproducibility prior to each experiment, the aluminium or iron plates were manually polished using abrasive paper, degreased in acetone, rinsed with distilled water, submerged in dilute  $\text{H}_2\text{SO}_4$  solution, rinsed again with distilled water and then dried before immersion in the synthetic solution. The used concentration were chosen based on previous studies dealing with fluoride and SDS removal. At the beginning of each experiment, the pH of the solution was adjusted to a desired value using either 0.1 M NaOH or 0.1 M HCl as necessary. Different samples of 10 mL were taken at 20 min intervals for 2 h and

filtered before being analysed to determine the residual fluoride and SDS concentration. NaCl was used as supporting electrolyte. All experiments were conducted at room temperature ( $23^{\circ}\text{C} \pm 2^{\circ}\text{C}$ ). All the experiments were repeated twice, and the experimental error was below 2%, the average data were reported.

Table 1  
Characteristics of electrocoagulation reactor

Electrodes	
Material (anode and cathode)	Iron (Fe) and aluminium (Al)
Shape	Square plate
Size (cm × cm)	10 × 10
Number	4
Thickness (mm)	2
Purity %	99.8
Plate arrangement	Parallel
Connection mode effective electrode surface area (cm <sup>2</sup> )	Bipolar parallel 54
Reactor characteristics	
Matter	Plexiglas material
Reactor mode	Batch
Dimensions (cm × cm)	16.2 × 13.7
Volume (L)	1.5
Power supply	
Current range (A)	0.15–0.30

The removal efficiency SDS and F<sup>-</sup> in synthetic photovoltaic wastewater treated by electrocoagulation is calculated as follows:

$$\eta\% = \left( \frac{C_0 - C_f}{C_0} \right) \times 100 \quad (8)$$

where  $C_0$  and  $C_f$  represent initial and final pollutant concentration (F<sup>-</sup>, SDS).

Charge loading, which is the charge transferred in electrochemical reaction for a given amount of water treated, is calculated by using the equation:

$$Q_{(C/L)} = I \times \frac{t_{(EC)}}{V} \quad (9)$$

where  $Q$  is the charge loading in C/L,  $I$  is the applied current in A,  $t$  is the treatment time in s, and  $v$  is the volume of treated water in L.

In the electrocoagulation process, the consumed mass of the electrode ( $C_{\text{electrode}}$ , kg/m<sup>3</sup>) and energy consumption ( $C_{\text{energy}}$ , kWh/m<sup>3</sup>) play a crucial role in respect of the economic efficiency of the proposed design set-up. They were calculated by the following equations:

$$C_{\text{energy}} = \frac{UIt(EC)}{V} \quad (10)$$

$$C_{\text{electrode}} = \frac{ItM}{nFV} \quad (11)$$

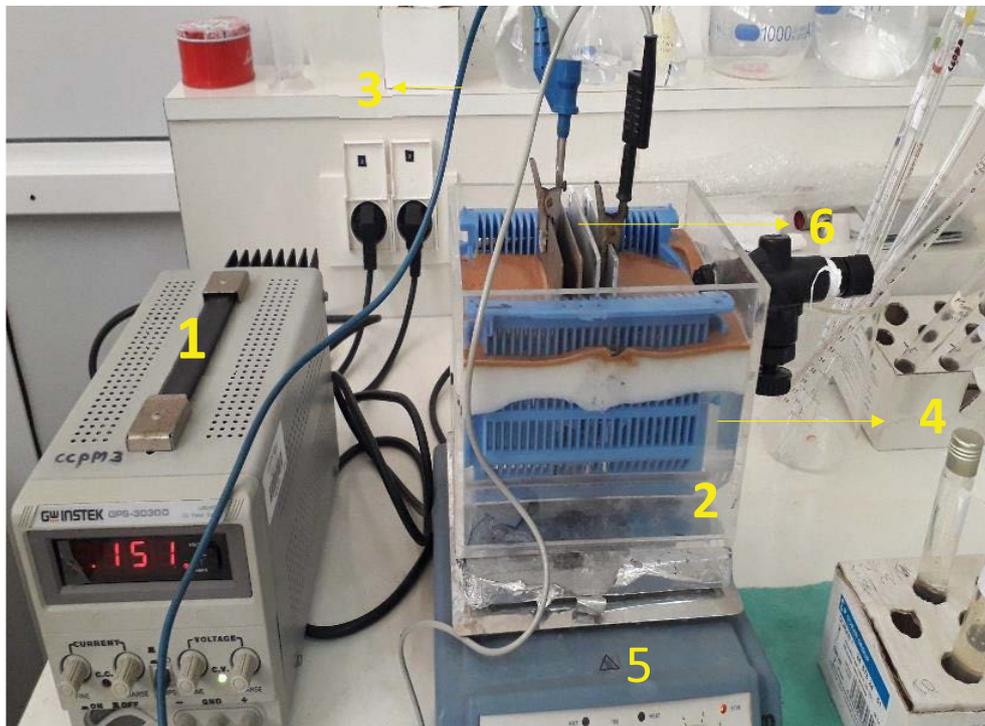


Fig. 1. Electrocoagulation experimental set-up: 1: DC power supply, 2: Electrocoagulation cell, 3: Electric wire, 4: Treated solution, 5: Magnetic stirrer, 6: Aluminum and iron electrodes, 4: Treated solution.

where  $U$  = electrical voltage (V),  $I$  = current intensity (A),  $t$  = operating time (h),  $M$  = molecular weight of aluminium ( $Al = 26.98$  g/mol),  $F^-$  Faraday's constant (96,500 Coulomb/mol),  $z$  = number of electrons involved in the oxidation/reduction, reaction and  $v$  = volume of the treated solution (L).

### 2.3. Chemicals and analytic methods

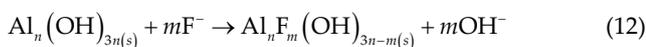
fluoride synthetic stock solution was prepared by dissolving the required quantities of NaF in deionised water. The NaF used was a Merck Reagent (USA) (99.5%), SDS (99%) and all chemicals were obtained from Biochem (France). All aqueous solutions were prepared by dissolution in deionized water. Concentration of fluoride was determined by a combined selective ion electrode (sensION1 from HANNA, USA), ionometric standard method was used [23]. To prevent the interference from other ions ( $Al^{3+}$ ,  $Fe^{3+}$ ,  $Cu^{2+}$  and  $Ca^{2+}$ ), TISAB II buffer solution containing cyclohexylenediaminetetraacetic acid (CDTA) was added to the samples. Hach DR 2500 spectrophotometer (USA) was used to measure SDS concentration according to Standard Methods [23,24]. The pH values were determined by using pH meter HANNA Instruments HI8424 (USA).

## 3. Results and discussion

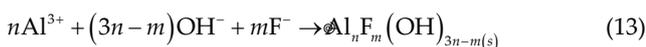
### 3.1. Effect of EC electrode material

According to the mechanism of EC process, the formation of hydroxides is due to the oxidation of the electrode which produces coagulation or flocculation agents and promotes the removal of the pollutants in the solution. Hence, the efficiency of the EC process is directly proportional to the electrode material. Aluminum and iron electrodes are both performant even though the amount of hydroxide generated by aluminium is less than the iron. Literature survey shows that the efficiency of electrode material is directly dependant on the nature of the target pollutant to be removed [2]. The aluminium electrodes are the more popular in fluoride removal than iron one because of aluminium species higher affinity in respect to fluoride ions. The mechanism of fluoride removal by EC using aluminium electrodes was studied by many authors [25–29] and two main mechanisms were proposed, adsorption and co-precipitation:

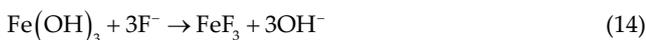
Adsorption on  $Al(OH)_3$ :



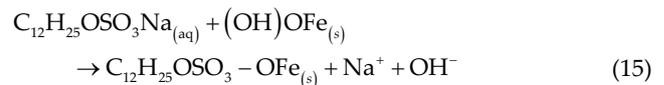
Co-precipitation:



Despite this, a fluoride removal mechanism by  $Fe(OH)_3$  was proposed by Martínez-Miranda et al. [28] as follows:



The  $Fe(OH)_3$  formed in electrocoagulation remove the organic pollutants from wastewater either by complexation or by electrostatic attraction, followed by coagulation [30]. In the surface complexation, it is suggested the following mechanism where SDS acts as a ligand to chemically bind hydrous iron [31]:



Furthermore, only few works were dedicated to the study of the efficiency of hybrid electrode Fe-Al in wastewater treatment. According to these studies, promising results are obtained when using these electrodes [32,33]. In the present study, different combinations of aluminium and iron electrodes were investigated. two pure configurations; 4Al-4Fe, three Al electrodes as anode and one Fe electrode as cathode (3Al-1Fe), two Al electrodes as anode and two Fe electrodes as cathode (2Al-2Fe), one electrode as anode and three Al electrodes as cathode (1Fe-3Al), one aluminium electrode as anode and three Fe electrodes as cathode (Al-3Fe) Fig. 2a shows that 4Fe and 2Al-2Fe electrode pairs have higher removal efficiency than with 4Al, Al-3Fe, and 1Fe-3Al electrode pairs in SDS removal. It can be seen in Fig. 2a that after 80 min of electrocoagulation complete removal is obtained by using only Fe plates (4Fe) and 2Al-2Fe as anode, whereas, the use of four Al plates results in the highest final SDS concentration of 40.46 mg/L. This is in perfect agreement with previous studies [2,34], which stated that EC-Fe is more suitable for SDS removal.

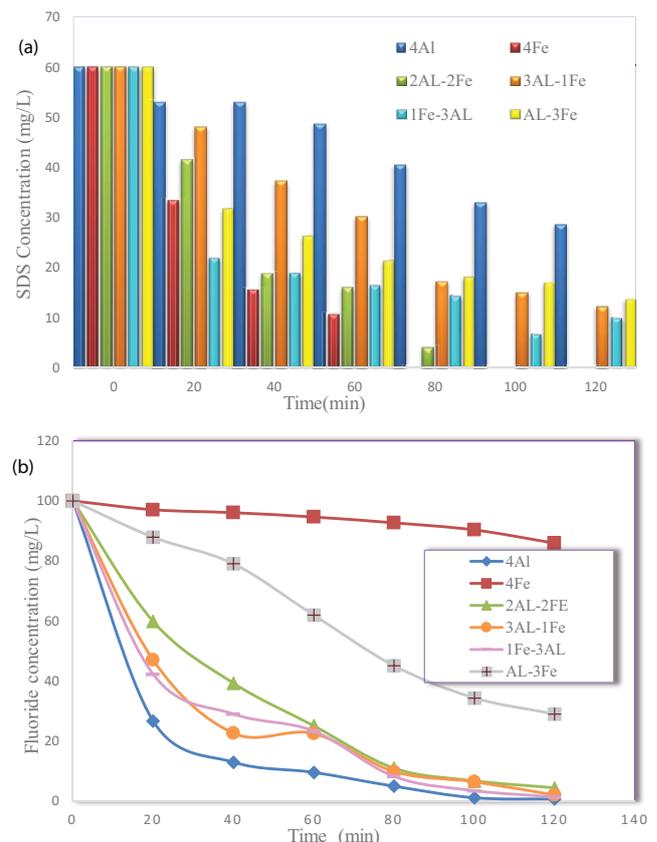


Fig. 2. (a) Residual concentration of sodium dodecyl sulfate as function of electrode material  $[SDS]_0 = 60$  mg/L,  $[F^-]_0 = 100$  mg/L,  $i = 37.03$  A/m<sup>2</sup>, initial pH = 7, and temperature = 23°C. (b) Residual concentration of F<sup>-</sup> as function of anode nature  $[SDS]_0 = 60$  mg/L,  $[F^-]_0 = 100$  mg/L,  $i = 37.03$  A/m<sup>2</sup>, initial pH = 7, and temperature = 23°C.

Fig. 2b shows the evolution over time of  $F^-$  removal efficiencies for different combinations of electrodes. The lowest  $F^-$  concentration was obtained with 4Al electrode combination at current density of  $37.03 \text{ A/m}^2$ , the residual fluoride is  $0.651 \text{ mg/L}$ . By using one, two or three iron plates as electrodes, an increase in final fluoride concentration is observed. It can be observed in Fig. 2b that 3Al-1Fe and 1Fe-3Al electrode pair have higher removal efficiency than with Al-3Fe and 4Fe electrode pairs. Also, the  $F^-$  removal decreased from 100 to  $4.38 \text{ mg/L}$  for 2Al-2Fe pair, from 100 to  $85.90 \text{ mg/L}$  for the 4Fe pair, from 100 to  $1.36 \text{ mg/L}$  for the 1Fe-3Al pair and from 100 to  $2 \text{ mg/L}$  for the 3Al-1Fe pair after 120 min of electrocoagulation. It is obvious that the treatment efficiency is strongly dependent on the number of Al and Fe electrodes. From these results, we can conclude that, EC-Al is more suitable for  $F^-$  removal and EC-Fe is more suitable for SDS removal.

### 3.2. Effect of electrolysis time

The electrocoagulation process is strongly influenced by the time of the reaction contact [35,36]. To investigate the effect of operating time; the current density is selected as  $37.03 \text{ A/m}^2$ , pH is hold at 7, combined electrodes: 2Al-2Fe and temperature of  $25^\circ\text{C}$ . The effect of electrolysis time on the simultaneous removal of  $F^-$  and SDS is shown in Fig. 3. The plot of Fig. 3 reveals that removal of both SDS and fluoride increases with increase in run time. Removal of 93.3% SDS is achieved in 80 min operation, while at 100 min, the SDS removal efficiency is 100%. This results can be due to the presence of a sufficient quantity of coagulant in the electrocoagulation reactor. It is also found that after 80 min of EC, fluoride concentration passes from 100 to  $10.83 \text{ mg/L}$  which represents 89.07% of removal efficiency, while passing from  $10.83$  to  $4.38 \text{ mg/L}$  is achieved in 120 min. The value of 80 min was taken as the optimum value for the electrolysis time because at this time both permissible values of fluoride and SDS are reached. Additionally, supplementary treatment results in excessive energy and electrode consumption.

### 3.3. Influence of initial pH

The pH has a crucial role in the appearance or disappearance of metal hydroxides forms, which directly affect

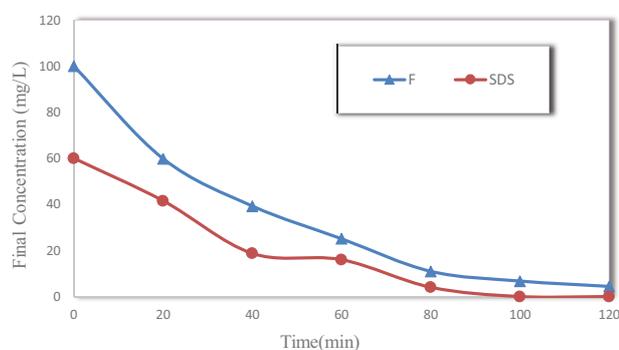


Fig. 3. Effect of time on final fluoride and sodium dodecyl sulfate concentrations:  $[\text{SDS}]_0 = 60 \text{ mg/L}$ ,  $[\text{F}^-]_0 = 100 \text{ mg/L}$ ; initial  $\text{pH} = 7$ ,  $i = 37.03 \text{ A/m}^2$ , and electrode: 2Al-2Fe.

the electrocoagulation performances [18]. The fluoride and SDS removal efficiency was determined in the pH range from 4 to 9. The current was settled to about  $37.03 \text{ A/m}^2$ . The results are shown in Table 2.

Additionally, from the results shown in Fig. 4, the variation of concentrations of SDS, and  $F^-$  with the solution initial pH could be identified. Results indicate that the removal of the SDS increased with increasing pH up to 7 and started to decrease at higher pH value of 9. These results are in good agreement with reported in the literature [34]. Barrera-Díaz et al. [37] also studied the effect of the initial pH on the reduction of organic matter using Al electrodes and found that the best removal efficiency is achieved within the pH range of 6 to 8. In alkaline conditions, the negatively charged complex ion  $\text{Al}(\text{OH})_4^-$ , which has low adsorption capacity, becomes the prevailing hydroxide species, causing an obvious decrease in the removal efficiency. Yüksel et al. [31] stated that the  $\text{Fe}(\text{OH})_n$  formed in electrocoagulation remains in the aqueous stream as a gelatinous suspension at  $3 < \text{pH} < 11$  which can remove the pollutants from wastewater either by complexation or by electrostatic attraction followed by

Table 2  
Influence of pH on simultaneous removal of sodium dodecyl sulfate and fluoride

Parameters	Values		
	4	7	9
Initial pH	4	7	9
Final pH	9.43	9.02	9.72
Current density ( $\text{A/m}^2$ )	37.03	37.03	37.03
Operating time (min)	80	80	80
Electrical voltage (V)	9.43	9.2	9.5
Initial sodium dodecyl sulfate concentration (mg/L)	60	60	60
Final sodium dodecyl sulfate concentration (mg/L)	19.25	4.02	13.27
Efficiency (SDS)	67.91%	93.3%	77.88%
Initial fluoride concentration (mg/L)	100	100	100
Final fluoride concentration (mg/L)	16.59	10.93	18.57
Efficiency ( $F^-$ )	83.41%	89.07%	81.43%

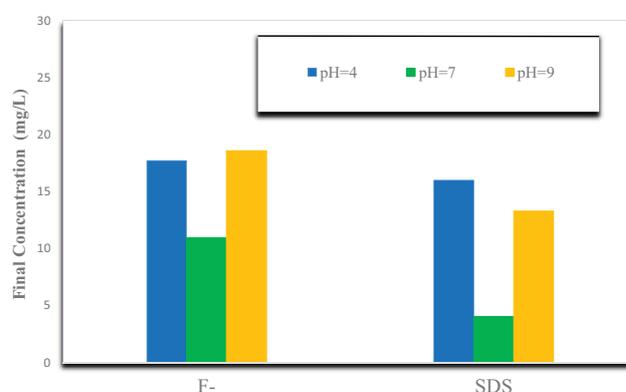


Fig. 4. Effect of initial pH on fluoride and sodium dodecyl sulfate removal:  $[\text{SDS}]_0 = 60 \text{ mg/L}$ ,  $[\text{F}^-]_0 = 100 \text{ mg/L}$ ,  $i = 37.03 \text{ A/m}^2$ ,  $t = 80 \text{ min}$ , and electrodes: 2Al-2Fe.

coagulation [34]. The same results were observed for  $F^-$ . When initial pH values are 4,7,9 the fluoride final concentrations are 16.59, 10.93 and 18.57 mg/L, respectively. This result is in good agreement with previous works [5,18]. It can be concluded that neutral pH 7 was the optimal. Besides, it has been found that the final pH increases steadily during the electrocoagulation process to a basic pH.

### 3.4. Effect of current density

Current density is a very important parameter that affects the electrocoagulation process because it directly determines coagulant dosage. The effect of current density on the removal of  $F^-$  and SDS was studied under the following operating conditions: 100 and 60 mg/L initial concentration for  $F^-$  and SDS, respectively, pH of 7, 80 min time of electrolysis and temperature of 23°C. Experimental conditions and measured parameters are presented in Table 3.

From Fig. 3 it can be concluded that current intensity has strong effect on removal of both  $F^-$  ions and SDS. The increase in current results in a decrease in residual SDS concentration. Applying an intensity of 150 mA (27.77 A/m<sup>2</sup>) gives 19.79 mg/L residual SDS at 80 min of treatment while 300 mA (55.55 A/m<sup>2</sup>) gives 1.3 mg/L. Similarly, for fluoride ions, the current increase leads to lower fluoride final concentrations. When current density varies from 27.77 to 55.55 A/m<sup>2</sup>, the final concentration of fluoride varies from 26.42 to 5.79 mg/L. However, when the current density increased from 27.77 to 55.55 A/m<sup>2</sup> the energy consumption increases from 0.957 to 3.03 kWh/m<sup>3</sup>, respectively. Similarly, for the electrode consumption which increases from 0.044 to 0.089 kg/m<sup>3</sup>, respectively. From these results, it could be considered that current intensity in the order of 37.03 A/m<sup>2</sup> (200 mA) provides the optimal conditions with fluoride/SDS removal efficiency and moderate energy consumption (1.54 kWh/m<sup>3</sup>).

### 3.5. Effect of initial pollutant concentration

The photovoltaic industry produces wastewaters with various levels of pollution, depending on the manufacturing process of the silicon cells. As a result, effluent quality is permanently changing. In this regard, experiments were conducted by changing initial pollutant concentration and

Table 3  
Experimental conditions and measured parameters for current density

Parameters	Values		
Current intensity (mA)	150	200	300
Current density (A/m <sup>2</sup> )	27.77	37.03	55.55
Operating time electrocoagulation (min)	80	80	80
Charge loading (C/L)	480	640	960
SDS removal efficiency %	67.01	93.3	97.83
$F^-$ removal efficiency %	73.58	89.07	94.21
Electrical voltage (V)	7.2	8.7	11.4
$C_{\text{energy}}$ (kWh/m <sup>3</sup> )	0.957	1.54	3.03
Anode consumption (kg/m <sup>3</sup> )	0.044	0.059	0.089

keeping the other parameters constant. It can be observed from Fig. 6a that removal of SDS (initial concentration: 60 mg/L) decreases from 4.02 to 1.85 mg/L with the decreases in initial fluoride concentration from 100 and 50 mg/L, respectively. It should be noted that a total elimination of SDS was obtained for the lower fluoride concentration (30 mg/L).

Similar trends are also observed in Fig. 6b. The fluoride concentration (initial concentration: 100 mg/L) decreased to 9.25, 10.93 and 23.44 mg/L when the initial SDS concentration changed from 30, 60 and 100 mg/L, respectively. However, fluoride standard limit was not reached for an SDS concentration of 100 mg/L. The improvement of the operating conditions is then necessary. The results obtained also

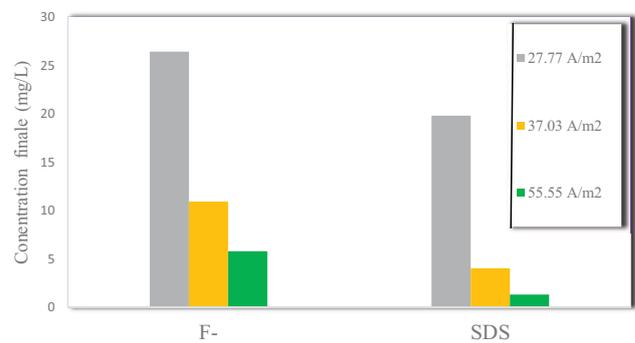


Fig. 5. Effect of current density on the removal of fluoride and sodium dodecyl sulfate:  $[SDS]_0 = 60$  mg/L,  $[F^-]_0 = 100$  mg/L, pH = 7,  $t = 80$  min, and electrodes: 2Al-2Fe.

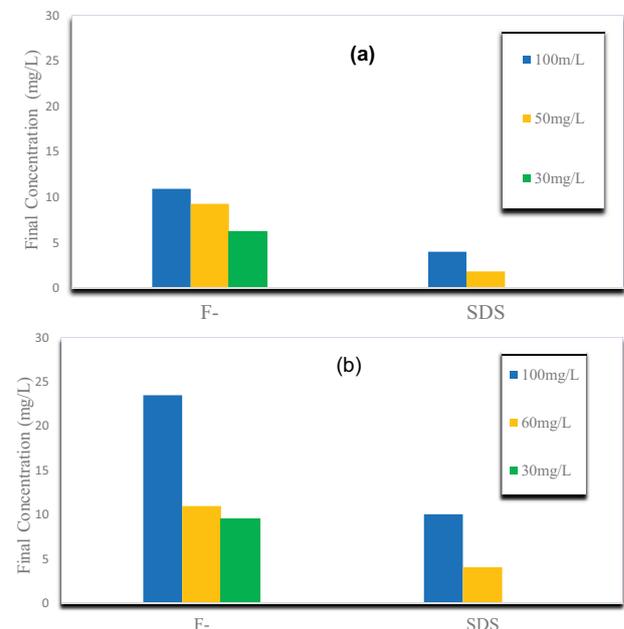


Fig. 6. (a) Residual concentration of  $F^-$  and sodium dodecyl sulfate as function of different fluoride initial concentrations:  $[SDS]_0 = 60$  mg/L, pH = 7,  $i = 37.03$  A/m<sup>2</sup>,  $t = 80$  min, electrodes: 2Al-2Fe. (b) Residual concentration of  $F^-$  and sodium dodecyl sulfate as function of different sodium dodecyl sulfate initial concentrations:  $[F^-]_0 = 100$  mg/L, pH = 7,  $i = 37.03$  A/m<sup>2</sup>,  $t = 80$  min, electrodes: 2Al-2Fe.

revealed lower is SDS concentration, better is the removal efficiency. It is also evident from Fig. 6a and b, that removal of both SDS and fluoride is responsive to alteration of their initial concentration.

#### 4. Conclusion

In this study, the EC process was successfully used in removing organic SDS and inorganic fluoride simultaneously from synthetic photovoltaic wastewater thanks to the use of hybrid electrode Al-Fe. The suggested process has great potential for alternative to conventional PV effluents. Some conclusions can be drawn from experimental results.

The residual concentration of pollutants decreases with the extension of electrolysis time, and the optimal electrolyse time was 80min.

The results show that the best fluoride and SDS removal efficiencies are 89.07% and 93.3%, respectively at initial pH of 7.

The optimal current density was 37.03 A/m<sup>2</sup> for an operating times of 80 min. Under these optimal conditions, the electrode consumption is 0.059 kg-electrode/m<sup>3</sup> and the power requirement was 1.54 kWh/m<sup>3</sup>.

Removal of both SDS and fluoride is responsive to alteration of their initial concentration.

EC-Al is more suitable for F<sup>-</sup> removal and EC-Fe is more suitable for SDS removal.

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