

Full Paper

Effect of Electrocoagulation Parameters on Chromium Removal, Sludge Settling, and Energy Consumption

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Abstract- Wastewater laden with toxic pollutants, such as chromium (Cr), can hurt humans, the environment, and aquatic life if not properly treated. The present work proposes to study the elimination of 100 mg/l of Cr(VI) ions from a synthetic wastewater effluent using a dynamic electrocoagulation process employing aluminum electrodes. The influences of several parameters are explored for the electrocoagulation process effectiveness in terms of Cr removal yield. This was done within the following ranges: current density (5-22 mA/cm²), initial pH (2-7), and chromium concentration (25-150 mg/l). A settling step was followed to determine the total suspended solids content and the sludge volume index after electrocoagulation treatment. Depending on the operating conditions, the data of specific electrical energy consumption were employed to determine the best operating parameters of the process efficiency. Furthermore, data analysis (Scanning Electron Microscope, Fourier Transform Infrared spectroscopy, X-ray diffraction analysis, and X-ray fluorescence) of sludge formed after electrocoagulation showed an amorphous nature with a high content of aluminum and function groups that should have a potential to be utilized as coagulant/sorbent in wastewater treatment.

Keywords- Chromium; Electrocoagulation; Sludge settling; Energy consumption; Aluminum electrodes; Wastewater

1. INTRODUCTION

One of the common problems of our time is pollution, which translates to the introduction of foreign substances into the environment resulting in adverse effects to nature and human health. Water pollution in particular occupies an important place in the world, because of the enormous development in industrial activities.

Among the many sources of pollution, the release of heavy metals has received special attention. Because of their persistence, metals cannot be reduced or eliminated but only transformed. Some of these metals are capable of being bio-accumulated in the trophic chain, leading to important public health problems [1].

Trace metals with high levels of pollution and toxicity at the trace level such as chromium.

Chromium compounds often exist in different forms. However, only the Cr(III) and Cr(VI) states are prominent in the environment, but they exhibit contrasting behaviors and toxicities. Hexavalent chromium Cr(VI) is toxic element and has a carcinogenic effect with considerable solubility in the aqueous medium [2]. In contrast, trivalent chromium Cr(III) is less toxic and low solubility in aqueous solutions. Cr(VI) is present in various industrial shears such as tannery and textile, which raises a great concern in relation to the wastewater discharged by these industries. This is why this metallic pollutant must be eliminated by an appropriate water treatment method such as adsorption [3], ion exchange [4,5] or reverse osmosis [6]. All have been used for the treatment of wastewater containing Cr(VI) [7].

Cr (III) is much less toxic and is known to be a micronutrient for human beings [8]. For this reason, the reduction of Cr(VI) to Cr(III) has drawn the attention in order to immobilize Cr(VI) in nature [9]. Precipitation, It is the most commonly used technique for Cr(VI) removal [10], in which Cr(VI) is first reduced to Cr(III) and then removed by precipitation. This method has some disadvantages, such as poor process control, high cost of chemical and the production of a large quantities of dangerous heavy metal waste [10].

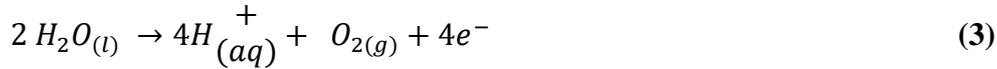
Electrocoagulation (EC), another electrochemical technique that offers numerous advantages, such as low amounts of sludge production, not harmful for environment, no chemical addition, and minimal area without reducing the quality of recovered water [11,12]. It has been successfully used for the elimination of variety range of pollutants [13]. Consequently, EC has the potential to significantly eliminate the drawbacks of conventional treatment techniques and could lead to a more sustainability in matter of polluted wastewater treatment economy [14].

The EC destabilizes suspended or dissolved contaminants in the aqueous medium by using a current in the medium and generating a coagulant in situ through electrolytic oxidation of the appropriate anode material (aluminum or iron). The reactions taking place in an electrochemical cell with the metal (M) as the “sacrificial electrode” can be represented as follows (1-2) [15]:

At the cathode:



At the anode:

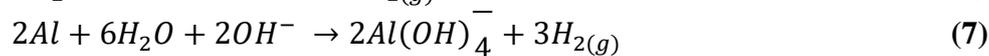


The M^{n+} ions generated by dissolution neutralize negatively charged impurities. The neutral entities thus formed are destabilized and can coalesce into larger, aggregates that can be separated. In addition, metal hydroxides may react with some dissolved compounds to form solids that can be caught by larger agglomerates and, thus, promote the treatment of the polluted medium by adsorption according to the pH of the medium [16]. In addition, the gas bubbles generated during electrolysis may support the pollutant to the top of the solution where it can be easily concentrated, collected, and removed [17]. So, the separation of the solid–liquid formed either by flotation or by settling is the last step of the treatment. In the case of aluminum electrodes subjected to a direct current, the main reactions occurring are the following:

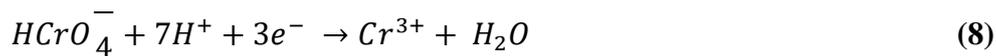
at the anode:



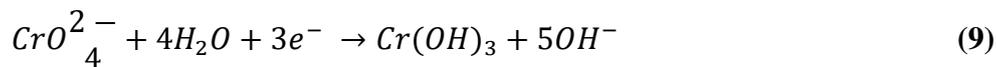
and at the cathode:



Hexavalent chromium can be also reduced to Cr(III) in contact with cathodes:



or



Previously, electrocoagulation (EC) was successfully used to eliminate chromium from wastewater [15,17]. The operating conditions for process optimization and the associated physico-chemical phenomena are described in detail in the literature.

Less studied are the effects of these conditions on the chromium removal yield, the solid content produced and sludge settling [19].

The present work was aimed to investigate the influence of the operating conditions such as current density, initial pH and initial Cr(VI) concentration on the Cr(VI) removal efficiency. Electrocoagulation processes generate sludge, which requires separation. Consequently the initial Cr(VI) quantity and its sedimentation properties are important parameters for predicting treatment success. To address this, a settling step was followed after

EC to determine the total solid content (TS) and sludge volume index (SVI). The obtained results were interpreted and compared for efficiency with respect to electrocoagulation operating conditions and the electrical energy consumed.

2. MATERIALS AND METHODS

2.1. Synthetic wastewater characteristics

A stock solution of 1000 mg/L Cr(VI) was prepared by dissolving the required quantity of potassium dichromate in distilled water. Lower concentration solutions were prepared by appropriate dilution. All chemicals were of analytical grade. The synthetic wastewater conductivity was adjusted by the addition of potassium chloride. The added amount of KCl was 1.5 g/L. The initial Cr concentrations of 100 mg/L and initial pH values around 5 were held constant across the tests.

2.2. Analyses of Cr(VI) ions

Hexavalent chromium reacts with diphenylcarbazide in a strongly acidic solution to produce a red-violet coloration that is detectable by molecular absorption spectrometry at 540 nm using a UV Visible spectrophotometer (Jenway 7315).

2.3. Experimental setup

Laboratory scale experiments were performed using a 1-liter cylindrical beaker maintained at the temperature of about 22 °C. Two parallel aluminum electrodes were placed inside the container. Each was about 15 cm long and 3 cm wide (area 45 cm²). The distance between the electrodes was set at 1cm to limit the ohmic voltage drop. The current density applied to the electrodes was determined by a power supply (Metrix-AX-502) and controlled with an ampermeter (SKY-Sronic-600-527) connected in series. The wastewater was continuously stirred in the recipient to ensure homogeneity in the recipient. The waste water was introduced to the bottom of the tank between the electrodes. The outlet was placed 8 cm from the top of the reactor to allow the liquid to circulate inside the reactor to support the contact time between the impurity and the hydroxide. The inlet and outlet flow rates were kept constant and equal using a peristaltic pump (Master Degree FlexL/SModel 77202-60) supplied with an identical cylinder head for two pipes. The vessel temperature was kept constant by heating/cooling water. Figure 1 illustrates the experimental setup.

A continuous recirculation system was used. The stirred reactor promoted homogenization of the effluent at low speed of (100 rpm) to avoid shearing the flocs and unintendedly facilitating their settling. Recirculation also inhibited the saturation of the effluent with dissolved metal at the electrochemical cell. This reactor enables a well homogenized effluent which is collected via a tap near the bottom of the apparatus. The agitation in this reactor also

allows the separation of the gases from the effluent and limits the formation of foam, which would otherwise hinder the electrocoagulation.

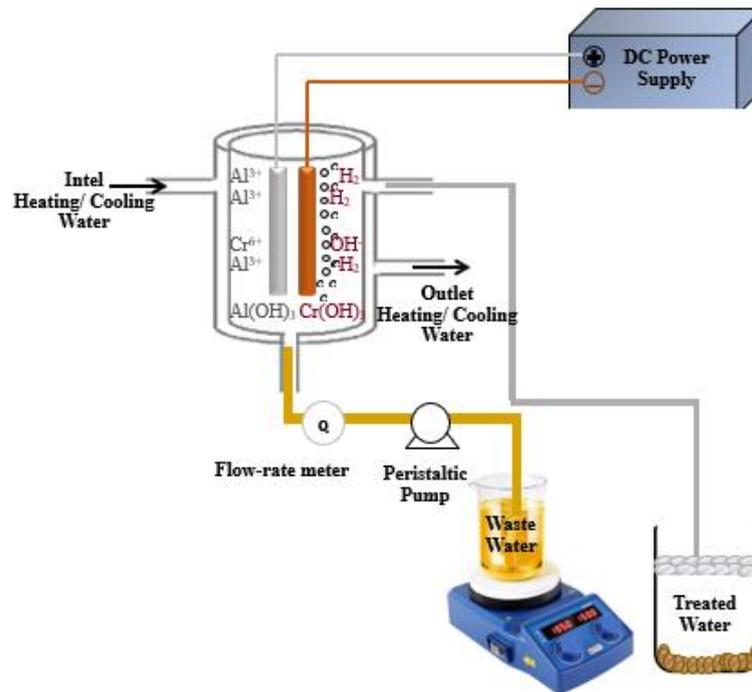


Figure 1. Experimental setup

During the one hour treatment time, 5ml of sample were taken, allowed to settle, filtered by Whatman 0.45 mm filters, and measured for Cr(VI) concentration. Equation (10) was used to calculate the percentage removal of Cr(VI), Equation 10.

$$R = (C_0 - C_f)/C_0 \quad (10)$$

C_0 : initial Cr concentration (mg/L)

C_f : final Cr concentration (mg/L)

R: percentage removal of Cr (%)

2.4. Estimation of operating cost

Operating cost is an elemental parameter in the consideration of any treatment method as it determines the viability of that method. For lab-scale electrocoagulation units, the cost of energy and electrode material are the major components of the operating expenses, which can be calculated using the following equation [20]:

$$\text{operating cost} = A \text{ Energy}_{\text{consumption}} + B \text{ Electrode}_{\text{consumption}} \quad (11)$$

A and B are the electrical energy and electrode material prices, respectively. The aluminum electrode material price is \$1.5/kg and the electrical energy consumption (kWh/m^3) and electrode consumption (kg Al/m^3) were calculated from the following equations [20]:

$$E = IUt/V \quad (12)$$

E: energy consumption in kWh/m³

U: The voltage in the cell in volts

I: the current in Ampere

t: The electrolysis time (min)

V: Volume of the solution (mL)

$$m = ItM/zFv \quad (13)$$

where:

m: anode consumption in (kg/m³)

I: the current (A)

t: the electrolysis time (s)

M: the specific molecular weight (g/mol)

z is the number of electrons involved in the reaction (z=3), and F is the Faraday's constant (96485 C/mol).

v: is the volume (m³) of the wastewater

Knowing that, for the Algerian market in February 2023 the electrical energy price is \$0.038/kWh for the first 125 kWh and \$0.03473/kWh beyond 125 kWh.

3. RESULTS AND DISCUSSION

3.1. Influence of current intensity

Current intensity is a major operating parameter that directly affects process performance and operating costs. The applied current is known to significantly affect the efficiency through the coagulant dosing rate [16,19].

According to Faraday's law (equation (13)), an increase in the current density leads to an increase in the coagulant dose per unit time. The process must therefore be accelerated. To evaluate current density effect, a series of runs were carried out with a constant initial concentration of Cr(VI) and with varying applied current densities ranging from 5 to 22mA/cm², with an inter- electrode distance of 1 cm and an initial pH value of 5.

3.1.1. Removal of Cr(VI)

Figure 2 shows the removal efficiency of Cr(VI) as versus time and current density. After the first 30 minutes, the removal was accelerated by increasing the current density from 5 to 11 mA/cm². Increasing the applied current leads to a larger quantity of sacrificial anode dissolution and, thus, the insoluble hydroxide ions of Al(OH)₃ formed in the solution and the reduction of Cr(VI) to Cr(III) at the cathode and its precipitation. However, applying current density higher than 11 mA/cm² after a period of 30 min leads to a slight decrease removal. Above this value, oxygen evolution reduces the solubility of aluminum while oxidation of Al³⁺

is enhanced. This means that not enough Al^{3+} ions are available for the reduction of Cr(VI). This optimal current density of 11 mA/cm^2 was used in the following electrocoagulation tests and achieved a yield removal of 78.38 %. Similar results were found by [21]. However, in that study, while the same Cr removal yield was achieved, the slower current densities required a longer duration.

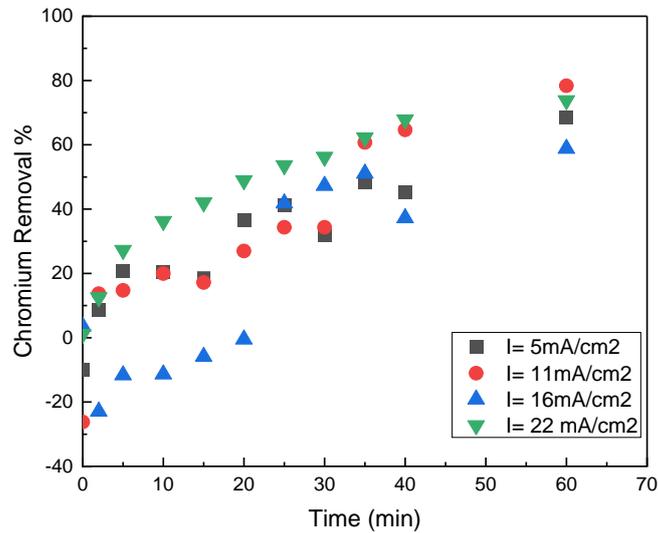


Figure 2. Removal efficiency of Cr(VI) versus time and current (optimal level shown with red circles)

3.1.2. Total solid content (TS)

Figure 3 shows the total solid content formed after EC, as a function of current densities ranging from 5 to 22 mA/cm^2 after a 30 min settling step. The TS increases considerably as current density rise until around 16 mA/cm^2 . For higher current densities, the degree of dissolution of the anode metal increases, resulting in more deposition.

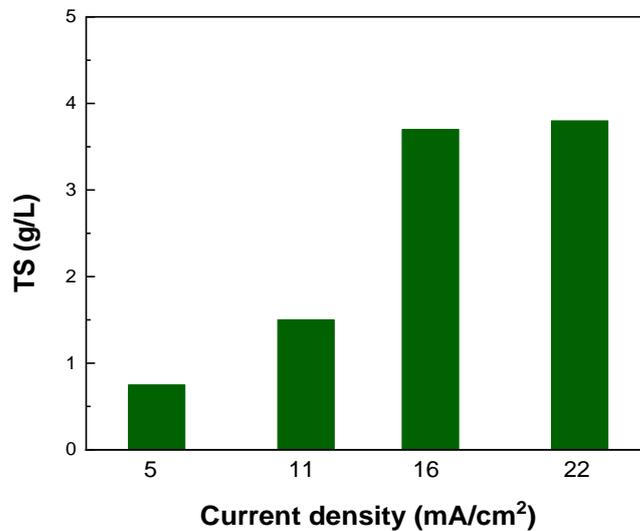


Figure 3. Effect of current density on the TS after EC

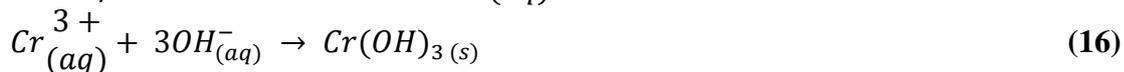
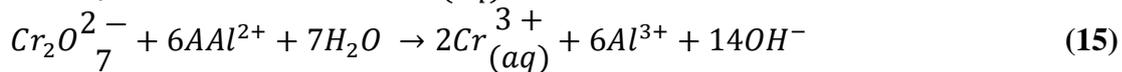
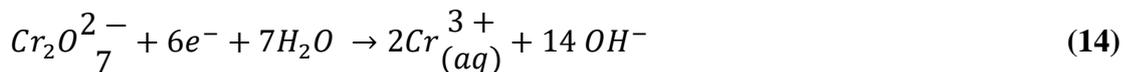
However, high concentrations of aluminum hydroxide can lead to significant gelation, which reduces the efficiency of the process. Similar results were obtained by [22]. In that study the amount of produced sludge was higher during the application of 32.52 mA/cm² current density when 16.26 mA/cm² was applied.

3.2. Influence of the initial pH

The role of pH is to play a significant effect in electrocoagulation [23]. In order to enhance the efficiency of chromium reduction in aqueous solution, several reactions should take place: production of Al³⁺ followed by production of insoluble Cr(OH)₃, reduction of Cr(VI) to Cr(III) and/or adsorption on Al(OH)₃. The speciation and solubility of these products is strongly dependent on the pH of the solution. Consequently, the Cr(VI) removal efficiency was tested for initial pH values of 2, 5 and 7 by adding 0.1 M HCl or NaOH. The applied current density of 11 mA/cm² and the initial concentration of Cr(VI) of 100 mg/l were kept constant.

3.2.1. Cr (VI) Removal

As illustrated in Figure 4, the Cr(VI) removal yield in acidic medium is about 44.52%. This increase to about 78.38% in a natural solution when pH=5.14. Beyond 5.14 the removal efficiency decreased to 61.15%. This could be explained by Eqs. (13) and (14), where an acidic environment is required for the reduction of Cr(VI) to Cr(III). After the reduction reaction, the pH of the aqueous medium increases and forms the insoluble compounds Cr(OH)₃ and Al(OH)₃ as in Eqs. (16) and (17).



The decrease of Cr removal at pH values less than 4 and higher than 8 was explained by Lai and Lin, 2004 [19] who demonstrated that precipitation of Al(III) is at its optimum at pH=7. For Cr(III), the best pH is about 9 and for both chromium or aluminum when media is more alkaline the soluble M(OH)₄⁻ formation is favored. Lai and Lin, 2004 [19] reported that chromium removal may be done either by precipitation after its cathodic reduction (Eq 8) or by adsorption on the aluminum hydroxide, Adhoun et al, 2004 [7] showed that electrocoagulation using aluminum electrodes allowed removal of chromium (VI) with a good performance in the pH range 6-8. Other results conform with those reported herein where the optimum solution pH ranged from 4 to 6 [13,23]. Consequently, the pH in the wastewater did not need to be adjusted for the proposed treatment.

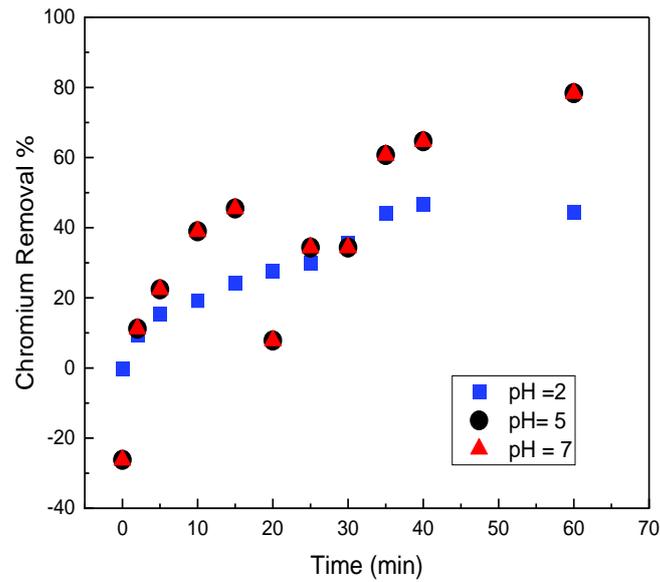


Figure 4. Cr(VI) removal efficiency versus time and initial pH of the effluent

3.2.2. Total solid content (TS)

The initial pH effect of the Cr(VI) effluent on TS after electrocoagulation is shown in Figure 5. The TS increases with higher pH level, reaching a greater mass of 2 g/L at neutral medium pH= 7. This pH interval does not favor the formation of Al^{+3} in solution nor that of Cr^{+3} but favor the existence of solids: $\text{Al}(\text{OH})_3$ and $\text{Cr}(\text{OH})_3$, which is why the dry matter content is important. The TS content formed under acidic pH conditions is practically negligible because of the predominance of ionic species in the medium.

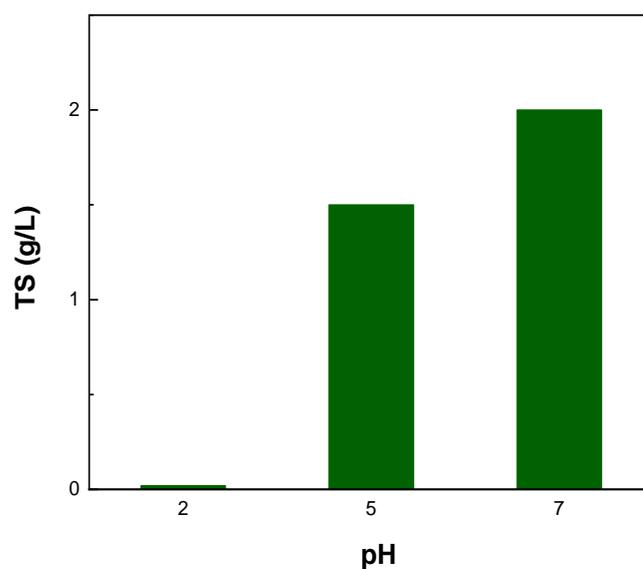


Figure 5. Influence of pH on TS formed after EC

3.3. Influence of initial Cr(VI)

The initial concentration of Cr has a significant impact on the course of the electrocoagulation process by the dose of the coagulant [24]. The initial concentration range from 25 mg/L to 150 mg/L were considered with Al electrodes under constant conditions of natural solution pH and current density of 11 mA/cm².

3.3.1. Removal of Cr(VI)

To examine the influence of initial Cr(VI) concentration present in the effluent on the removal yield, concentrations of 25-150 mg/L were treated under a natural initial pH solution. Figure 6 shows the yields of Cr(VI) removal for each initial Cr(VI) concentration at different times of electrolysis.

Generally, the EC process efficiency reduced as the initial concentration of Cr(VI) increased [25], as shown Figure 6. This is caused by the treatment of concentrated solutions needing more Al dissolved than that for more dilute solutions.

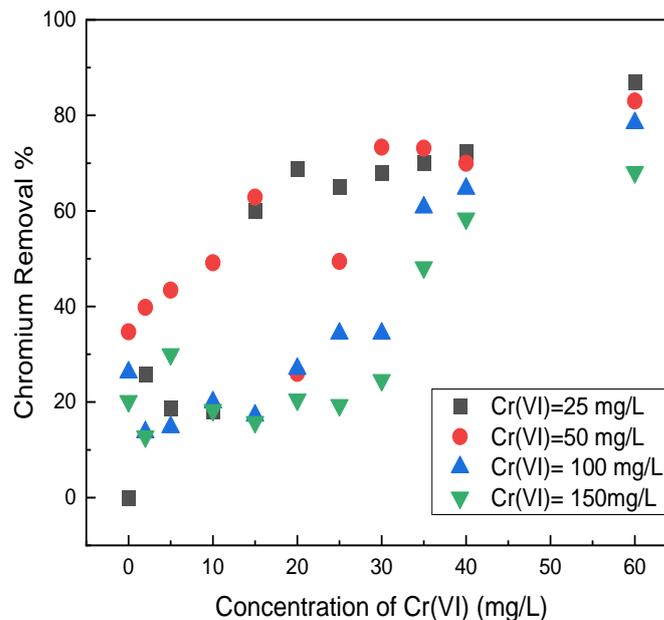


Figure 6. Effect of initial Cr(VI) concentration on its removal by EC process

3.3.2. Total solid content TS

The influence of variations in the initial Cr(VI) concentration on the sludge settling after electrocoagulation is shown in Figure 7. The increase of initial concentration of Cr(VI) corresponded to a decrease in solid content.

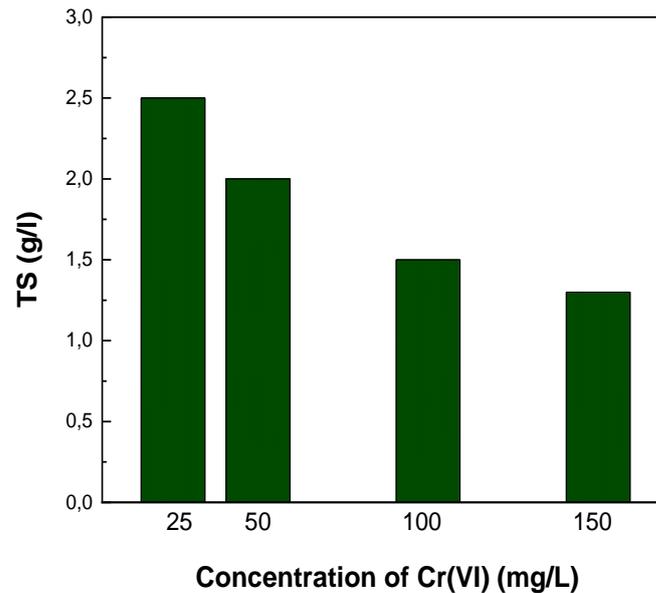


Figure 7. Influence of initial Cr(VI) concentration on sludge volumes produced after EC

3.4. Sludge settleability

This section investigates the processes during sludge settling both after chemical oxidation or and after EC. By definition, settling is the separation by gravity of two immiscible phases, of which at least one is liquid. Sedimentation can be expressed by the Molhman Sediment Index (5MI) or by the Sludge Volume Index (SVI) [25]. The SVI is usually required to characterize the formation of sludge and is defined considering a precise point on the settling curve, namely $t=30$ min according to Eq. (18):

$$MI\left(\frac{\text{mL}}{\text{g}}\right) = \frac{V_{30}}{TS} \quad (18)$$

where V_{30} is the volume of settled sludge after 30min settling (mL/L) and TS the total solid of the waste after electrocoagulation treatment period (g/L). Sludge with good settling properties has an SVI of 100 g/mL. In the presence of bulking phenomenon, this can be as high as 500 g/mL [26].

Figures 8–10 illustrate the SVI variations versus various operating parameters in the EC process, such as current density, initial pH, and initial Cr(VI) concentration, respectively. Figure 8 shows SVI variation as a function of current densities from 5 to 22 mA/cm². Here, the SVI increases considerably with the greater levels of current density. The lowest value at 95,32 mL/g was obtained with 11 mA/cm², whereas the highest of 132 mL/g was reached with 22 mA/cm².

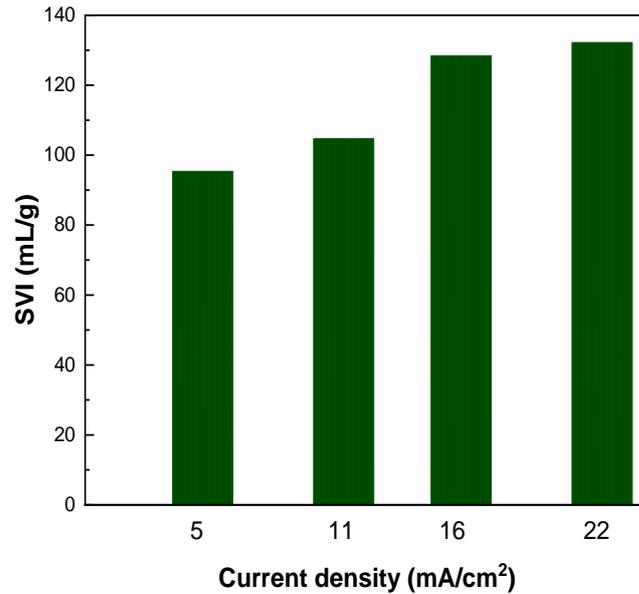


Figure 8. Effect of current density on SVI values

The performance of sludge settling in relation to the SVI at different initial pH values is shown in Figure 9. SVI values increased in acidic solution (pH= 2). The SVI values then decreased with increasing pH and reached 104.66 mL/g at pH= 5.14.

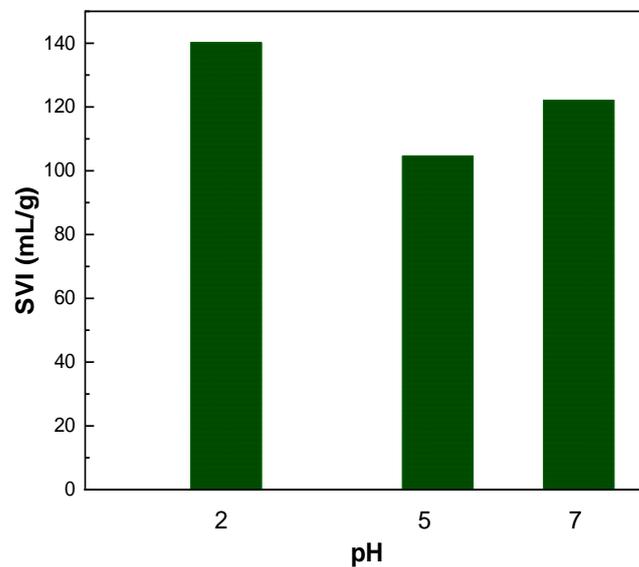


Figure 9. SVI at various values of initial pH

For the EC-treated effluent containing different concentrations of Cr(VI) ions (Figure 10), more rapid settling was obtained with lower concentrations of chromium. SVI values range from 54.57 to 160.78 mL/g for an initial concentration in the range of 25-150 mg/L.

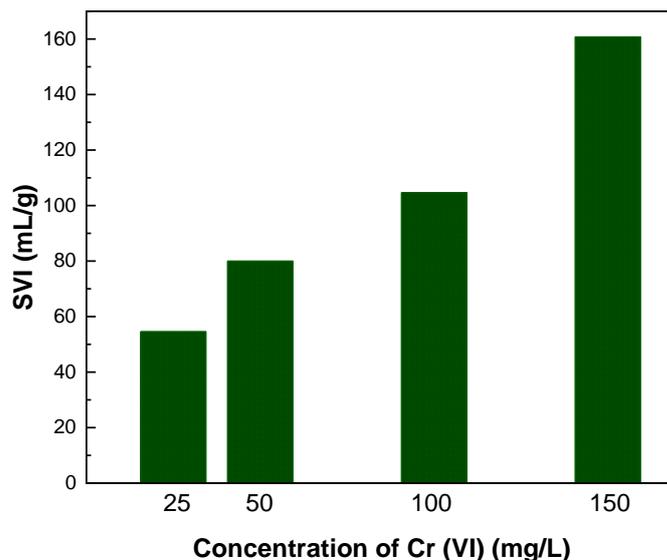


Figure 10. SVI at various values of initial Cr(VI) concentration

3.5. Results of optimum operational conditions and operation cost

In this study, calculation of operating cost was done only for the optimal operating conditions via the formula (11). Energy and electrode consumption were evaluated based on treating 1 L of synthetic wastewater containing 100 mg/L of Cr(VI) over a period of 60 min at a current density of 11 A/cm². This achieved a removal yield of chromium of 78.38%. The associated electrical energy consumed was 2.25 kWh/m³ which corresponds to 0.07273€/m³ operational costs including those of electrode material consumption (0.016790 kg/m³) energy consuming ($E_{\text{consumption}}=2.5$ kWh/m³) and more efficient in Cr(VI) reduction, with the additional benefit of not needing to adjust the pH solution. This value remains reasonable and within the range of $E_{\text{consumption}}$ reported previously by Cherifi et al. [18]. for the treatment of the Cr(VI) ions by EC.

3.6. Characterization of the sludge formed during electrocoagulation

The characterization of the sludge produced after electrocoagulation treatment is required to evaluate the possibility of the reuse and recovery. For this purpose, the sludge recovered after Cr(VI) removal by electrocoagulation using aluminum electrodes was analyzed by Scanning Electron Microscope (SEM), Fourier Transform Infrared Spectroscopy (FTIR), X-ray diffraction analysis (XRD) and X-ray fluorescence (XRF). Based on the elemental analysis of the sludge achieved through XRF (Table 1), aluminum is the element present in the largest amounts. This is caused by the degradation of the aluminum electrodes during the treatment process. The presence of chromium in the sludge, knowing well that Cr(VI) is an element highly insoluble, confirmed the Cr removed from the effluent during after electrocoagulation reaction and its displacement to the formed sludge. Other detected elements

such as potassium, silicates and other trace metals come from adsorption of conductive electrolytes (KCl), the chemicals used in the treatment ($K_2Cr_2O_7$) and residual impurities formed at the surface electrodes.

Table 1. Elemental analysis of produced sludge after EC process

Element	Al ₂ O ₃	K ₂ O	Cl	Cr ₂ O ₃	SiO ₂	CuO	CaO	MgO	ZnO
%	73.7	12.2	4.96	4.55	1.63	0.779	0.675	0.366	0.167

The SEM image shown in Figure 11 illustrates the structure of the sludge, which is dense with an irregular and porous surface containing a very fine particular structure at μm size accumulated in the form of aggregates. The XRF confirmed the presence of an abundance of aluminum in the amorphous phase in the chemical speciation of aluminum hydroxide and/or aluminum oxyhydroxide. Previous research [27] reported that crystallization process of Al hydroxide/ oxyhydroxide was very slow and that the aluminum oxide layer does not layer does not contain a pure compound of crystalline aluminum but instead contains a compound of amorphous aluminum [28].

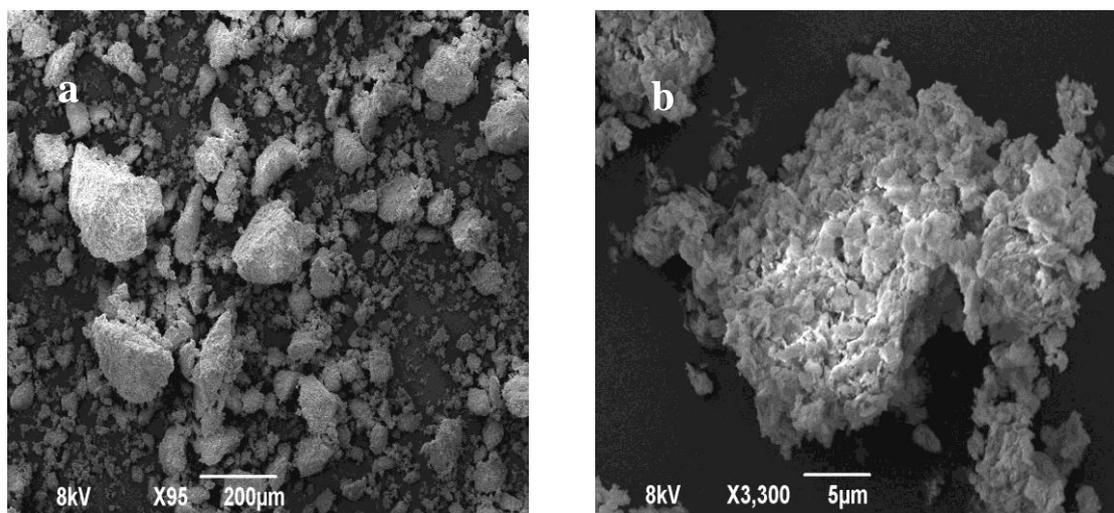


Figure 11. SEM images of sludge generated during EC process at (a) 95 X (b) 3300 X magnification

The FTIR spectra were obtained to show the functional groups on the sludge surface (Figure 12). The large and high vibration about 3437.95 cm^{-1} indicates the presence of amino and hydroxyl groups on the surface of adsorbent [29]. The sorption peak around 2927 cm^{-1} was assigned to CH stretching [30]. The amine group is present at NH_2 at 2362.33 cm^{-1} , and the peak at $1634\text{ (}1519.80\text{)}\text{ cm}^{-1}$ is related to stretching vibrations of $\text{C}=\text{C}$ in aromatic rings or $\text{-N}=\text{N}$ stretching [31]. At 1022.54 , the band is associated to C-O stretching vibrations [29].

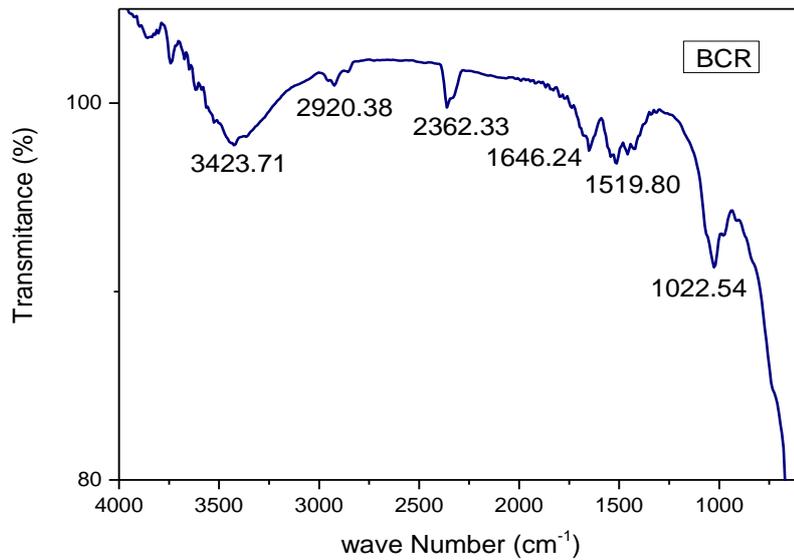


Figure 12. FTIR spectra of sludge Cr(VI)

Crystal composition of sludge was established using XRD to allow the determination of the mineral compounds which constitute the sludge sample (Figure 13). The powder XRD pattern shows very large and flat diffraction peaks. Bragg reflections with very large protuberances and low intensity show that the analyzed phase has a short-range order (i.e. amorphous or very weakly crystalline). Their diffractograms show that the mineral matter of this sludge is mostly composed of Illite $(KAl_2Si_3AlO)_{10}(OH)$ and chlorite $(Mg,Fe)_3(Di,Al)_4O_{10} \cdot (Mg,Fe)_3(OH)_6$, as well as some minor components such as mansfeldite, albite $(NaAlSi_3O_8)$, quartz (SiO_2) , hematite, and calcite $(CaCO_3)$ [31,32]. Some peak values corresponding to 25.4° , 38.5° and 44.8° 2θ indicate complex oxides of Al and Cr formation [32,33].

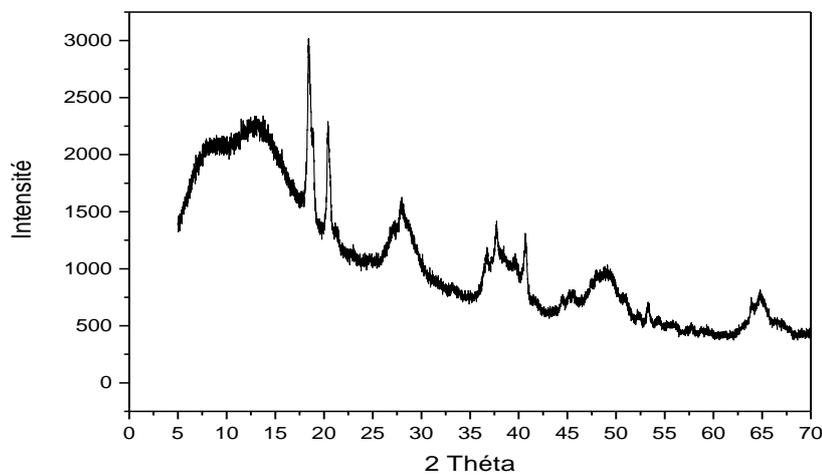


Figure 13. XRD spectra of sludge Cr(VI)

Average, physico-chemical analysis of the sludge obtained during electrocoagulation show that oxygen, aluminium, potassium, chlore and chromium are the main components and could be valorized. In previous studies, sludge with high contentment of aluminum was used as coagulant or as a sorbent [34,35]. Shen et al. and Cherifi et al. [36,37] demonstrated the effectiveness recovery of aluminum and chromium from waste water sludge by both chemical or electrochemical techniques. Wang et al. [39] studied the use of alum sludge for forming aluminum hydroxide and layered double hydroxide.

4. CONCLUSION

Treatment of synthetic wastewater containing 100 mg/L of Cr(VI) was conducted in a continuous electrocoagulation vessel with aluminum electrodes. Current density, pH, Cr(VI) concentration were studied to optimize operational conditions in terms of Cr(VI) removal, solid content generation, and treated effluent settling velocity. Based on those experimental tests, the following points can be highlighted:

- During EC process, while increasing current density enhances Cr removal, smaller currents can generate similar results but with longer treatment durations. An acidic medium hinders the decrease of Cr(VI) to Cr(III), while a pH range of 5-7 enhances the removal, because $\text{Al}(\text{OH})_3$ and $\text{Cr}(\text{OH})_3$ dominate in this pH range. Under the variables tested, the maximum Cr(VI) removal with Al electrodes was 78.38%.
- Significant amounts of suspended solids were observed after treatment with higher applied currents. This leads to greater levels of anodic dissolution and, thus, more electro-generated Al species. A moderate current density of $11\text{mA}/\text{cm}^2$ was chosen as the optimal condition to achieve them most chromium elimination with only a moderate amount of solid content (TS= 1.5 g/L). Acidic conditions slowed the TS and lowered chromium removal.
- The settling velocity increased when the current densities increased. According to Cr removal and the amount of suspended solids, the treatment process was more efficient with a moderate applied current. This achieved rapid settling with a SVI value of 104.66mL/g
- The EC energy consumption process in linked to the current. This was calculated to be $2.5\text{KWh}/\text{m}^3$ with the moderate current density of $11\text{mA}/\text{cm}^2$ and a natural pH of the effluent.
- Physicochemical sludge analyses confirm the formation of aluminum and chromium content as a result of the EC process. This indicates that from such sludge metals could be recovered and that the sludge can be recycled to be used as a raw material.

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