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Chromium(VI) removal from industrial wastewater using hydrogen peroxide assisted electrocoagulation

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ABSTRACT

In this study, removal of hexavalent chromium, Cr(VI), from real industrial wastewater using hydrogen peroxide (H_2O_2) assisted electrocoagulation was investigated. The material for anode and cathode was iron. The impact of operating parameters including pH, current density and the H_2O_2 concentration on removal efficiency and energy consumption was explored in detail. The results showed that the chrome removal efficiency improved at acidic conditions (lower pH) and also with the increase of current density whereas the addition of H_2O_2 at any concentration decreased the removal efficiency. An initial Cr(VI) concentration of 518.9 mg/L was reduced to 0.523 mg/L after 60 min which was equal to overall removal efficiency of 99.92% when current density of 10 mA/cm^2 and pH 3.0 were applied while no H_2O_2 was added. At these conditions, 1.61 kWh/m^3 energy was consumed and the estimated operational cost was 0.803 USD/m^3 of wastewater. Our findings showed that the electrocoagulation was an effective treatment method for chromium rich wastewater but the hydrogen peroxide did not improve the outcomes.

Keywords: Electrocoagulation; Hexavalent chrome; Hydrogen peroxide; Fenton effect

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1. Introduction

Wastewater with toxic metal ions like chromium, nickel and zinc are harmful to the health if discharged without treatment. Many technologies have been developed to reduce the concentration of toxic metal ions in wastewaters such as membrane separation, biosorption, ion-exchange and electrocoagulation (EC). Treatment of wastewater by EC has been widely practiced for various industrial wastewaters like dairy wastewater [1], textile wastewater [2], pulp and paper [3], toxic metal ions from plating industry wastewater [4] and battery industry wastewater [5]. EC has also been found as an effective and economical method for toxic metals abatement from wastewater [6–9]. Heidmann and Calmano applied EC using iron electrodes where removal process at low electric currents was found much more efficient [10]. Vlachou et al. used both iron and aluminium electrode where interestingly the combination of two metals resulted in higher Cr(VI) and Ni removal (76 and 82% respectively) [11]. Application of Fenton reagent along with EC process was found beneficial over the traditional EC process when applied for landfill leachate treatment [12]. This positive effect is reported in many studies when wastewaters with high organic content were treated [2,13,14]. Malakootian and friends applied Fenton process on a nickel containing wastewater and reported a 98% removal [15]. Zhao and friends published on H_2O_2 oxidation followed by the anodic Fenton process for efficient treatment of electroplating wastewater containing toxic metal like chromium and organics [16]. Shih et al. applied Fered-Fenton and chemical precipitation process for the treatment of elec-

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trodes nickel plating wastewater [17]. Hamdan and friends has implemented EC for treatment of groundwater with a successful 100 % removal efficiency [18].

Cheballah et al. [19] reported on simultaneous removal of hexavalent chromium and COD from industrial wastewater with 99% Cr and 95% COD removal where 22.07 kWh/kg direct energy was consumed. The precipitated Cr(VI) in sludge was reused as a stable pigment at high temperatures for ceramics paint in a research elsewhere [20].

Authors didn't find a report among the literature on the hydrogen peroxide assisted EC for treatment of Cr(VI) containing electroplating wastewater. In addition, treating a chromium rich wastewater with no organic content is not addressed in the previous works using neither solely Fenton process nor in a combination of Fenton process with other techniques.

In this study, use of hydrogen peroxide assisted EC for treatment of a real industrial wastewater with hexavalent chromium ions is investigated. The article presents the removal efficiency along with the fate of consumed energy in the above mentioned combination. An estimation of operational cost is also presented.

2. Electrocoagulation and Fenton effect

EC is a method in which the coagulant is produced in situ by a sacrificing anode which inherently results in the formation of flocs of sizes greater than pollutants. The pollutants are trapped in these flocs and settle down as sludge that can be discharged as solid waste leaving treated water [21]. With iron used as a sacrificial anode, in an electrolytic system, iron hydroxides including ferric and ferrous hydroxides are produced. The main reactions during EC are as follows [22];

Mechanism 1

Anode:

$$4Fe_{(s)} \rightarrow 4Fe^{2+}{}_{(aq)} + 8e^- \tag{1}$$

$$4Fe^{2+}{}_{(aq)} + 10H_2O_{(l)} + O_{2(g)} \rightarrow 4Fe(OH)_{3(s)} + 8H^+{}_{(aq)} \tag{2}$$

Cathode:

$$8H^+{}_{(aq)} + 8e^- \rightarrow 4H_{2(g)} \tag{3}$$

Overall:

$$4Fe_{(s)} + 10H_2O_{(l)} + O_{2(g)} \rightarrow 4Fe(OH)_{3(s)} + 4H_{2(g)} \tag{4}$$

Mechanism 2

Anode:

$$Fe_{(s)} \rightarrow Fe^{2+}{}_{(aq)} + 2e^- \tag{5}$$

$$Fe^{2+}{}_{(aq)} + 2OH^-{}_{(aq)} \rightarrow Fe(OH)_{2(s)} \tag{6}$$

Cathode:

$$2H_2O_{(l)} + 2e^- \rightarrow H_{2(g)} + 2OH^-{}_{(aq)} \tag{7}$$

Overall:

$$Fe_{(s)} + 2H_2O_{(l)} \rightarrow Fe(OH)_{2(s)} + H_{2(g)} \tag{8}$$

In addition, the generated Ferrous ion can reduce Cr(VI) to Cr(III) at both acidic and alkaline pH, but precipitates as $Cr(OH)_3$ at higher pH according to the following reactions [22]:

Under basic conditions;

$$CrO_4^{2-}{}_{(aq)} + 3Fe^{2+}_{(aq)} + 4H_2O_{(l)} + 4OH^-{}_{(aq)} \rightarrow 3Fe(OH)_3\downarrow + Cr(OH)_3\downarrow \tag{9}$$

Under acidic conditions:

$$Cr_2O_7^{2-}{}_{(aq)} + 6Fe^{2+}_{(aq)} + 14H^+{}_{(aq)} \rightarrow 6Fe^{3+}{}_{(aq)} + 2Cr^{3+}{}_{(aq)} + 7H_2O_{(l)} \tag{10}$$

Hydrogen peroxide is one of the strongest oxidant after fluorine with an oxidation potential of 2.8 V [23]. Addition of H_2O_2 readily increases the number of hydroxyl ions available for EC to take place. The complex phenomena of the effect of hydrogen peroxide is presented in the following reactions [23]:

$$S + H_2O_2 \leftrightarrow S^+ + OH^- + OH^\bullet \tag{11}$$

$$S^+ + H_2O_2 \leftrightarrow S + H^+ + HO_2^\bullet \tag{12}$$

$$S^+ + HO_2^\bullet \leftrightarrow H^+ + S + O_2 \tag{13}$$

$$S + HO_2^\bullet + H^+ \leftrightarrow S^+ + H_2O_2 \tag{14}$$

$$S + HO^\bullet \leftrightarrow S^+ + OH^- \tag{15}$$

In the above reactions, letter S is the solid electrode material used. The above mentioned mechanism, describes reactions on the electrode surface while there are many complex heterogeneous reactions taking place in the solution. Because of the reductive nature of the treatment here, i.e. the reduction of Cr(VI) to Cr(III) and then its precipitation according to Eqs. (9) and (10), the main effect of produced strong radical oxidants might be through Fentonic synthesize of ferrous and ferric ions and hydroxides as stated in Eqs. (11)–(15) which result in production of coagulants according to Eqns. (2), (4), (6) and (8). It is direct oxidative effects on the pollutants as is expected when dealing with organic wastewaters [23,24] which is not expected to be significant here. This has been discussed in more detail in next sections.

3. Materials and method

3.1. Characterization of wastewater

The wastewater used in this study was obtained from a wastewater treatment facility, in Eskisehir, Turkey, which receives wastewaters from many metal plating industries. The initial concentration of Cr(VI) was evaluated using ICP Optical Emission Spectroscopy (ICP OES, Varian 720-ES). All the reagents used were of analytical grade. The initial concentration of Cr(VI) was found as 518.9 mg/L, pH of wastewater was 3 and the conductivity of the wastewater was 4.2 mS/cm.

3.2. Electrocoagulation procedure

A cylindrical vessel made of iron with dimension of 10 cm in diameter, 10 cm in height and 0.5 cm of thickness was used as cathode. The anode was an iron stirrer with 4 rectangular blades, each blade being 3.5×7 cm^2 wide and 0.2 cm thick. There were 14 holes with a diameter of 12 mm punched in a regular pattern on the blades. The total available anode area was 94 cm^2. The sample was agitated at 50 rpm. A sketch of the set-up is shown in Fig. 1. The volume of sample used in every experiment was 800 ml. A SatronDC power supply with operating range of 0–45 V/0–50 A was used to maintain constant power supply. The pH value of wastewater was measured using OHAUS Starter 3000 pH meter and the pH values were adjusted using either 0.1 N NaOH or 0.1 N H_2SO_4 solutions. The conductivity of the wastewater was measured using in oLab conductivity meter. Upon preliminary tests it was observed that an electrolyte must be added to initialize the reaction. Na_2SO_4 was used as the electrolyte at a concentration of 0.1 M. The conductivity of the solution before and after adding the electrolyte was 4.2 mS/cm and 18.643 mS/cm respectively. The electrode was washed with dilute H_2SO_4 solution prior to each experiment. Samples of 10 ml were collected periodically and filtered using 2 μm (MACEHREY-NAGEL 640de) filter paper to remove the sludge residue. The concentration of metal ions was obtained using ICP OES.

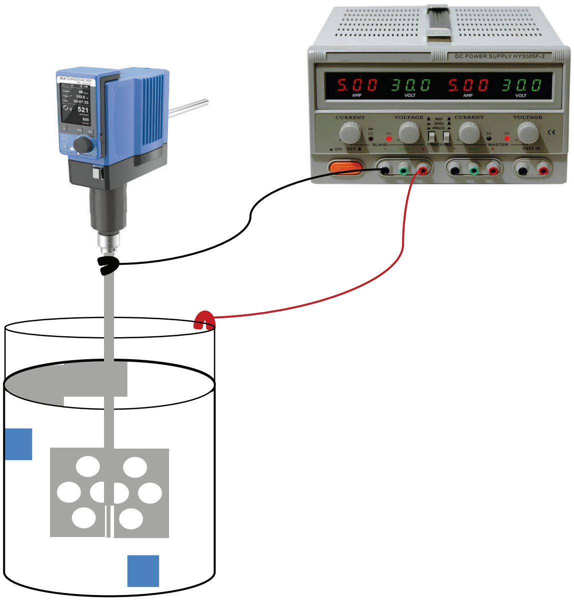

Fig. 1. Experimental set-up.

3.3. Calculations

The electrical energy consumption is one of the most important parameters in the evaluation of EC process. The energy consumption was calculated using the following formula [25]:

$$E = \frac{U \cdot I \cdot t}{V} \tag{16}$$

where E is the energy consumption in kWh/m^3, U is the applied voltage (V), I is the current, t is the time in h and V is the volume of the sample.

4. Results and discussion

4.1. Effect of wastewater pH

The initial pH of wastewater was found to have profound influence on the performance of EC process [26]. Lower acidic pH helps the reduction of Cr(VI) to Cr(III) and also accelerates the dissolution of Fe so that the electrode passivation will be avoided. But on the other hand, a final alkaline pH value of solution will help the precipitation of Cr (III)and also Fe(III) according to Eq. (9) [27]. In our study the effect of initial pH in the range of 2–4 on the EC process was investigated at a current density of 10 mA/cm^2 with Na_2SO_4 electrolyte concentration of 0.1 M. Hydrogen peroxide was added and well mixed with the solution to maintain 0.1 M concentration before applying electric current. The solution pH was then adjusted to the desired value. The obtained result is presented in Fig. 2a. The removal efficiency at pH 2, 3 and 4 was 66.55%, 99.92 and 99.55% respectively after a 60-min reaction. It was observed that a decrease in the pH as low as 2, had negative effect on the overall performance of the system. On the other side, increasing pH from 3 to 4 did not alter the overall removal efficiency. The results for the pH progress (Fig. 2b) revealed that pH almost continuously increased, which is expected because of continuous synthesize of hydroxide ion according to Eqs. (7) and (15). Moreover, starting with the strong acidic pH 2 resulted in the final pH 3.39 which was still highly acidic and prevented the formation of $Fe(OH)_3$ flocs and the precipitation of Cr(III) according to Eq. (9). Similar results reported by Arroyo and friends where they investigated EC without addition of H_2O_2 for removal of Cr(VI) [27]. In this study, further experiments carried out at initial pH 3.

4.2. Effect of current density

Current density plays an important role in determining the performance of an EC process. The current density is one of the most influencing parameters in the formation of first bubbles in EC process [28]. In this study, the effect of current density was determined at 5 mA/cm^2, 10 mA/cm^2 and 15 mA/cm^2 with adjusted initial pH of 3, Na_2SO_4 electrolyte concentration of 0.1 M and H_2O_2 concentration of 0.1 M. The results for the effect of current density were as shown in Figs. 3 and 4. The obtained graph (Fig. 3) reveals three distinguishable stage of the process. There is a semi lag phase at initial times, a fast reaction rate step in between and a reduced rate at the higher reac-

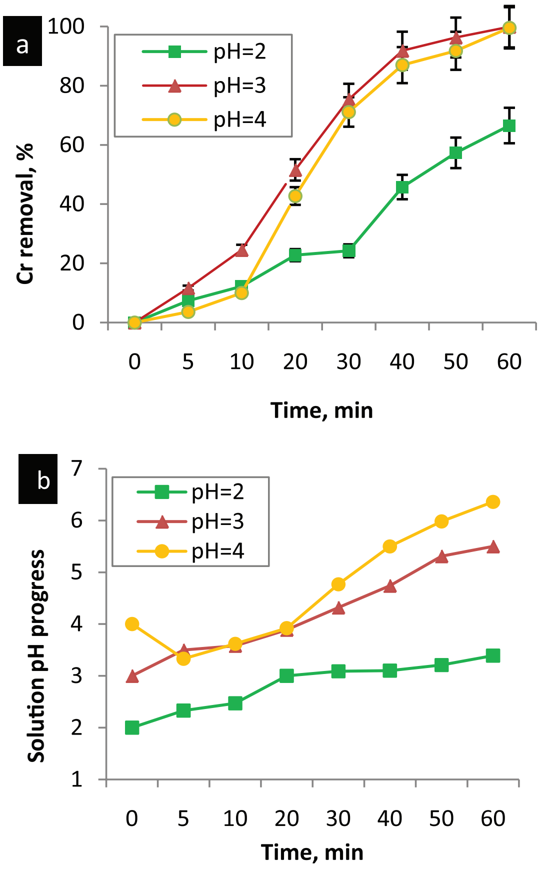

Fig. 2. (a) The effect of initial pH on Cr removal efficiency. I: 10 mA/cm², 0.1 M Na_2SO_4. (b) The pH progress.

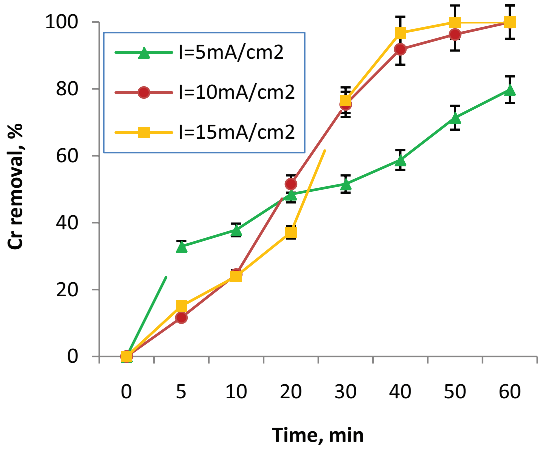

Fig. 3. The effect of the current density on the Cr removal efficiency. pH: 3 , 0.1 M Na_2SO_4.

tion times. It is understood from Eqs. (9) and (10) that the removal rate is dependent on both available Cr ions and the produced Fe ions. At the beginning the produced Fe ions are few while Cr concentration is high. It is concluded that the rate depends more on Fe concentration in the solution at the initial step. On the other hand, at

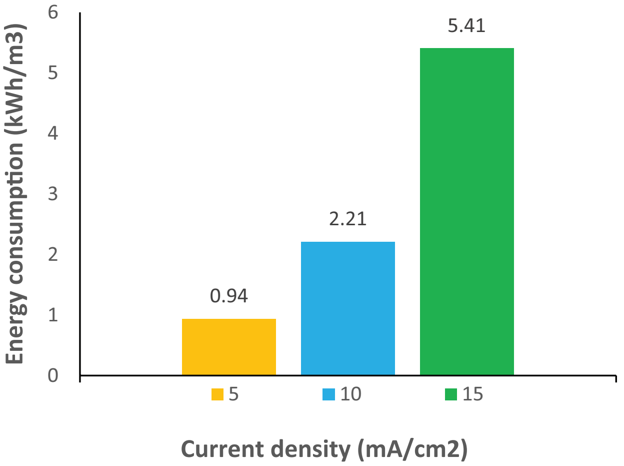

Fig. 4. Energy consumption of EC for different current densities, pH: 3 , 0.1 M Na_2SO_4.

the higher times most of the Cr ions removed and the rate reduced again where the process rate is controlled by Cr concentration. The removal efficiency and energy consumption increased with an increase in current density. The removal efficiency at 5 mA/cm², 10 mA/cm² and 15 mA/cm² were 79.74%, 99.92% and 99.97%, respectively. The removal efficiency at 10 mA/cm² and 15 mA/cm² were almost the same but the results for consumed energy, as depicted in Fig. 4 was considerably different where total consumed energy after sixty minutes of reaction was 2.21 and 5.41 kWh/m³ respectively for 10 and 15 mA/cm² current densities. In order to reduce the overall energy consumption, the current density value of 10 mA/cm² was the more favorable value and was used for the remaining experiments.

4.3. Effect of H_2O_2 (Fenton effect)

In this study the Fenton effect was studied at four different concentrations of H_2O_2 including 0.05 M, 0.1 M, 0.15 M and no addition of H_2O_2 at a pH of 3 and current density of 10 mA/cm². The obtained results for the removal efficiency and the energy consumption is shown in Figs. 5 and 6. It can be seen clearly that the addition of H_2O_2 lowers the rate of Cr(VI) removal significantly for all tested concentrations although the reaction was almost completed (~100% removal) after 60 min of reaction. The final total chromium concentration for 0.05 M, 0.1 M, 0.15 M and no addition of H_2O_2 were 0.48, 0.42, 10.01 and 0.52 mg/l respectively. According to the standards of Turkish Ministry of Environment and Urbanization for electroplating industry, the maximum allowed concentration of total chromium at the discharge is 1 mg/l. So with the exception of the 0.15 M value, the rest of the values felt under the maximum allowed level. The results for energy consumption of the reaction in Fig. 6 reveals that the addition of hydrogen peroxide has increased the consumed energy. The addition of hydrogen peroxide did not improve the removal efficiency which can be interpreted as the consequence of two concurrent opposite phenomena; one of them is the positive effect of Fenton

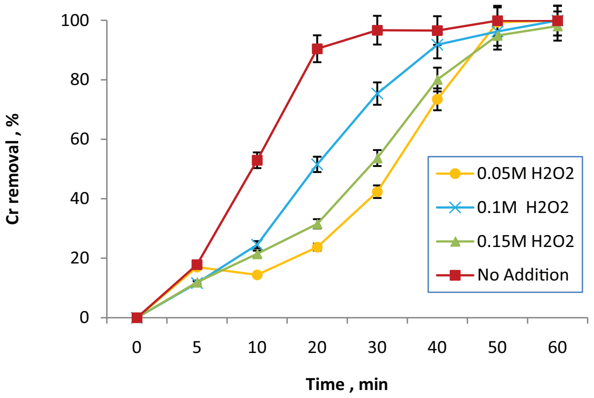

Fig. 5. The effect of H_2O_2 concentration on the Cr(VI) removal efficiency.

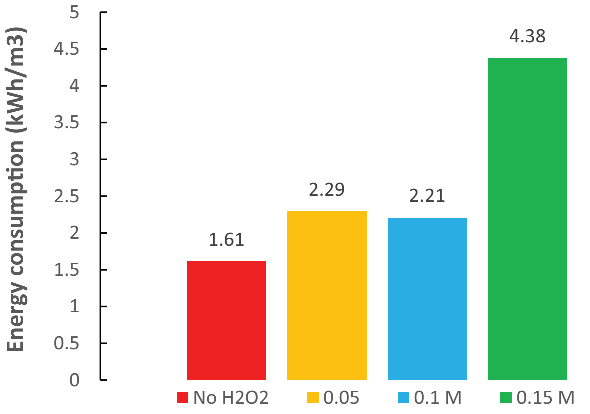

Fig. 6. The effect of H_2O_2 concentration on the EC energy consumption.

reaction on Fe anode which according to Eqs. (11)–(15) is expected to enhance the result, and the negative effect is the high oxidative power of produced hydroxyl radicals which prevents the reduction of Cr(VI) to Cr(III). But the removal mechanism in EC is not solely direct chemical precipitation to conclude zero removal rate in the cases as for example for Cr(VI). As can be seen from obtained data (Fig. 5), it did not stop the reaction or reverse it. In addition, the addition of hydrogen peroxide will promote floc formation which is favorable in EC. These findings revealed that the combination of EC with Fenton effect is not successful for Cr(VI) removal.

The s-shape of the removal progress curve with the time for all four cases are similar. The lower removal rate at the beginning can be attributed to lower concentration of dissolved iron at anode which is responsible for flocs formation. After almost ten minutes the removal rate has increased significantly and reduced again at higher times because of reduced concentration of Cr(VI) in the solution. The process kinetics cannot be explained by either a first nor a second order kinetic model because the process progress depends on the applied current as well, which directly affect the dissolved iron ions. This was also concluded by Al-Shannag et al. [4].

4.4. Operational cost estimation

The cost of any proposed process is the key factor to decide between multiple choices. Here the best found condition was taken as reference for cost estimation. The main source of operational expenditure supposedly are electricity cost and dissolved iron electrode [29]. The operational electricity consumption was 1.61 kWh/m^3. The amount of dissolved iron was estimated using Eq. (17) using the faraday's law.

$$MassFe = \frac{M \cdot t \cdot I}{z \cdot F \cdot V} \tag{17}$$

where in this equation; I is current intensity (A), t is the time (s), M is the molecular weight of iron (g/mol), z is the number of transferred electrons as in Eq. (5), F is Faraday's constant equal to 96500 Cb/mol and finally V is the wastewater solution volume (m^3).

The current density of 10 mA/cm^2 when applied to the anode electrode with a total area of 94 cm^2 was used in Eq. (17), the mass of dissolved iron equals to 1.23 kg/m^3 wastewater. The steel price in Turkey by January 2019 was 0.53 USD/kg. The electricity price for the same time period was 0.094 USD/kWh. Therefore, the expenditure for electricity and iron loss would respectively be 0.151 USD/m^3 and 0.652 USD/m^3. It is revealed that the main cost will be spent for iron electrode supply. Totally, treatment of each cubic meter of Cr(VI) contaminated wastewater using EC would cost 0.803 USD.

5. Conclusions

In this study, the effectiveness of H_2O_2 assisted EC for removal of hexavalent chrome from electroplating wastewater was investigated. The effect of operating parameters like pH, current density and H_2O_2 concentrations were evaluated and the pH value of 3, current density of 10 mA/cm^2 with no H_2O_2 addition were observed to result in the highest removal efficiency with the lowest energy consumption rate. In view of these results it can be concluded that the EC assisted by H_2O_2 is not an effective method for reduction of Cr(VI) from industrial wastewater. The operational cost including electric energy and iron loss from anode electrode for 100% Cr(VI) removal would cost 0.803 USD/m^3 of wastewater with initial concentration of 518.9 mg/l.

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