


## Article

# Electrochemical Removal of Chromium (VI) from Wastewater

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**Abstract:** The removal of hexavalent chromium has attracted much attention as it is a hazardous contaminant. Electrochemical reduction technology was applied to remove chromium (VI) from wastewater. The mechanisms and parameters that affect the reduction process were investigated. The results showed that the reduction efficiency was significantly affected by the concentration of  $\text{H}_2\text{SO}_4$ , current density, and reaction temperature. The reduction efficiency was up to 86.45% at an  $\text{H}_2\text{SO}_4$  concentration of 100 g/L, reaction temperature of 70 °C, current density at 50 A/m<sup>2</sup>, reaction time at 180 min, and stirring rate of 500 rpm. The reduction process of chromium (VI) followed a pseudo-first-order equation, and the reduction rate constant could be expressed as  $K_{\text{obs}} = k [\text{H}_2\text{SO}_4]^1 \cdot [j]^4 \cdot \exp^{-4170/RT}$ .

**Keywords:** electrochemical; chromium; kinetic

## 1. Introduction

In recent years, heavy metal pollutions like Cr, Ni, Cd, Pb, Hg, Zn, Co, and Cu—which mainly discharge from industrial wastewaters—have become a severe environmental issue [1–3]. As they attribute to toxicity, carcinogenicity, and natural-resource scarcity, the removal and recovery of heavy metals from wastewaters is an urgent matter [4]. Hexavalent chromium (Cr(VI)) is a high toxicity heavy metal contaminant that is widely released by various industries, such as the tanning and leather industries, manufacturing industries, catalysts and pigments, fungicides, ceramics, crafts, glass, photography, the electroplating industry, and corrosion control application. The International Agency for Research on Cancer (IARC) has classified chromium (VI) in Group 1 (carcinogenic to humans), and it is therefore a matter of great urgency to remove chromium (VI) from wastewater before its discharge into the aquatic system.

Many methods have been demonstrated to be efficient for Cr(VI) removal, such as: ion exchange [5,6], electrochemical treatment [7–9], chemical precipitation [10], coagulation [11], chemical reduction [12,13], and adsorption [3,14–17]. Some biological treatments [18,19] (phytoremediation [20]) are also applied to remove chromium (VI) from wastewater and groundwater. Lead sulfate as a precipitant can be used to precipitate chromium (VI) however, although it can remove chromium (VI) from 0.2 mol/L to 0.15 mmol/L [10], lead sulfate is a second pollutant which is harmful for environment. Additionally, difficulties and challenges such as high costs, large scale application, and causing secondary pollution still remain in the current technologies. It is necessary to overcome these challenges and develop new effective techniques for the removal of chromium (VI).

In a recent study [8], an electro coagulation-like technology, electro-reduction, was applied to reduce hexavalent chromium to trivalent chromium. The results showed that the chromium (VI) was easily being reduced by  $\text{Fe}^{2+}$  and the free electron, while the current intensity had little effect on the reduction process. In this paper, electrochemical technology was applied to reduce chromium (VI) in an

acidic medium. The mechanisms and parameters affecting the reaction—including the concentration of  $\text{H}_2\text{SO}_4$ , reaction time, reaction temperature, and current density—were investigated; the kinetic model was also simulated.

## 2. Materials and Methods

### 2.1. Materials

All reagents were analytical grade, including potassium dichromate ( $\text{K}_2\text{Cr}_2\text{O}_7$ ) and sulfate acid ( $\text{H}_2\text{SO}_4$ ), and were purchased from Kelong Co., Ltd., Chengdu, China. All solutions were prepared with deionized water with a resistivity greater than  $18 \text{ M}\Omega/\text{cm}$  (HMC-WS10).

### 2.2. Experimental Procedure

Reduction experiments were carried out in a 250 mL beaker fixed in a thermostatic water bath with a temperature precision of  $\pm 0.1^\circ\text{C}$ . In the batch experiments, 100 mL of solution containing 1.000 g/L chromium (VI) was prepared by dissolving a certain  $\text{K}_2\text{Cr}_2\text{O}_7$  in distilled water, and the acidic medium was prepared by adding different volumes of  $\text{H}_2\text{SO}_4$ , then the current supplied by a DC power supply was applied as the solution was heated to a predetermined temperature. During the experiments, the samples were collected at different intervals (5 min), and analyzed for the residual concentration of chromium (VI) in the solution [10]. The electrode used in the experiments was a plate-like dimensionally stable anode (Baoji Zhiming Special Metal Co., Ltd., Shanxi, China) with a surface area of  $1 \text{ cm}^2$  ( $1 \times 1 \text{ cm}$ ). A cathode with an identical surface area was fixed at a distance of 2 cm [21].

The concentration of chromium (VI) in the solution was determined by inductive couple plasma-optical emission spectrometry (ICP-OES) [22]. The reduction efficiency ( $\eta$ ) was calculated as Equation (1):

$$\eta = \frac{C_1 \cdot V_1 - C_2 \cdot V_2}{C_1 \cdot V_1}, \quad (1)$$

where  $C_1$  and  $C_2$  are the concentrations of chromium in the solution before and after the experiment, in g/L; and  $V_1$  and  $V_2$  are the volumes before and after the experiment, in L.

### 2.3. Kinetics Model

The reduction kinetic of chromium (VI) with electricity was investigated in order to establish the controlling step of reduction experiments [23–25]. Recent studies were studied extensively, and the controlling step was found to be the diffusion of chromium (VI) to the surface of the electrode. The reduction rate could be expressed as Equation (2):

$$v = \frac{dc}{dt} = -K_{\text{obs}}c, \quad (2)$$

which was integrated to make:

$$-\ln c = K_{\text{obs}}t - \ln c_0, \quad (3)$$

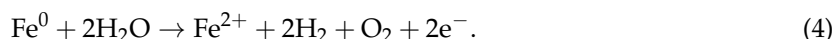
where  $c$  is the concentration of chromium (VI) at time  $t$ , in g/L;  $K_{\text{obs}}$  is the reaction rate constant which depends on fluid flow and reaction temperature conditions; and  $c_0$  is the initial concentration of chromium (VI) in the wastewater, in g/L.

### 3. Results and Discussions

#### 3.1. Electro-Reduction of Chromium (VI)

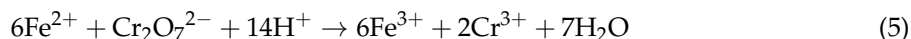
##### 3.1.1. Reaction Mechanism

The electro coagulation process of chromium (VI) [11,26,27] involved the following stages of reducing agent formation, and subsequently reducing chromium (VI) and chromium (III). The first step was the formation of  $\text{Fe}^{2+}$ , by the oxidation of a steel electrode by a DC power supply:

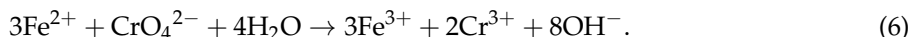


The second step was the reduction reaction between the reducing agent and oxidant. In this process, it was the reaction between  $\text{Fe}^{2+}$  and chromium (VI). The reactions are described in Equations (5) and (6), taking the pH of the wastewater into account.

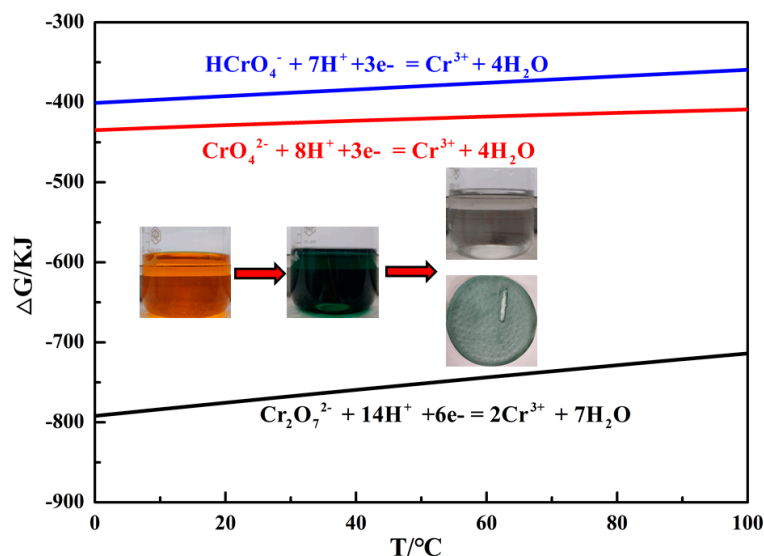
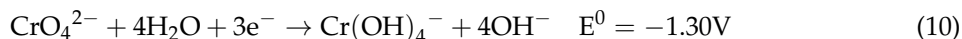
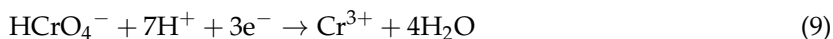
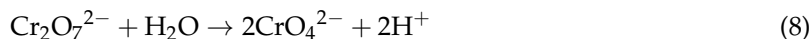
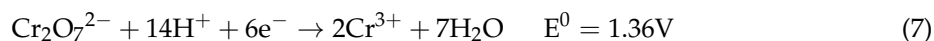
$0.5 < \text{pH} < 6.5$ :



$\text{pH} > 6.5$ :



In this work, the reduction process was different from electro coagulation, as the electrode used was stable and not being corroded. The reducing agent in this work was only a free electron ( $\text{e}^-$ ) supplied by the DC power supply. The reaction that occurred between the  $\text{e}^-$  and chromium (VI) is shown in Equations (7) to (10). The Gibbs free energy of the equations at different reaction temperatures was calculated, and the results are shown in Figure 1. The negative of  $\Delta G$  indicated that the reduction reaction was feasible in thermodynamics [28].



**Figure 1.** Relationship between  $\Delta G$  and the temperature of the reduction of chromium (VI).

### 3.1.2. Effect of Concentration of $\text{H}_2\text{SO}_4$

The effect of the concentration of  $\text{H}_2\text{SO}_4$  on the reduction efficiency was investigated, while other conditions—the initial concentration of Cr (VI) of 1.000 g/L, current density of  $50 \text{ A/m}^2$ , reaction temperature of  $70^\circ\text{C}$ , and stirring rate of 500 rpm—were kept constant. The concentration of  $\text{H}_2\text{SO}_4$  was set as 20 g/L, 40 g/L, 60 g/L, 80 g/L, and 100 g/L.

The results shown in Figure 2 indicate that the reduction efficiency of chromium (VI) increased as reaction time and acid concentration were increased. Also, the reduction process of chromium (VI) is acid-dependent and a higher chromium (VI) reduction efficiency could be achieved at a higher acid concentration. The acceleration of the Cr(VI) reduction in the system at higher acid concentrations could be attributed to the fact that the chromium (VI) species existing as  $\text{Cr}_2\text{O}_7^{2-}$  was more easily reduced in acidic conditions than in neutral/alkaline conditions. This can be seen in Figure 1, where the  $\Delta G$  was smallest according to Equations (7), (9), and (10) [8]. The chemical equilibrium of Equation (8) was destroyed and the reaction proceeded to the generation of  $\text{Cr}_2\text{O}_7^{2-}$  with the increasing acid concentration. Following this, the molar fraction of the  $\text{Cr}_2\text{O}_7^{2-}$  was drastically increased, which was beneficial for the reduction of chromium (VI). Therefore, a concentration of 100 g/L was selected for further experiments.

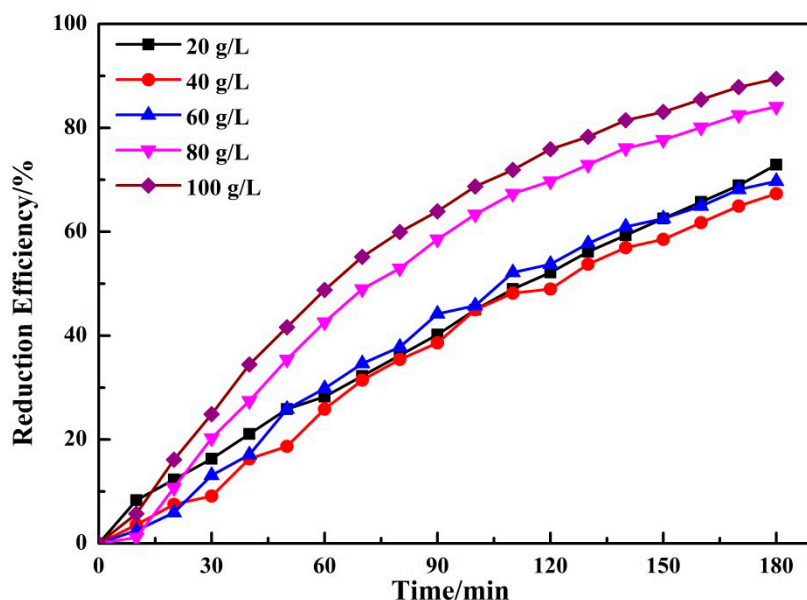


Figure 2. Effect of concentration of  $\text{H}_2\text{SO}_4$  on the reduction efficiency of chromium (VI).

### 3.1.3. Effect of Reaction Temperature

Reaction temperature is an important parameter affecting the reaction during the diffusion process [8,10,22,29,30]. The effect of reaction temperature on the reduction efficiency was investigated while other conditions—the initial concentration of chromium (VI) of 1.000 g/L, current density of  $50 \text{ A/m}^2$ , concentration of  $\text{H}_2\text{SO}_4$  of 100 g/L, and stirring rate of 500 rpm—were kept constant. The results shown in Figure 3 indicate that the reduction of chromium (VI) could be easily achieved at a higher reaction temperature ( $\geq 50^\circ\text{C}$ ), which was partially consistent with recent studies [8]. A higher temperature would decrease the diffusion resistance and favor the contact of the free electron and chromium (VI), resulting in a high reduction efficiency. Thus, a reaction temperature of  $70^\circ\text{C}$  was chosen to be the optimum.

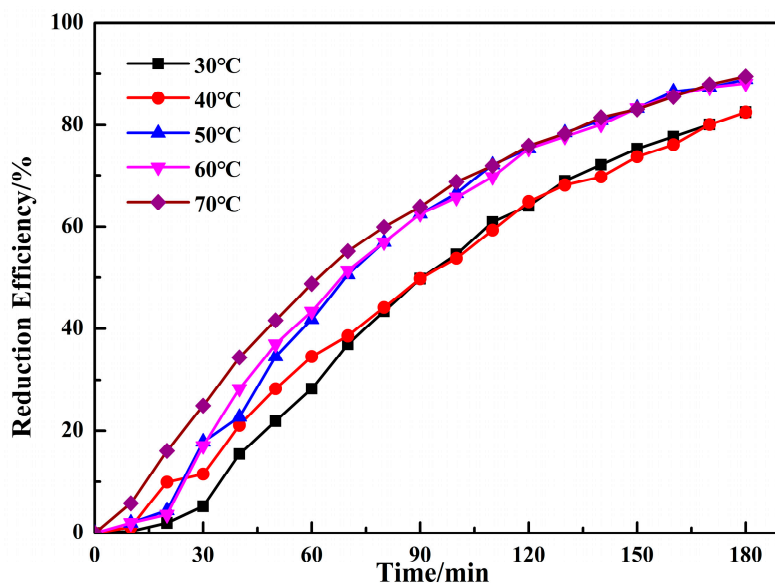


Figure 3. Effect of reaction temperature on the reduction efficiency of chromium (VI).

### 3.1.4. Effect of Current Intensity

The free electron supplied by the DC power supply was the main reducing agent which reduced chromium (VI) to chromium (III). The results shown in Figure 4 summarize the effect of current density on the reduction efficiency, while other conditions—the initial concentration of chromium (VI) of 1.000 g/L, concentration of  $\text{H}_2\text{SO}_4$  of 100 g/L, reaction temperature of 70 °C, and stirring rate of 500 rpm—were kept constant. It could be seen that the reduction efficiency increased when current density was increased, even though it was not increased by very much. The reduction efficiency was up to 86.45% at a current density of 50  $\text{A}/\text{m}^2$ . In other words, a higher current density could achieve a high reduction efficiency.

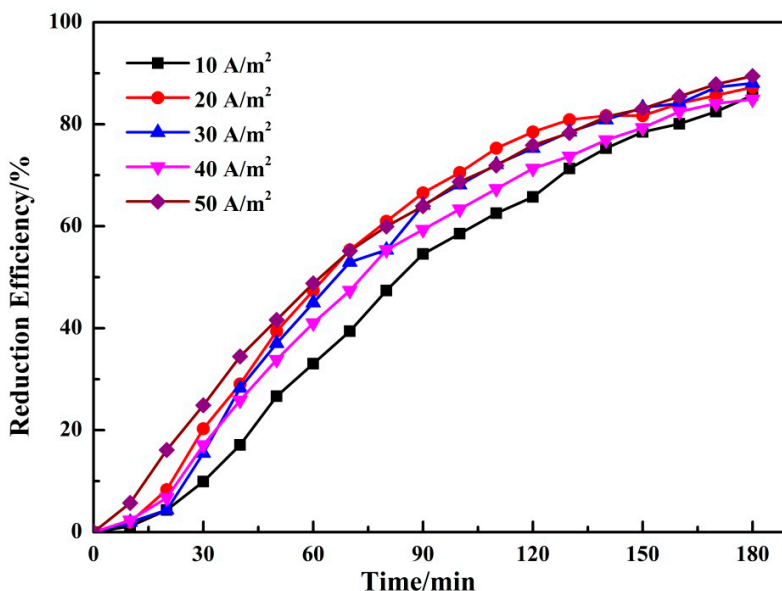


Figure 4. Effect of current intensity on reduction efficiency of chromium (VI).

### 3.2. Kinetic Model

The experimental data was analyzed using Equation (3), with the results displayed in Figure 5a–f and Table 1. The results shown in Figure 5a–c indicate that the reduction efficiency was well-fitted,

as the coefficients ( $R^2$ ) were all close to 1, which indicates that the kinetic model followed a pseudo-first-order model equation. The results shown in Figure 5a indicate that the reduction rate constant ( $K_{\text{obs}}$ ) increased when the concentration of  $\text{H}_2\text{SO}_4$  ( $[\text{H}_2\text{SO}_4]$ ) was increased. Figure 5d displays the relationship between  $K_{\text{obs}}$  and  $[\text{H}_2\text{SO}_4]$ . The results show that  $K_{\text{obs}}$  changed linearly with  $[\text{H}_2\text{SO}_4]$ —the relationship between them could be expressed as Equation (11). The effect of current density ( $[j]$ ) was a little complicated, with the  $K_{\text{obs}}$  shown to be multipower with  $[j]$  following Equation (12). The relationship between  $K_{\text{obs}}$  and the reaction temperature ( $T$ ) could be expressed with the Arrhenius equation (Equation (13)) and the specific apparent activation energy could be calculated. The results shown in Figure 5f show that the simulated Arrhenius equation and the  $E_a$  was calculated as 4.74 KJ/mol, so the Arrhenius equation could be changed to make Equation (14):

$$K_{\text{obs}} = 8.50\text{E}^{-5}[\text{H}_2\text{SO}_4] \quad (11)$$

$$K_{\text{obs}} = 3\text{E}^{-8}[j]^4 \quad (12)$$

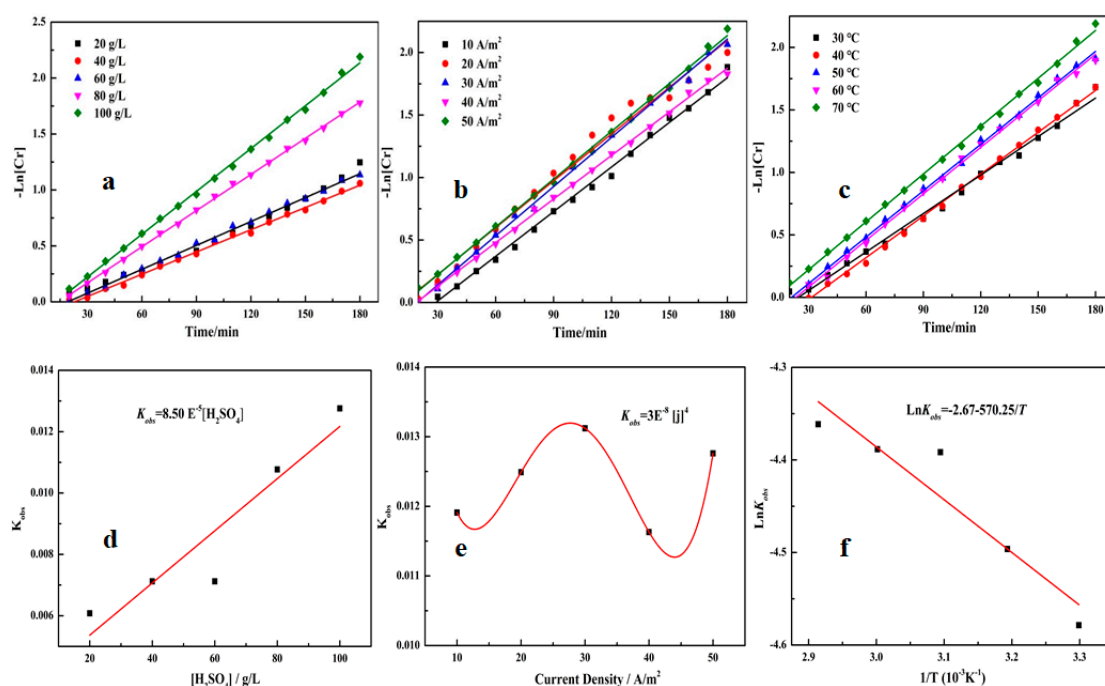
$$\ln K_{\text{obs}} = \ln A - E_a/RT \quad (13)$$

$$K_{\text{obs}} = Ae^{-4170/RT}, \quad (14)$$

where  $E_a$  is the apparent activation energy,  $A$  is the pre-exponential factor,  $R$  is the molar gas constant, and  $K$  is the reduction rate constant at different reaction temperatures.

In summary, the chromium (VI) could be efficiently reduced by electricity, and the reduction process was significantly affected by the concentration of  $\text{H}_2\text{SO}_4$ , current density, and reaction temperature. The reduction rate constant could be express as Equation (15).

$$K_{\text{obs}} = \frac{dc}{dt} = \frac{d[\text{Cr}]}{dt} = k[\text{H}_2\text{SO}_4]^1 \cdot [j]^4 \cdot e^{-4170/RT}. \quad (15)$$



**Figure 5.** Pseudo-first-order models for the reduction of chromium (VI). (a): Effect of  $\text{H}_2\text{SO}_4$  concentration; (b): Effect of current density; (c): Effect of reaction temperature; (d): Plot of  $K_{\text{obs}}$  as a function of  $\text{H}_2\text{SO}_4$  concentration; (e): Plot of  $K_{\text{obs}}$  as a function of current density; (f): Plot of  $K_{\text{obs}}$  as a function of reaction temperature concentration.

**Table 1.** Constants and correlation coefficients of pseudo-first order for reduction of chromium (VI).

	$K_{obs}$	$R^2$
[H <sub>2</sub> SO <sub>4</sub> ]		
100 g/L	0.01276	0.9985
80 g/L	0.01077	0.9993
60 g/L	0.00712	0.9978
40 g/L	0.00712	0.9849
20 g/L	0.00608	0.9964
Current density		
50 A/m <sup>2</sup>	0.01276	0.9985
40 A/m <sup>2</sup>	0.01163	0.9987
30 A/m <sup>2</sup>	0.01312	0.9975
20 A/m <sup>2</sup>	0.01249	0.9836
10 A/m <sup>2</sup>	0.01191	0.9946
Reaction temperature		
70 °C	0.01276	0.9985
60 °C	0.01242	0.9980
50 °C	0.01238	0.9983
40 °C	0.01115	0.9967
30 °C	0.01027	0.0017

#### 4. Conclusions

In this paper, the electrochemical reduction technology succeeded in reducing chromium (VI) from wastewater.

(1) The effect of some experimental parameters on the reduction efficiency of chromium (VI) was investigated, and the results showed that the reduction efficiency was up to 86.45% at an H<sub>2</sub>SO<sub>4</sub> concentration of 100 g/L, reaction temperature of 70 °C, current density at 50 A/m<sup>2</sup>, reaction time of 180 min, and stirring rate of 500 rpm.

(2) The reduction process of chromium (VI) followed a pseudo-first-order model equation, and the controlling step was the diffusion of chromium (VI) to the surface of electrode. The reduction process was significantly affected by the concentration of H<sub>2</sub>SO<sub>4</sub>, current density, and reaction temperature. The reduction rate constant of chromium (VI) to chromium (III) could be expressed as  $K_{obs} = k [H_2SO_4]^{1.1} \cdot j^4 \cdot \exp^{-4170/RT}$ .

(3) Electrochemical reduction technology was more likely but not the same as electro-coagulation. The reduction reaction occurred between the free electron and chromium (VI) without any other ions, and this technology was more environmentally-friendly than other removal methods.

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**Conflicts of Interest:** The authors declare no conflict of interest.

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