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Cadmium, Cyanide and Cadmium-Cyanide Removal from Wastewater Solutions by Electrocoagulation Process

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Abstract

Treatment of cadmium (Cd), cyanide (CN) and cadmium-cyanide (Cd-CN) wastewater solutions by electrocoagulation (EC) using iron electrodes have been investigated in this research. CN and Cd removals at current density of 30 A/m2 and EC time of 50 min from Cd-CN containing wastewaters were 99.70 and 99.52%, respectively; and 2.1746 $/m3 at operation cost at these EC conditions. EC gives CN removal efficiency of 99.81% and 2.5978 $/m3 operation cost for wastewater contaminated only by CN with 30 A/m2 and 50 min of process time. For wastewaters contaminated only by Cd, 99.8% removal efficiency, with 2.052 $/m3 operation cost under the same current density and process time. Approximately the same high removal efficiencies were achieved for each of the wastewater (Cd-CN, CN, Cd) used in the experiments. The highest removal efficiency of CN was achieved at pH 8.6 for wastewaters contaminated by CN or CN-Cd whereas the highest Cd removal efficiency was obtained at pH 10.6 for wastewaters contaminated by Cd. As a result, EC process gives quite high removal efficincies of Cd and CN for wastewaters coming from metal plating rinse baths.

Keywords: Cadmium and cyanide removal, Electrocoagulation, Operating cost.

1. INTRODUCTION

Hazardous wastes containing free and metal cyanides are generated in large volumes during various industrial activities such as electroplating processes and metal finishing and mining [1, 2]. The metal finishing industry typically uses highly alkaline cyanide plating baths, which consist mainly of heavy metal cyanide complexes (Ag, Au, Cd, Cu, Ni, Zn etc.) and free cyanide salts because of the brightness of the metal deposits and their good adherence [3]. Large of contaminated rinse waters are generated as a result of drag-out from the plating bath to the rinse water tank. In the presence of metallic compounds in the alkaline bath solutions, cyanide ion readily combines with the metal ion, Me^{z+}, to form a stable cyanide complex ion of the form, $[Me(CN)_y]^{(y-z)-}$ [4]. In this case the wastewaters which are generated during the rinsing of worked pieces bear the metal cyanide complexes. The treatment of wastewaters in the electroplating industry generates a strong concern related to environmental impacts due to contain toxic metal ions such as Cd, Cu, Au, Pb, Ni, Ag, and Zn ions, as well as acids, alkalis, and cyanide [5].

For complete treatment of wastewater containing metal-cyanide complexes, strategies for effective removal of both metals and cyanide must be specified. Therefore, the toxic and hazardous effluents that elimanate from the metal plating factories must be properly treated, so that they do not cause more damage to the environment.

Various techniques have been employed for the treatment of metal electroplating wastewaters containing heavy metals and cyanides, including alkaline-chlorination-oxidation, electrocoagulation, chemical precipitation, adsorption, ion-exchange, membrane processes, reverse osmosis and biological treatment [6]-[8]. Application of electrochemical processes for the treatment of industrial wastewater is gaining increasing attention [9], [10]. The EC has been

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successfully used to treat different industrial wastewaters [11]-[13]. EC is an effective technology for the treatment of heavy metal containing industrial wastewaters.

In the present work, the treatment of cadmium and syinade wastewater using a laboratory-scale electrocoagulation process is presented. The effect of initial pH, current density and operating time on the cadmium and cyanide removal efficiencies are explored and the optimium operational conditions were determined. In the EC process, the electrode and energy consumptions, and operating cost for each wastewater was also calculated.

2. A BRIEF DESCRIPTION OF EC PROCESS

EC is a simple and efficient method where the flocculating agent is generated by electro-oxidation of a sacrificial anode, generally made of iron or aluminium. In an EC process, the coagulating ions are produced in situ involving three successive stages: (i) formation of coagulants by electrolytic oxidation of the "sacrificial electrode", such as iron or aluminum, (ii) destabilization of the contaminants, particulate suspension and breaking of emulsions, (iii) aggregation of the destabilized phases to form flocs. Chen showed a conceptual sketch of the EC mechanism [10].The anodic reaction involves the dissolution of metal, and the cathodic reaction involves the formation of hydrogen gas and hydroxide ions [1]. Ferric ions generated by electrochemical oxidation of iron electrode may form monomeric species, $Fe(OH)_{3(s)}$ and polymeric hydroxy complexes namely, $Fe(H_2O)_6^{3+}$, $Fe(H_2O)_5(OH)^{2+}$, $Fe(H_2O)_4(OH)^{2+}$, $Fe_2(H_2O)_8(OH)^{24+}$ and $Fe_2(H_2O)_6(OH)^{44+}$ depending on the pH of the aqueous medium. These hydroxides/polyhydroxides/polyhydroxyoxide metallic compounds have strong affinity for dispersed particles as well as counter ions to cause coagulation. Contaminants present in the wastewater stream are treated either by chemical reactions and precipitation or physical and chemical attachment to colloidal materials being generated by the electrode erosion. In this study, iron electrode material was used as an anode produced iron hydroxide, Fe(OH)*n* where *n*= 2 or 3. During electrolysis, the wastewater solution became green and bubbles of gas seen at cathode during the EC process. The effluent became clear and a green and yellow sludge were formed. The green and yellow colours were attributed to Fe^{2+} and Fe^{3+} hydroxides [15]. The following shows the major reactions taking place in the EC reactor:

$$Fe_{(s)} \rightarrow Fe^{2+}_{(aq)} + 2e \quad \text{(Anode)} \tag{1a}$$

$$Fe^{2+} \rightarrow Fe^{3+} + e^- \quad \text{(Anode)} \tag{1b}$$

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

3. MATERIALS AND METHODS

3.1. Characterization of used wastewater samples

In this study, cadmium (Cd), cyanide (CN) and cadmium-cyanide (Cd-CN) wastewater solutions were prepared using a known quantity of NaCN ve $3CdSO_4.8H_2O$ compounds. All solutions were prepared with high quality pure water using Millipore Water Purification System. Physicochemical properties of the synthetic wastewaters before EC treatment are presented in Table 1.

Table 1. Physicochemical properties of the used wastewaters

Parameters	Cd-CN containing wastewaters	**CN containing wastewater**	**Cd containing wastewater**
pH	8.60	9.80	6.70
Conductivity (mS/cm)	0.96	0.494	0.730
CN^- (mg/L)	120	120	-
Cd^{2+} (mg/L)	102	-	102
TOC (mg/L)	60	45	65
COD (mg/L)	180	120	130

3.2. Experimental set-ups and procedure

The experiments were conducted in a Plexiglass batch reactor. The apparatus for EC consist of an electrolytic cell, which is a 1 liter plexiglas reactor with a dimension of 110 mm × 110 mm × 110 mm. It is equipped with thermostated for the temperature control. Cast iron (Fe) plates (50 mm × 53 mm × 3 mm) were chosen as the anode/cathode pair for their performances and the electrodes were situated 1.1 cm apart from each other and connected monopolar parallel mode. The electrodes' total effective electrode surface area was 159 cm^2. The electrodes were connected to a digital dc power supply (Agilent 6675A model; 120 V, 18A). All test runs in EC process were carried out at a constant temperature (22 $^{\circ}$C) and mixing speed (250 rpm). In each run, 650 cm^3 of the wastewater solutions was placed into the EC reactor. The current density was adjusted to a desired value and the EC process was started. At the end of EC, the solution was filtered and then was analyzed. At the end of the run, the

electrodes were washed thoroughly with 0.50 N HCl solution for 2 min and rinsed with deionized water, then dried in the drying-oven and were placed in a desiccator to cool down. The same process was also applied after each experiment.

3.3. Analytical methods

The cyanide concentration in the samples was determined using UV spectrophotometer (PerkinElmer Lambda 25 UV/VIS spectrophotometer, USA), the cadmium concentrations were measured in using the Inductively Coupled Plasma-Optical Emission Spectrometer (ICP-OES) instrument (Perkin Elmer, Optima 7000DV). [24]. The COD was analyzed according to Standard Methods [24]. The TOC were determined using a non-dispersive IR source (Shimadzu, TOC-L model). The accuracy of these measured values for cyanide, cadmium, nickel, COD and TOC was estimated around 3%. The pH and conductivity of the samples were measured by a pH and conductivity meter (Hach Lange HQ40). All the inorganic chemicals used were anaytical grade and all reagents were prepared in Millipore milli-Q deionised water.

3.4. Calculation of operating cost

The operating cost expressed as US \$/m^3 of wastewater treated includes material, mainly electrodes, electrical energy and chemicals used in the process [16].

$$\text{Operating cost} = a\,C_{\text{energy}} + b\,C_{\text{electrode}} + c C_{\text{chemicals}} \quad (1)$$

where C_{energy} (consumption kWh energy per m^3), $C_{\text{electrode}}$ (consumption kg electrode per m^3) and $C_{\text{chemicals}}$ (consumption kg chemicals per m^3) of wastewater treated. Unit prices, a, b and c given for the Turkish Market, July 2020, are as follows: (a) electrical energy prices 0.12 US \$/kWh, (b) iron electrode material price 0.952 US \$/kg, and (c) chemicals prices (NaOH and H_2SO_4 pireces 1.01 US \$/kg and 0.40 US \$/kg). Costs for Cenergy and for $C_{\text{electrode}}$ calculated from Faraday's law [16].

4. RESULTS AND DISCUSSION

4.1. Effect of operating parameters on treatment of CN-Cd containing wastewater

The effect of current density and pH on wastewater including Cd and CN was investigated depending on EC operation time. Figure 1 and 2 show removal efficiency and effluent concentration of CN and Cd at different current densities at pH 8.6. The removal efficiency of CN and Cd was 99% (0.21 mg/L) and 99.7% (0.31 mg/L) at current density 5 A/m^2 end of the process time of 100 min, respectively. At the same condition, 99.8% (0.12 mg/L) and 99.7% (0.29 mg/L) removal efficiency was obtained for CN an Cd at 10 A/cm^2, respectively.

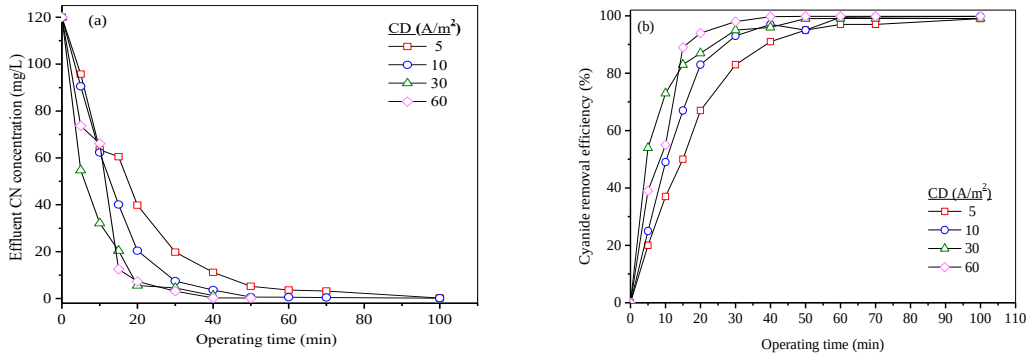

Figure 1. Effect of current density cyanide removal from the Cd-CN electroplating wastewater.

The CN and Cd concentration reduced almost 99.8% at the current density 30 and 60 A/m^2 with 50 min EC process. CN removal efficiency was achieved in the range of 94-95% at lower operating times (20 min) and higher current density (30-60 A/m^2). As a result, for cyanide removal from wastewater containing cadmium-cyanide (Cd-CN), desired cyanide removal efficiencies can be obtained at low operating times and high current densities or at low current densities and high operating time. On the other hand, it was observed that the cadmium removal was similar to the cyanide removal. However, it was determined that the cadmium removal efficiencies were lower than the cyanide removal efficiencies, especially at low EC operating times.

The energy and electrode consumption, which constitutes operating cost are an major issue in the EC process. It was calculated that as the operating time increased at different current densities, the energy and electrode consumption increased. It was observed that as the current density increased during the same operation period, the energy and

electrode consumption increased, especially over 30 A / m^2 current density. The electrode and energy consumption were 0.6437 kg/m^3 and 7.6442 kWh/m^3 at the current density of 30 A/m^2 end of the process time of 50 minutes, respectively. The operating cost was 2.172 \$/m^3 at the same conditions.

Water Municipal of Istanbul (Istanbul Water Sewage Administration: ISKI) regulations for releasing cadmium and cyanide in water to the sewage is below 2 and 10 mg/L, respectively. On the other hand, 92% cyanide and 98% cadmium remmoval efficiency from this Cd-CN wastewater must be achieved to meet the ISKI standards [17]. Accordingly, if we evaluate the EC results of wastewater containing Cd-CN, it was determined that as the operating time (t_{EC}) decreases, the current density increases in order to meet the outlet discharge limits. At this point, operating cost was increased. The operating cost was 4.131 \$/m^3 at the current density 60A/m^2 and t_{EC}=40 minute.

Another parameter affecting treatment of Cd-CN-containing wastewater by EC process is pH. Figıre 3 shows CN and Cd removal efficiencies and effluent concentration at conditions CD = 30 A/m^2 and t_{EC} = 30 minutes. When the initial pH of Cd-CN wastewater was between 5.6-10.6, removal efficiency was obtained 91.8% - 99.4% for CN and 89.6-99.2 for Cd; removal efficiency was achieved as 99.4-7.4% for CN and 99.2 - 33.5% for Cd at initial pH range of 10.6 - 12.6 during the operation period of 30 minutes. According to these results, it can be said that the initial pH of wastewater has an effect on performans of electrocoagulation of wastewater including Cd and CN. The removal efficiency increased at initial pH value between 5.6-10.6 and decreased over pH 10.6. It has been found that CN and Cd removal efficiencies are lower, especially at low pH. The effluent pH value was 6.6 when the initial pH value was 5.6 after EC process time 30 minutes. At this pH value, removal of pollutant was achieved by iron hydroxide precipitates to be formed by the EC process with adsorption and at high pH values Cd-CN complexes and $Cd(OH)_{2(s)}$ cadmium hydroxide precipitates contrubited to an incearse in removal efficiency. In case the initial pH is over 10.6, iron hydroxide complexes to be formed in the process are negatively charged complexes as $Fe(OH)_4^-$ and their precipitation is very difficult. Therefore, especially, at high current densities, these ions can adversely affect the process and cause corrosion. However, even if the Cd removal efficiency was low at these high pH values, 33.5% removal efficiency was achieved. It can be interpreted that cadmium is removed by precipitation, possibly in the form of cadmium hydroxide.

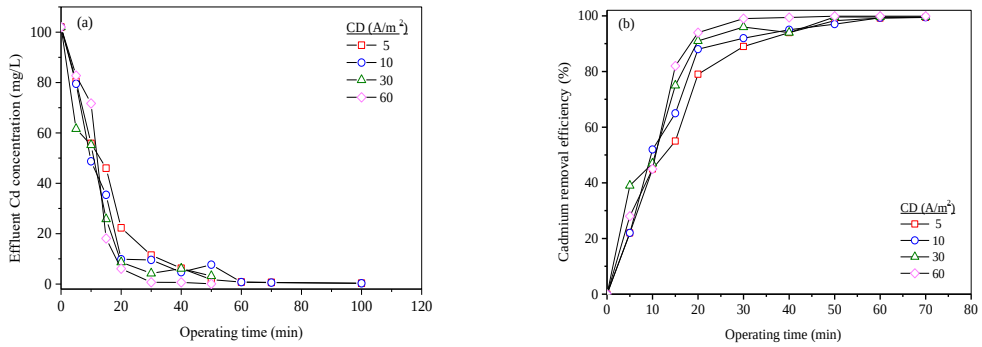

Figure 2. Effect of current density cadmium removal from the Cd-CN electroplating wastewater.

During the EC process, it was observed that the pH value increased continuously as the OH^- ions in the increased as a result of the reduction at the cathode. Coating wastewater containing Cd-CN generally has basic pH values. The initial pH of studied wastewater was 8.6. The effulent pH of the wastewater reached a pH value between 10.5 and 12.5 at a short time after the beginning of the process. It is an advantage of EC process due to forming $Cd(OH)_{2(s)}$ and precipitation with an incraese in OH^- ion concentration. As is known, depending on the pH of wastewater, different cadmium hydroxy species occur [15]. On the other hand, the cyanide removal efficiency decreased considerably at high pH. It indicates that the cyanide ions form highly stable complexes and the removal of these complexes is not affected by the pH of process too much, and the removal efficiency is low. Therefore, the cyanide removal efficiencies were higher than the cadmium removal efficiency below the initial pH 10.6. It can be concluded that the EC process would be effective at the initial pH range between 7 and 10.6.

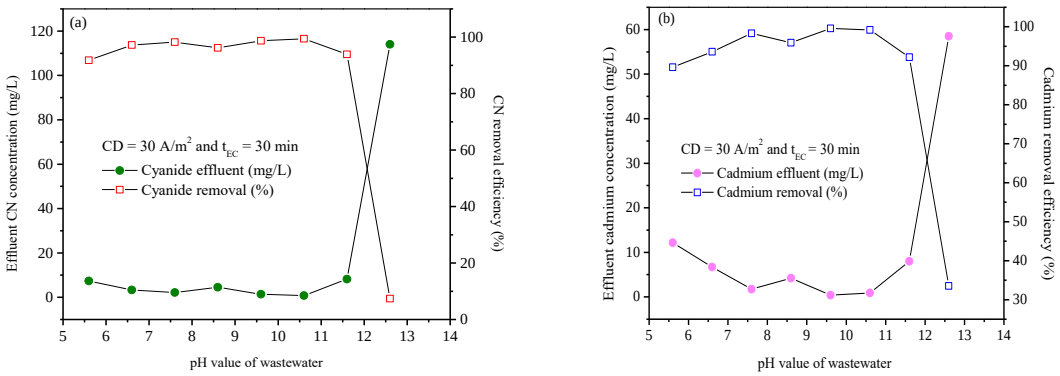

Figure 3. Effect of initial pH on cyanide and cadmium removal from the Cd-CN wastewater.

4.2. Effect of current density and pH on treatment of Cd containing wastewater

The effect of current density is investigated with the following experimental conditions; 0-50 min operating time, for cadmium wastewater. Figure 4 shows the removal efficiencies of cadmium as a function of current density. As it can be seen from the figures and tables given, it is seen that as the current density and operating time increase, the Cd removal efficiency increases. The removal efficiency of cadmium at current density values in the range 5-60 A/m^2 increased from %80 (20.4 mg/L) to %99.9 (0.10 mg/L). According to Faraday's law, since the current density increases, the efficiency of ion production on the anode and cathode increases. Therefore, there is an increase in flocs production in the solution and hence an improvement in the efficiency of cadmium removals. In this study, current density and operating time for cadmium wastewater are selected as current density of 30 A/m^2 and t_{EC} = 40 min, and CD = 60 A/m^2 and t_{EC} = 20 min, respectively since it meets sewage discharge standards for the ISKI Standards for removal of these pollutants [17]. Furthermore, the operating costs increased from 0.96 to 2.40 $/m^3 for cadmium wastewater with increasing current density from 30 to 60 A/m^2, respectively. Higher current density caused high electrical energy consumption which increased operating costs for the treatment. The energy consumption was 5.18 kWh/m^3 and 11.4 kWh/m^3 for current density of CD = 30 A/m^2 and 60 A/m^2, respectively. The electrode consumption was 0.44 kg/m^3 and 0.490 kg/m^3 at the current density of CD = 30 and 60 A/m^2, respectively.

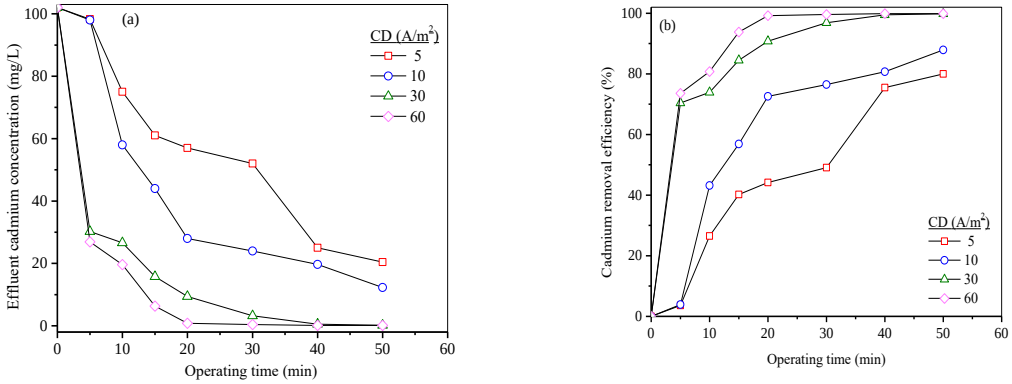

Figure 4. Effect of current density on cadmium removal from cadmium containing wastewater.

In order to study its effect on the treatment, the initial pH of the wastewater was studied in the range of 4.6-12.6 at CD = 30 A/m^2 and 50 min. Removal efficiencies of cadmium was obtained above 90% for studied pHs. Especially higher values of pH 8.6, the Cd concentration decreased from 102 mg/L to 25.6 mg/L at pH 9.6. Removal efficiencies of cadmium at constant operating time of t_{EC} = 50 min and current density of 30 A/m^2 was better than 99% at pH 10.6 and 11.6. Operating the EC process at very high pH values improves Cd removal efficiency. It was also observed and explained for wastewater including Cd-CN. The major iron complexes formed with respect to pH from the Pourbaix diagram are $Fe(OH)^{2+}$ and $Fe(OH)_{2(s)}$ and $Fe(OH)_{3(s)}$ [10]. At large cathodic potentials, the solution can become alkaline. Under these conditions, $Cd(OH)_{2(s)}$ is formed and can be removed from the solution by adsorption on the electrode as charged colloidal particle. Insoluble metal hydroxides as $Cd(OH)_{2(s)}$ was precipitated as increase in solution pH. These results showed that pH was very significant parameter which had an influence on the economic applicability of EC process.

4.3. Effect of current density and pH on treatment of CN containing wastewater

The effect of the current density on the EC process of wastewater containing cyanide was investigated between 5-60 A/m^2 at initial pH = 8.9. As seen in Figure 5, removal efficiency of cyanide increased as the current density and operating time increased. Removal efficiencies were % 84.33 (18.81 mg/L), %90.9 (10.92 mg/L), %99.81 (0.234 mg/L) ve %99.88 (0.15 mg/L) at CD = 5, 10, 30, and 60 A/m^2, respectively. The ISKI criteria (<10 mg/L) required for discharge at current density of CD = 30 and 60 A/m^2 were met in the t_{EC} = 5 minute as 5.75 and 2.57 mg/L at the initial pH 8.9, respectively.

The operating costs were 2.5978 and 8.113 \$/m^3 at 30 and 60 mA/m^2 with the EC process time of 50 minutes, respectively. The energy consumption was 9.4238 kWh/m^3 and 33.3044 kWh/m^3 for current density of 30 A/m^2 and 60 A/m^2, respectively. The electrode consumption was 0.6974 kg/m^3 and 1.1933 kg/m^3 at the current density of 30 and 60 A/m^2, respectively.

The pH is a major other parameter affecting removal of cyanide from wastewater. Figure 6 shows effluent concentration and removal efficenicy of cyanide during operation time at 30 A/m^2. While the initial pH of cyanide wastewater was between 4.6-10.6 and the effluent concentration of CN was 0.145-7.03 mg / L, 99.86% -95.16% CN removal efficiency was achieved in the 50 min operation period, the effluent concentartion was 7.03-95.67 mg/L with 95.16-8.95% CN removal efficiency was obtained at the initial pH range of 10.6-12.6.

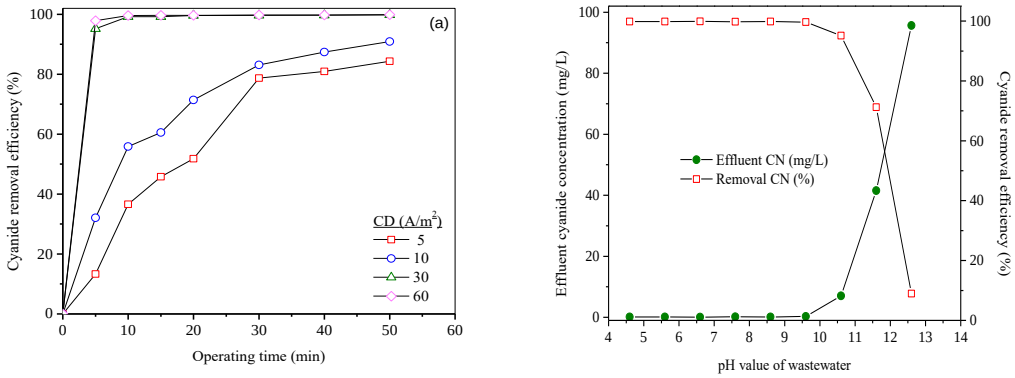

Figure 5. Effect of current density and initial pH on cyanide removal from cyanide containing wastewater.

5. CONCLUSIONS

In this study treatment of wastewater containing Cd-CN, CN and Cd from metal plating rinse baths was carried out by electrocoagulation (EC) technique. Effect of current density, pH, and operating time on EC were investigated for each wastewater. Operating costs also calculated. Removal efficiencies of CN and Cd in EC for wastewater containing Cd-CN were increased with incresing of current density with respect to operating time, and it was determined that higher current densities requires lower operating time. In the EC process, a current density of a limit value is recommended to avoid adverse effects such as heating the wastewater in the EC reactor and to prevent excessive O_2 formation and unfeasible operating cost. Optimum operating conditions for wastewater containing CN-Cd are current density of 30 A/m^2, operating time of 50 min, and removal efficiencies of CN and Cd were 99.70% and 99.59% and a total operating cost of 2.1746 \$/m^3 was calculated. Current density of 30 A/m^2 and operating time of 50 min were determined as the optimum conditions for wastewater containing CN. Removal effciency of wastewater containing Cd was 99.8%. Removal eficiencies for three wastewaters (Cd-CN, CN ve Cd) used in this study were found to be greater than 99% which met wastewater discharge standard set by ISKI. Optimum pH for wastewater containing Cd-CN and CN is 8.60 and for wastewater containing Cd is 10.60. As a result, the electrocoagulation process is a very complex process, and the treatment of Cd-CN containing wastewater with this process is even more complex. Therefore, more detailed studies are required to explain the removal mechanisms in a comprehensive way. It has been shown in this study that wastewater containing Cd-CN, CN and Cd can be treated effectively with the EC process.

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