JEE Journal of Ecological Engineering

Journal of Ecological Engineering, 2025, 26(8), 318–326
https://doi.org/10.12911/22998993/204101
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Received: 2025.03.28
Accepted: 2025.05.26
Published: 2025.06.10

Response surface methodology application in optimizing mercury removal from leachate via electrocoagulation using an iron electrode

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ABSTRACT

Leachate is wastewater containing various pollutants that are harmful not only to the environment, but also to the human beings. The current study aimed to analyze the efficacy of electric voltage, time, and electrode spacing to obtain optimum mercury (Hg) removal using an electrocoagulation method with iron electrodes. Leachate samples were collected from the TPA Terjun (Medan City, Indonesia). The mercury level in the leachate was categorized as exceeding the quality standard. Response surface methodology (RSM) was used to build models, design experiments, evaluate the effects of independent variables, and determine optimal conditions for efficient mercury removal. Leachate cannot be decomposed naturally; therefore, certain processing steps are needed to prevent leachate from polluting the environment. Electrocoagulation is an alternative to leachate treatment that can remove heavy metals, including Hg. In this study, the electrocoagulation process was used with iron plate of various voltages (8, 10, 12 V), the distance between electrodes amounted to 1, 2, and 3 cm, and contact time 10, 20, and 30 minutes. To maximize the efficiency of mercury (Hg) removal, data analysis was conducted using RSM. The results showed removal efficiency of 98.86% with recommended voltage, time, and electrode spacing of 12 V, 29.52 minutes, and 1.11 cm, respectively.

Keywords: leachate, electrocoagulation, mercury, iron electrodes, RSM.

INTRODUCTION

Particularly in developing countries, the rapid expansion of a number of enterprises, including mining, metal plating plants, tanneries, batteries, paper, and pesticide manufacturing, has increased the direct or indirect discharge of heavy metal wastewaters into the environment. In contrast to organic contaminants or pollutants, heavy metals do not biodegrade and have a propensity to accumulate in biological systems. It is also recognized that many heavy metal ions are carcinogenic or dangerous (Tchounwou et al., 2012; Xu et al., 2017). Mercury and other toxic heavy metals are especially problematic when treating industrial effluent. Mercury is one of the neurotoxins that can harm the central nervous system. Increased amounts of mercury can lead to dyspnea, chest discomfort, and impairment of kidney and pulmonary function (Rafati-Rahimzadeh et al., 2014; Andreoli and Sprovieri, 2017).

The final processing site – Tempat Pemrosesan Akhir (TPA) serves as the terminal phase for integrated waste management systems. Waste is received by TPA from surrounding municipalities, typically comprising mixed residential, commercial, and institutional materials. Among multiple byproducts generated, leachate (Taqwa and Syakdani, 2017) emerges as a critical concern, a contaminated liquid originating from precipitation or groundwater infiltration that permeates waste deposits, dissolving numerous chemical compounds. This results in exceptionally high pollutant concentrations, including biochemical oxygen demand (BOD), ammonia, and heavy metals, with documented environmental risks (Kasmuri and Tarmizi, 2018). Leachate affects water quality by causing ground and surface water pollution. The environmental consequences are multifaceted: leachate infiltration compromises both groundwater reserves and surface water systems. Compositional factors vary significantly based on site-specific characteristics, precipitation volumes, and concentrations of organic/inorganic materials. Notably, Hg persists as a hazardous component within TPA leachate, a toxic element posing severe risks through inhalation, ingestion, or dermal exposure. Such risks necessitate rigorous operational protocols. To ensure continuity, waste management strategies must prioritize containment efficacy while addressing these complex interdependencies.

Many different methods, including ion exchange (Lai et al., 2016), adsorption (Verma et al., 2008), as well as biological (Javid et al., 2019), electrochemical (Assadi et al., 2016), and membrane technology (Li et al., 2017), have been utilized to remove heavy metals from aqueous media. Due to its effectiveness, the treatment of landfill leachate using unconventional methods, such as electrochemical techniques, has gained importance in recent years. Electrocoagulation (EC), an electrochemical process, has been applied extensively to remove heavy metals from industrial and municipal wastewaters (Tegladza et al., 2021; Islam, 2019; Bazrafshan et al., 2015; Shaker et al., 2023; Al-Qodah and Al-Shannag, 2017; Ebba et al., 2022). However, there has not been much research on treating solid waste landfill leachate with the intention of removing heavy metals, particularly mercury (Hg). By running an electric current through water, the process of electrocoagulation destabilizes contaminated suspensions, emulsions, and solutions, resulting in the creation of easily separable lumps. With electrocoagulation, the mercury in the leachate can be removed by forming flocs that will undergo flotation so that they will separate from the leachate.

Electrocoagulation is a method that uses electrical energy and converts it into a chemical reaction using two electrodes, namely, positive and negative electrodes (Naje et al., 2017). The electrode will form a coagulant that is used to separate the contaminants in the waste (Kobya et al., 2003). The electrocoagulation process broadly occurs in 3 stages, namely the electrolytic reaction on the surface of the electrode, the formation of coagulant in the solution phase, and the adsorption of the solution or the formation of pollutants into colloids on the coagulant as well as removal by precipitation or flotation (Kobya et al., 2003). In the electroelution mechanism, a coagulation or agglomeration process occurs with the help of electricity via the process of electrolysis to remove particles and metal ions in water. The electrocoagulation mechanism occurs when the anode metal plate is oxidized to produce metal ions and release electrons (Masrullita et al., 2021; Jovanovic et al., 2021). Metal ions can act as coagulants and will undergo hydrolysis to produce charged metal hydroxide ($M^{n+}(OH)_n$) ions that act as flocculants and bind the particles to water. Thus, organic pollutants can experience coagulation and flocculation. At the cathode, H_2 gas and oxygen gas are formed, which can push the floccules to float above the surface of the liquid. The reactions that occur at the electrodes can be described as follows. The reaction at the cathode is expressed by following Equations (1, 2):

$$2H^+ + 2e \rightarrow H_2 \tag{1}$$

$$2H_2O + 2e \rightarrow 2OH^- + H_2 \tag{2}$$

The reaction at the anode is expressed by following Equations (3–5):

$$Fe^{3+} + 3H_2O \rightarrow Fe(OH)_3 + 3H + 3e \tag{3}$$

$$4OH \rightarrow 2H_2O + H_2 + 4e \tag{4}$$

$$2H_2O \rightarrow 4H^+ + O_2 + 4e \tag{5}$$

The material of electrodes, distance from one another, wastewater temperature and composition, voltage, as well as current density all have an impact on the electrocoagulation process. The efficiency of electrocoagulation declines as the concentration of suspended particles rises above 100 mg/l. The energy required for the anodic dissolution of metal decreases as the distance between the electrodes decreases. The compactness and controllability of the units, the lack of chemicals, the low sensitivity to changes in the cleaning process conditions, and the production of sludge with good mechanical and structural qualities are all benefits of the electrocoagulation method.

This study explored potential methods for removing heavy metals, specifically mercury, from leachate using the electrocoagulation process. It examined the effects of various operational factors, including electrode materials, current density, interelectrode distance, and operating time.

MATERIALS AND METHODS

The leachate samples were taken from TPA Terjun in Medan City. The electrocoagulation experiment was carried out in a glass reactor with a volume of 1000 ml. The electrodes (anode and cathode) used were iron with a size of 4 × 10 cm and a thickness of 1 mm. In this study, a batch system was used with a series of tools consisting of 1-liter beakers with an iron electrode connected to a power supply. The series of tools can be seen in Figure 1.

This research aimed to evaluate the impact of varying electric voltages (8, 10, and 12 V) and electrode distances (1, 2, and 3 cm) on the efficiency of Hg removal from TPA leachate using the electrocoagulation method. The used in this study contact time was from 10 to 30 minutes.

Research process

The research process that was carried out consisted of testing the initial leachate sample and the running process. The testing of the initial leachate sample aimed to determine the initial levels of mercury in the leachate. The stages in the running process were as follows – 1000 mL of the leachate sample was placed into the reactor. The Fe electrodes were inserted with distances of 1, 2, and 3 cm between them. The power supply was turned on at 8 V to operate the electrocoagulation process, which was timed with a stopwatch. After 10 minutes, 50 mL of the solution was removed using a measuring pipette. After all processes were complete, the power supply was turned off, and another experiment was performed at each variation of the voltage and electrode distance. Mercury content testing was then carried out on the sample.

Sample testing

For sample analysis, mercury levels were measured using inductively coupled plasma (ICP) following the APHA 3120B standard method (Table 1).

Data analysis

In this study, data analysis was performed using the Design Expert software with the Box-Behnken methodology to determine the impact of key variables (voltage, contact duration, and electrode distance) on the effectiveness of mercury removal. The data obtained were analyzed with multiple linear regression tests. The mercury removal efficiency was calculated by

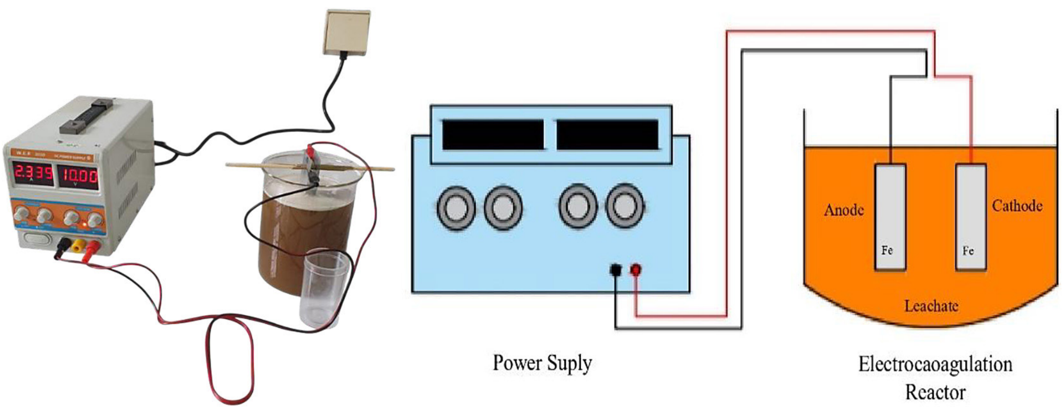

Figure 1. Series of electrocoagulation equipment

Table 1. Number of sample tests

Run	Voltage (V)	Time (min)	Electrode distance (cm)
1	10	10	1
2	10	10	3
3	8	20	1
4	12	20	1
5	10	20	2
6	8	20	3
7	10	20	2
8	8	10	2
9	10	30	1
10	12	10	2
11	10	30	3
12	12	20	3
13	10	20	2
14	8	30	2
15	12	30	2

comparing the influent and effluent concentrations expressed in percent with the Equation (6):

$$Percentage\ (\%) = \frac{C_0 - C_1}{C_0} \times 100\% \tag{6}$$

where: C_0 – level before processing, C_1 – levels after electrocoagulation treatment

RESULTS AND DISCUSSION

The experimental results based on the Box-Behnken design are presented in Table 2. The highest mercury removal efficiency, 98.39%, was achieved after 15 runs with a variation of 12 V, contact time of 30 min, and electrode distance of 2 cm. Conversely, the lowest removal efficiency, 95.02%, was recorded in the eighth run using an 8-volt variation, 10-minute contact time, and a 2-centimeter distance. In all experiments, the final mercury levels remained below the established quality standard.

Response model selection analysis

The goal of the statistical model analysis in this study was to select an appropriate mathematical model based on the experimental data and evaluate it using statistical methods to determine the impact of the independent variables on mercury removal efficiency. In response surface methodology (RSM), several criteria are used for model selection, including the Sequential Model Sum of Squares (which assesses the order of the model), Lack of Fit tests (which evaluate model accuracy), and Model Summary Statistics (which provide an overall assessment of model performance). Table 3 presents the findings of the Sequential Model Sum of Squares analysis for Hg removal efficiency. Tables 4 and 5 display the results of model selection based on summary statistics and lack of fit analysis, respectively.

On the basis of the established model selection methods, the linear model was chosen to describe the effect of voltage, contact time, and electrode distance on mercury removal efficiency. The influence of each independent variable on the response was evaluated using analysis of variance (ANOVA). The ANOVA results are presented in Table 6.

According to Table 6, the p-value model < 0.0001 is smaller than the alpha or significance value of 0.05. The model deviation value (lack of fit) for mercury removal efficiency shows a p-value of 0.8254, which means p > 0.05 states "not significant". This shows that the model fits the plot of the linear model.

Impact of voltage variation on the efficacy of mercury removal

Figure 2 illustrates that the efficacy of mercury removal increased when the voltage was applied. Continuous voltage application will cause the number of Fe^{2+} from the produced electrodes to rise, which will raise the number of $Fe(OH)_2$ flocs as well. This implies that throughout the electrocoagulation process, more flocs will develop and adhere to the electrodes the higher the voltage applied. Furthermore, the greater the strength of the voltage used, the greater the percentage of allowance produced (Afifa et al., 2021). The deposition of $Fe(OH)_3$ flocs in the electrocoagulation bath follows the coagulation-flocculation principle. As the floc mass increases, its specific gravity also rises, eventually leading to sedimentation. The strength of the current and voltage used during the electrocoagulation process has had an immediate impact on this. The greater the strength of the current and voltage applied, the more floc is produced which can bind the contaminants in the leachate. In addition, the distance between the electrode plates greatly affects the process of decreasing the concentration of Hg.

Table 2. Efficiency of removal of mercury levels

Run	Voltage (V)	Time (min)	Electrode distance (cm)	Removal efficiency (%)
1	10	10	1	96.259
2	10	10	3	96.117
3	8	20	1	95.643
4	12	20	1	98.248
5	10	20	2	96.638
6	8	20	3	95.299
7	10	20	2	97.420
8	8	10	2	95.020
9	10	30	1	97.687
10	12	10	2	98.058
11	10	30	3	97.682
12	12	20	3	98.153
13	10	20	2	97.443
14	8	30	2	95.643
15	12	30	2	98.390

Table 3. Sequential model sum of squares

Source	Mean square	Sum of squares	df	F-value	p-value	
Total vs Mean	1453.65	1453.65	1			
Mean vs Linear	0.0153	0.0460	3	50.06	< 0.0001	Suggested
Linear vs 2FI	0.0000	0.0001	3	0.0906	0.9632	
2FI vs Quadratic	0.0003	0.0008	3	0.5217	0.6859	
Quadratic vs Cubic	0.0005	0.0014	3	0.8610	0.5769	Aliased
Residual	0.0005	0.0011	2			
Total	96.91	1453.70	15			

Table 4. Lack of fit tests

Source	Sum of squares	df	Mean Square	F-value	p-value	
Linear	0.0023	9	0.0003	0.4691	0.8254	Suggested
2FI	0.0022	6	0.0004	0.6695	0.7024	
Quadratic	0.0014	3	0.0005	0.8610	0.5769	
Cubic	0.0000	0				Aliased
Pure Error	0.0011	2	0.0005			

The magnitude of the voltage is directly proportional to the strength of the current. The greater the electric current given, the greater the anode is oxidized; therefore, there is a reduction in the anode mass (Hasyyati et al., 2020). A higher voltage leads to an increased production of Fe^{3+} ions, forming more coagulants that aid in pollutant removal. Additionally, as voltage increases, bubble production intensifies while bubble size decreases, enhancing removal efficiency through the flotation process (Wang et al., 2015). A greater applied voltage also generates a stronger electric current (Hur and Kim, 2000). According to Khorram and Fallah (2018), electric current flowing through the electrolyte solution and electrodes induces chemical changes. Since electric current represents a continuous flow of electrons in a conductor, an increase in current accelerates anode dissolution. This, in turn, produces larger quantities of metal hydroxide flocs, ultimately improving the mercury removal efficiency (Qasem et al., 2021).

Table 5. Summary statistics model

Source	Standard deviation	R^2	Adjusted R^2	Predicted R^2	PRESS	
2FI	0.0202	0.9340	0.8845	0.7832	0.0107	
Linear	0.0175	0.9318	0.9131	0.8859	0.0056	Suggested
Cubic	0.0233	0.9781	0.8464		*	Aliased
Quadratic	0.0223	0.9497	0.8592	0.4973	0.0248	

Table 6. Results of analysis of variety (ANOVA)

Source	Sum of squares	df	Mean Square	F-value	p-value	
Model	0.0460	3	0.0153	50.06	< 0.0001	significant
A-Tegangan	0.0408	1	0.0408	133.37	< 0.0001	
B-Waktu	0.0050	1	0.0050	16.44	0.0019	
C-Jarak	0.0001	1	0.0001	0.3649	0.5581	
Residual	0.0034	11	0.0003			
Lack of Fit	0.0023	9	0.0003	0.4691	0.8254	not significant
Pure Error	0.0011	2	0.0005			
Cor Total	0.0493	14				

Effect of time variation on mercury removal efficiency

Figure 3 illustrates that the efficiency of mercury removal increases with the length of electrocoagulation time. According to Faraday's law, which indicates that more coagulant will produce the longer the processing time, the longer the electrocoagulation processing time, the better the reduction in pollution parameters. The reduction in pollutant parameters improves with the number of coagulants that develop (Purwati and Erwin, 2018). More flocs are generated and better outcomes are obtained with extended contact times (Yudhistira et al., 2018). An increase in electrocoagulation time enhances removal efficiency, as prolonged operation allows more ions to be generated by the electrodes and promotes the gradual formation of small flocs that grow larger and eventually settle at the bottom of the reactor (Qasem

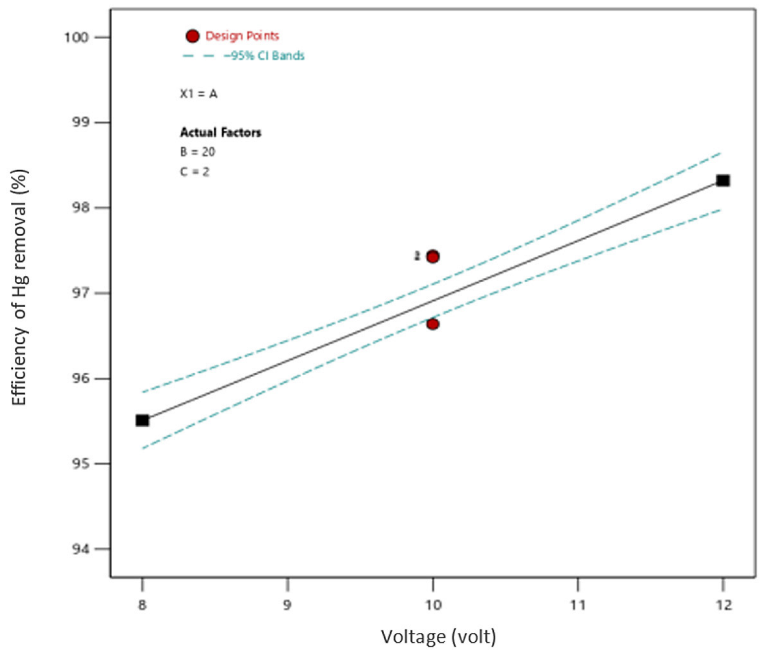

Figure 2. An investigation concerning how voltage affects the effectiveness of mercury removal

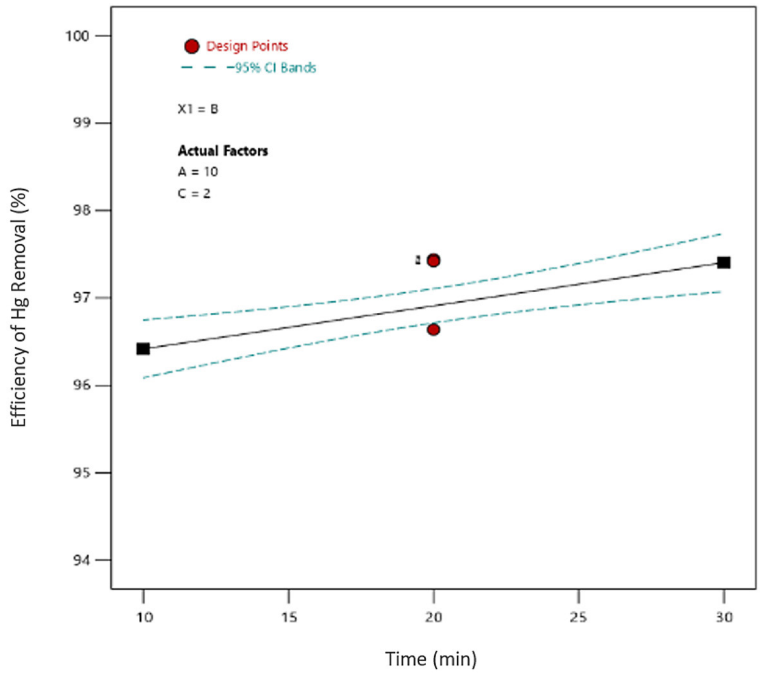

Figure 3. The effect of time on mercury removal efficiency

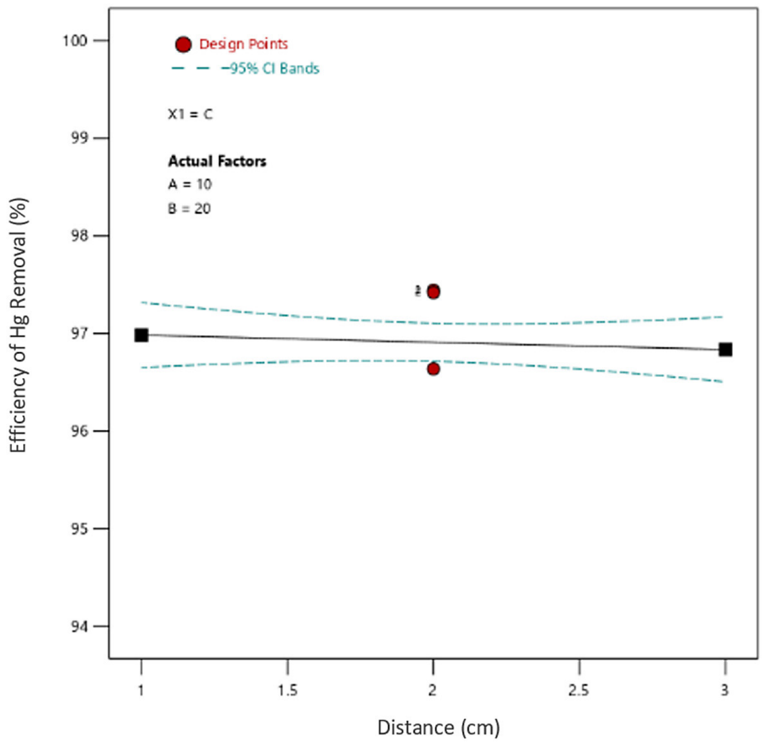

Figure 4. The effect of electrode distance on mercury removal efficiency

et al., 2021). Additionally, longer electrolysis times contribute to a greater reduction in mercury levels, as extended exposure leads to increased mercury removal. However, prolonged electrocoagulation also results in a greater reduction in electrode mass due to the extended oxidation process at the anode, which accelerates anode dissolution.

Effect of distance variation on mercury removal efficiency

As shown in Figure 4, the mercury removal efficiency improved as the electrode distance increased. The electrolyte resistance is influenced by the distance between the electrodes;

the more spaced apart the electrodes are, the higher the resistance, and hence the lower the current flow (Purwati and Erwin, 2018). A small current causes a short current, so the reaction that occurs is not optimal because the amount of Fe^{2+} becomes small, so the pollutant that is precipitated is also small. Removal of Hg occurs when an increasing number of Fe^{3+} ions are produced at the anode and form $Fe(OH)_3$ flocs which act as coagulants. Then, metals and organic molecules in water can be bound by the $Fe(OH)_3$ floc. The mechanism by which iron is removed during the electrocoagulation process is as follows Equations (7–9):

$$\text{Anode: } Fe \rightarrow Fe^{3+} + 3e \quad (7)$$

$$\text{Cathode: } 2H_2O + 2e \rightarrow 2OH^- + H_2 \quad (8)$$

$$\text{Overall: } Fe^{3+} + 3H_2O \rightarrow Fe(OH)_3 + 3H + 3e \quad (9)$$

Variations in distance and resistance also affect the effectiveness of this electrocoagulation. A large distance will create a large obstacle resulting in decreased electron transfer rates and the oxidation process of Fe^{2+} ions is not optimal (Fadhila and Purnama, 2022).

CONCLUSIONS

The effectiveness of mercury removal is significantly impacted by voltage. The effectiveness of mercury removal increases along with voltage. This is caused by the more coagulant formed to remove mercury and the production of larger bubbles will increase the efficiency of removal through flotation. The effectiveness of mercury removal is significantly impacted by time. The efficiency of mercury removal increases with the length of the electrocoagulation process. This is because an increasing amount of coagulant is produced by the electrodes so that small flocs are formed, which will enlarge and settle to the bottom over time. The effectiveness of mercury removal is significantly impacted by distance. The efficiency of mercury removal decreases with increasing electrode spacing. This is due to the decrease in electrostatic power, which results in a decrease in the formation of flocs to remove mercury. In addition, the current will be smaller so that less coagulant will be formed.

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