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CHEMICAL OXYGEN DEMAND AND TURBIDITY REMOVAL OF LANDFILL LEACHATE USING ELECTROCOAGULATION TECHNIQUE WITH Al ELECTRODE

(Penyingkiran Permintaan Oksigen Kimia dan Kekeruhan daripada Larut Resap Tapak Pelupusan dengan Menggunakan Elektrod Al)

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Abstract

The electrocoagulation process is classified as green technology to treat landfill leachates. A 16 full factorial experimental design evaluated and optimized the electrocoagulation method and compromised efficiency and operational costs. This study assessed three factors: operation time, voltage, and electrode size, to determine the most influencing parameters and describe the interaction between chemical oxygen demand (COD) and turbidity removal. Statistical analysis results using a half-normal plot demonstrated that all main factors significantly affected the removal efficiency of COD and turbidity. The Pareto chart reveals that the order of significance for COD and turbidity removal efficiency was voltage > operation time > electrode size> interaction between voltage and electrode. The best regression coefficients (R^2) were obtained for COD and turbidity, which reached 0.9597 and 0.9908, respectively, confirming that the predicted values complied with the experimental values. This implied the appropriateness of the employed regression model. The optimization process results showed that for maximizing the removal efficiency of COD and turbidity, the optimal level of operation time was 30 min, voltage 30 V and electrode 10 cm2 by using a batch reactor.

Keywords: electrocoagulation, landfill leachate, chemical oxygen demand, turbidity

Abstrak

Proses elektrokoagulasi diklasifikasikan sebagai teknologi hijau untuk merawat tapak pelupusan larut lesap. Reka bentuk eksperimen 16 faktorial penuh telah digunakan untuk menilai dan mengoptimumkan kaedah elektrokoagulasi dan untuk mencapai kompromi antara kecekapan dan kos operasi. Kajian ini menilai tiga faktor: masa operasi, voltan dan saiz elektrod untuk menentukan parameter yang paling mempengaruhi dan menerangkan interaksi antara parameter untuk permintaan oksigen kimia (COD) dan penyingkiran kekeruhan. Keputusan analisis statistik menggunakan plot separa normal menunjukkan bahawa semua faktor utama mempunyai kesan yang ketara ke atas kecekapan penyingkiran COD dan kekeruhan. Carta Pareto mendedahkan, untuk COD dan kecekapan penyingkiran kekeruhan, urutan kepentingan ialah voltan > masa operasi > saiz elektrod > interaksi antara voltan dan saiz elektrod.. Pekali regresi terbaik (R^2) diperoleh untuk COD dan kekeruhan mencapai nilai masing-masing

0.9597 dan 0.9908 mengesahkan bahawa nilai yang diramalkan adalah mematuhi nilai eksperimen yang membayangkan kesesuaian model regresi yang digunakan. Hasil proses pengoptimuman menunjukkan bahawa untuk memaksimumkan kecekapan penyingkiran COD dan kekeruhan, tahap masa operasi yang optimum adalah 30 minit, voltan 30 V dan elektrod 10 cm^2 dengan menggunakan reaktor batch.

Kata kunci: elektrokoagulasi, larut lesap tapak pelupusan, permintaan oksigen kimia, kekeruhan

Introduction

Malaysia produces about 42,672 metric tons of solid waste every day, and the value may be increased every year due to urbanization, increase in population, and per capita waste generation [1]. Generally, organic wastes constitute 40 – 60 % of the overall weight in most developing countries. Despite several advantages of landfilling, the resulting heavily polluted leachate has caused an urgent concern because landfilling is a widely used method for solid waste disposal. Various factors influence leachate quality and quantity, such as landfilling technique, seasonal weather variation, waste type and composition, and landfill structure [2]. Therefore, environmental specialists are determined to develop effective treatments for vast quantities of heavily polluted leachates.

The EC process is a process to form floc with the help of oxidized metal in wastewater, whereby it needs to be cleaned by the electro-dissolution anode [3]. EG can also be defined as a process of removing the suspended solids from wastewater by using electricity to neutralize the harmful particles through hydroxide complexes formation in water to gather the suspended solid, help bridge, bind, and strengthen floc sedimentation due to gravity force. This process collects the suspended solids in water without coagulants, and then coagulation occurs when a direct current is applied, thus capable of removing small particles and setting them into motion. Several factors that influence the EC process's efficiency are electrode types, the gap between electrodes, electrode size, the configuration of metals, current density, charge loading, pH value of the sample, the addition of supporting electrolyte, and operation time. The electrodes that are usually used are iron, aluminum, and stainless steel. This metal sheet pair is anode and cathode [4].

Nowadays, most treatment methods widely used worldwide to treat wastewater are physical-chemical and biological processes, but the treatment of leachates by using these methods is complex due to low biodegradability and high toxicity. The biological process is a method that uses natural methods that depend on microorganisms, nematodes, and tiny creatures. At the same time, physical-chemical processes require added synthetic substances [5]. For example, Castillo-Suárez [6] used peroxicoaguation and solar peroxicoagulation in a batch electrolytic reactor in the presence of solar ultraviolet light and studied three main parameters to treat the leachates, which are pH value, current density, and treatment time. The use of these three parameters gives the optimal value of biochemical oxygen and COD removed at 55.5% and 62.3%, respectively. Jang et. al [7] studied the effect of adding lithium manganese oxide and activated carbon electrodes in the electrochemical sorption of the lithium-ion battery leachate. The authors found that increased capacity, selectivity, and purity depended on the reaction time and applied current. The same parameters; time, and current density, were also studied by Galvão [8] in the EC process treatment. Five responses were recorded: ultraviolet absorbance, biological oxygen demand, COD, turbidity, and color, while the percentage values of removal when applied with 128 Am^{-2} for 90 min were 40%, 90%, 40%, 82%, and 82%, respectively. The removal efficiency was increased to a maximum by increasing the electrolysis time, leading to a constant rate. The number of generated metal hydroxides increased with the electrolysis time.

In this regard, a group of researchers used the EC process to remove cefazolin and COD from hospital wastewater [9]. The tests were performed in three specific voltages (15V, 30 V, 50 V) and periods (10 min, 30 min, 50 min). The result showed that the maximum efficiency of contamination removal in 50 min was more than 92%. Chairunnisak et al. [10] reported that the electrocoagulation method resulted in the optimum COD reduction of 94.53% from operating time of 39.28 min, 20 V, and without electrolyte concentration. A study conducted by Amarine et al. [11] showed that the removal efficiency depended on the electrodes' applied voltage and immersed surface. This removal efficiency was 94.41% after 150 min, the electrical voltage of 30 V, and the immersed surface of 33.75 cm^2. Another research by Bajpai et al. [12] found that an optimum condition (14 V and 47 min) at a pH value of 7.35, which provided experimental removal efficiency (75.6% COD, 78.7% total dissolved solids, 93.4% turbidity, and 63.2% chloride).

There is in situ generation of coagulants by the dissolution of sacrificial anodes due to direct current flow. Different studies have proven that the aluminum electrode was the most effective and successful. Therefore, aluminum was used as the electrode material in this study. The anode produces metal cations after its oxidation process by applying direct current; this cation involves further reactions and produces different polymeric hydroxides of metal, similar to salts used in conventional coagulation.

The reactions when using aluminum electrodes can be summarized as follows;

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Anode reactions:

$$Al(s) \rightarrow Al^{3+}_{(aq)} + 3e \quad (1)$$

$$Al^{3+}_{(aq)} + 3OH^- \rightarrow Al(OH)_3 \quad (2)$$

Alkaline reactions:

$$Al^{3+}_{aq} + 3H_2O \rightarrow Al(OH)_3 + 3H^+ \quad (3)$$

Acidic pH range

Cathode reactions leads to mainly hydrogen evolution:

$$3H_2O + 3e \leftrightarrow {}^{3}/_{2}\, H_2 \uparrow + OH^- \quad (4)$$

As seen in the above reactions, EC combines oxidation, flocculation and flotation. The EC occurs in three steps. In the first step, a coagulant is formed due to oxidation of the anode. In the second step, pollutants get destabilized, while in the last step, the destabilized matters get united and removed.

EC is an exceptional strategy for water and wastewater treatment. While the EC has been applied to different modern gushing, for example, nourishment businesses, tannery, mechanical shops (dissolvable oil), polymerization production, and wastewater material industry. Naje & Abbas [13] limited studies were carried out on the effective voltage, operation time and electrode side in the electrocoagulation process time in the COD and turbidity removal. Simple, affordable, and efficient leachate treatment systems are urgently needed in developing countries because most conventional technologies in industrialized nations are too expensive and complex. Rusdianasari et al. [14]. EC is a simple method to treat wastewater efficiently, and it seems to be a promising treatment method due to its high effectiveness, lower maintenance cost, less need for labor, and rapid achievement of results. In this study, statistical analysis via factorial design was used to investigate factors that influence the reduction of two primary contaminants in leachate, which were COD and turbidity.

Materials and Methods

Sample collection

A close landfill that was shut down for over five years was selected to collect the leachate samples. Even though the landfill was shut down, the leachate was still being produced in the landfill. The amount of leachate produced was 200 m^3 per day, while the volume of leachate discharged was 190 m^3. The grab sampling technique was used for collecting the samples. Samples were kept at a temperature of 4 °C in an icebox to preserve the original circumstances of the sample. The main characteristics of the studied leachate samples are shown in Table 1.

Table 1. Characteristics of the leachate sample used in this study

Parameters	Units	Value
Chemical Oxygen Demand (COD)	mg/L	675
Turbidity	NTU	705

Experimental set-up

The batch experimental was set up as the electrochemical unit, which consisted of an electrocoagulation cell, a DC power supply, and aluminum electrodes. In this study, EC cell was made from aluminum anodes, and cathodes with 2 cm spacing were placed vertically on the cell floor. The electrodes had dimensions of 5.65 cm × 13.9 cm, with a functional area of 28.76 cm^2 (anode). The stirrer was set at 100 rpm to maintain the composition and avoid flocs in the solution. The electrodes were soaked in 0.4 M of HCl (hydrochloric acid) for 10 min and then rinsed with deionized or distilled water to remove any organic proteins from the surface of the electrodes. The procedure started with an electrocoagulation cell cleaned with distilled water and dried using a dryer. The experiments were carried out in batches. In each experiment, a 500 ml leachate sample was filled into the electrochemical cell with the Al electrodes dipped into the sample at room temperature. The electrodes (anode and cathode) were clamped at the electrode stand. The circuit was completed by connecting wires to the electrodes (anode and cathode) and the DC power supply.

Analysis

The removal efficiency of COD and turbidity was determined according to the following formulas:

$$\text{COD (\%)} = \frac{\text{CODo} - \text{CODt}}{\text{CODo}} \times 100 \tag{5}$$

where COD_O and CODt are the chemical oxygen demand (COD) at time = 0 (initial) and at t (reaction time, t), respectively.

$$\text{Turbidity (\%)} = \frac{\text{Co} - \text{Ct}}{\text{Co}} \times 100 \tag{6}$$

where C_0 and C_t are turbidity registered (in NTU) at time t=0 (initial) and at t (reaction time), respectively.

Full factorial design of experiments

The three independent factors used in the full factorial design were the voltage, operation time, and electrode size. In contrast, the responses used were the removal percentage of COD and turbidity to achieve the optimum conditions in the experimental design method approach. The input factor variables with their range and levels are shown in Table 2. In this work, 16 experiments were run wholly, including the combination of all levels of other factors, and performed randomly to reduce experimental errors. As the output of factorial screening tests, the critical interactions between all variables were given. The model's validity is verified by analyzing values in the analysis of variance (ANOVA) table. The interactive statistical data analysis tool software in the analysis design was used to construct the model for factorial screening design.

Table 2. Factors and levels used in this experimental design

Parameter	**Low Level (-)**	**High Level (+)**
Operation Time (min)	10	30
Voltage (v)	10	30
Size of electrode (m^2)	10	30

Results and Discussion

The effect of the voltages

The initial concentration is one of the remarkable factors in the electrocoagulation process. The influence of this factor on the COD and turbidity removal efficiency is depicted in Figures 1(a) and 1(b). As demonstrated in both figures, the COD and turbidity removal efficiency increased by increasing the voltages for the electrocoagulation process from 5V to 30V. However, more voltage increments caused a descending trend in the removal efficiency. The descending trend can be attributed to the fact that there is a threshold limit for the elimination. The decaying of COD and turbidity removal efficiencies due to the increase in electrical power reduced the lifespan of the electrodes.

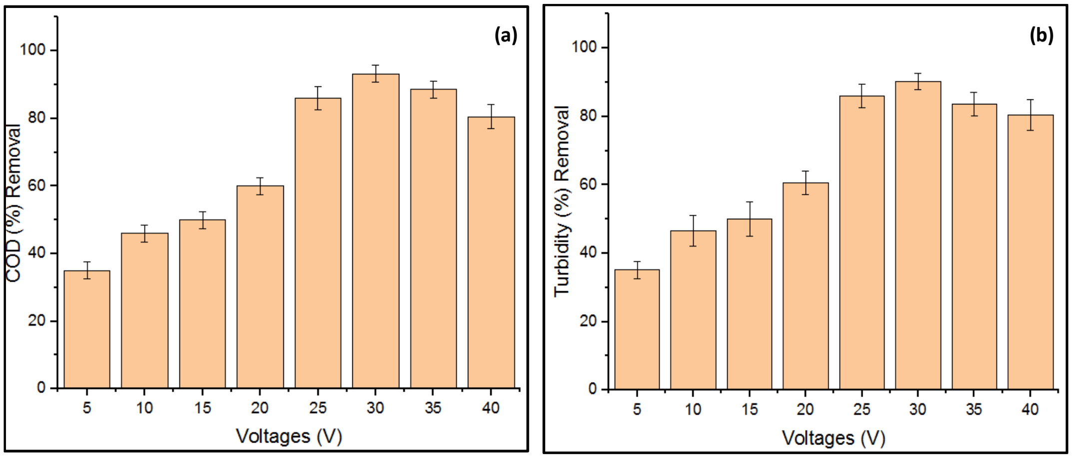

Figure 1. Removal percentages for (a) COD and (b) turbidity removal after 30 min of the mode of operation at different voltages

The effect of the operation time

Figure 2(a) and Figure 2(b) depict the effect of operating time on COD and turbidity removal efficiency. By changing the operating time from 5 min to 30 min, the removal percentages of COD and turbidity increased in the same trend of increment. In this process, EC involved two stages: destabilization and aggregation. The first stage is usually short, whereas the second stage is relatively long. Metal ions, as destabilization agents, are produced at the anode through electrochemical reactions. For 30 min, they indicated an increase in electrolysis duration and increased destabilization of colloidal particles. Both graphs demonstrated that the maximum efficiency of the EC process is obtained at a treatment time of 30 min, and a further increase in treatment time to 40 min did not result in any significant improvement in the removal efficiency of the studied parameters.

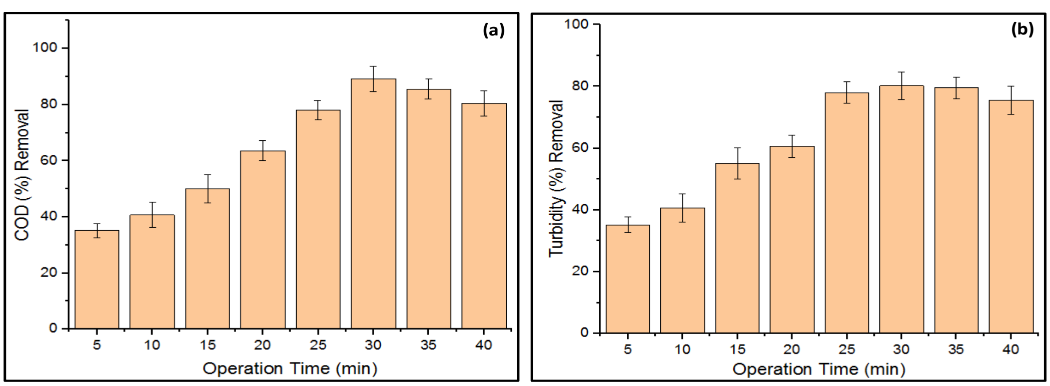

Figure 2. Removal percentages for (a) COD and (b) turbidity removal for 30 voltages for different operation time

The effect of the size of electrodes

Figures 3(a) and 3(b) shows that larger electrodes contribute to high COD and turbidity removal efficiencies. A larger surface area electrode has more area for this oxidation process. However, larger electrodes will incur higher costs and worsen portability. This study aims to preliminarily ascertain the effect of the electrode size on COD and turbidity removal and provide optimal electrode size for electrocoagulation

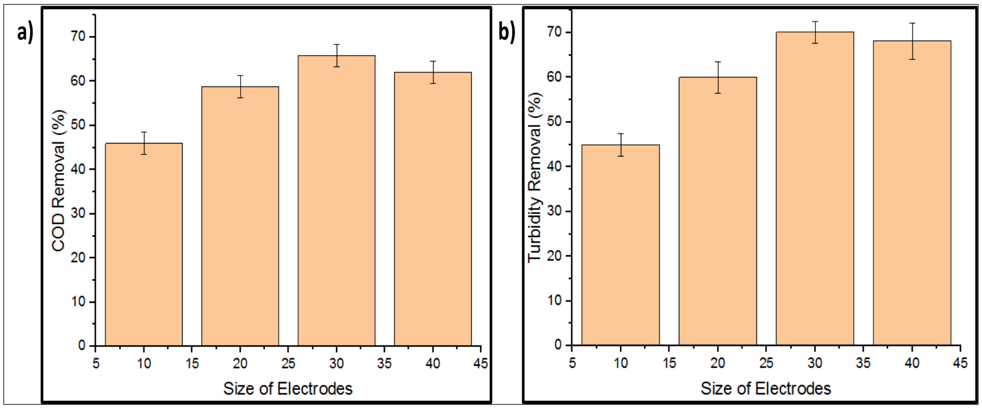

Figure 3. Removal percentages for (a) COD and (b) turbidity removal at the different sizes of electrodes for 30 min operation time and 40 volt

Statistical validity of the model

A matrix of full-factorial design from the two levels (24) is shown in Table 3. Each combination is experimentally carried out in a randomized sequence to meet the statistical requirement for independence of observations. As a result, two responses are examined in the leachate treatment: the COD and turbidity removal.

Table 3. The 3-factor full factorial design matrix and the response function

Exp	Factors			Removal Efficiencies	
	Operation Time	Voltage	Electrode Size	COD (mg/l)	Turbidity (NTU)
1	10	30	10	96	100
2	10	10	10	200	209
3	10	30	10	98	97
4	30	30	30	65	60
5	30	10	30	92	95
6	30	10	30	97	100
7	10	10	30	140	141
8	30	10	10	156	151
9	10	30	30	49	51
10	30	10	10	150	152
11	10	30	30	87	90
12	10	10	10	170	190
13	30	30	10	56	60
14	30	30	10	40	42
15	30	30	30	70	75
16	10	10	30	138	140

Analysis of variance (ANOVA)

Effect of variables on COD removal

The ANOVA for the correlative model development step is presented in Table 4 to describe the removal efficiency of COD as a function of the studied variables. It demonstrated that all the above factors are significant, and the (B) voltage is a highly remarkable variable. The ANOVA shows that the p-value for the model is less than 0.0001, and an F value of 27.22 is obtained for the model, demonstrating the significance of the developed correlation. Besides the main factors A, B, and C, which refer to the operation time, voltage, and electrode size, the model also suggested the interactions of AB, AC, BC, and ABC. Those interactions in the model are less than 0.05, except for the interactions of AB and AC. Nevertheless, these higher values of AB and AC did not affect the total p-value of the model.

Furthermore, the ANOVA table displays supplementary pieces of beneficial information. For instance, the quantity "R-squared" is defined as [15];

$$R^2 = \frac{\text{model sum of squares}}{\text{total sum of squares}} \tag{7}$$

The coefficient of determination (R^2), which shows proportional variation in the response explained by the independent variables in the linear regression model, is 0.9597, which means that the model could describe 95.97% of the variations in the response. The adjusted coefficient of determination (R^2adj) is 0.9244. The value of R^2, which is close to 1, revealed a significant linear relationship between the factors and response. There is a slight difference between R^2 and R^2adj values, which shows that some insignificant conditions (error) are included in the model. The final empirical models in terms of actual parameters are determined as follow

$$COD = +106.50 - 15.75{*}A - 36.38{*}B - 14.25{*}C + 3.38{*}AB + 4.50{*}AC + 11.88{*}BC + 7.62{*}ABC \tag{8}$$

Table 4. ANOVA test for the removal of COD

Source	**Sum of Squares**	**df**	**Mean Square**	***F*-Value**	***P*-Value**	
Model	32081	7	4583	27.21901	<0.0001	significant
A-Operation time	3969	1	3969	23.57238	0.001	
B-Voltage	21170.25	1	21170.25	125.7327	<0.0001	
C-Size electrode	3249	1	3249	19.29621	0.002	
AB	182.25	1	182.25	1.082405	0.3286	
AC	324	1	324	1.924276	0.203	
BC	2256.25	1	2256.25	13.40015	0.0064	
ABC	930.25	1	930.25	5.52487	0.0466	
Pure Error	1347	8	168.375			
Cor Total	33428	15				
			Model Summary			
s	R-sq		R-sq(Adj)		R-sq(Pre)	
12.98	95.97%		92.44%		83.88%	

Figure 4 depicts the half-normal plot and Pareto chart, which shows that the primary factors and interaction factors might positively or negatively affect electrocoagulation process COD and turbidity removal efficiencies. A half-normal plot (Figure 4a) shows that the non-significant ones fell along with a straight line normal distribution and centered near 0. Meanwhile, the essential factors contributed to COD removal efficiencies away from the straight line. Therefore, in order of significance, B (voltage), A (operation time), and C (size of the electrode) have the effects of the most critical factors in COD removal efficiency. Other significant factors included interaction between (BC) voltage and size of electrodes and (ABC) interaction between three factors, which are operation time, voltage, and size of electrodes (ABC). Factors with the tallest bars significantly affected the process in the Pareto chart. As depicted in Figure 4b, the Pareto chart had two lines: Bonferroni (3.584) and t value limit lines (2.306). The factors with t values above the Bonferroni line are essential. Also, the factors with t values between the Bonferroni and t value lines are significant. However, those with t values below the t value-line are not remarkable.

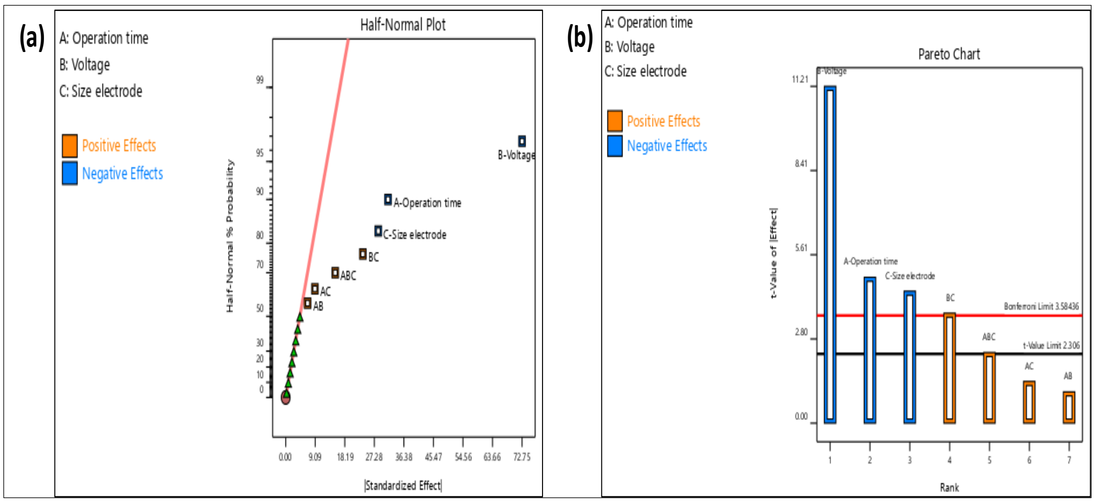

Figure 4. (a) Half-normal probability plot of the effects for the removal of COD and (b) standardized Pareto chart for COD

Effect of variables on turbidity removal

Table 5 shows the ANOVA test for the removal of turbidity. The models or variables with a *p*-value < 0.05 are considered significant with a 95% confidence level. Therefore, the result of ANOVA in Table 4 showed that all variables A, B, and C and the interactions of AB, AC, and BC are significant model terms in terms of turbidity, except for the ABC interaction. The model obtained a *p*-value of 0.0001, implying that the model is significant. The ANOVA result shows that the coefficient of determination, R^2, for the model was 0.9908, which means that the quality of the model is 99.08%. The adjusted R^2 for the model is 0.9522, which shows a good agreement between the results predicted by the regression design and experimental data. A reasonable agreement between the adjusted R^2 and predicted R^2 indicated an adequate model, as shown in Table 5. The final empirical models in terms of actual parameters are determined as follows,

$$\text{Turbidity} = +109.56 - 17.69 * A - 37.69 * B - 15.56 * C + 5.06 * AB + 6.19 * AC + 12.69 * BC + 4.94 * ABC \quad (9)$$

Table 5. ANOVA test for the removal of turbidity

Source	**Sum of Squares**	**df**	**Mean Square**	***F*-Value**	***P*-Value**	
Model	35594.44	7	5084.92	32.98	< 0.0001	significant
A-Operation time	5005.56	1	5005.56	32.46	0.0005	
B-Voltage	22725.56	1	22725.56	147.39	< 0.0001	
C-Size electrode	3875.06	1	3875.06	25.13	0.0010	
AB	410.06	1	410.06	2.66	0.1416	
AC	612.56	1	612.56	3.97	0.0814	
BC	2575.56	1	2575.56	16.70	0.0035	
ABC	390.06	1	390.06	2.53	0.1504	
Pure Error	1233.50	8	154.19			
Cor Total	36827.94	15				

Model Summary

s	R-sq	R-sq(Adj)	R-sq(Pre)
12.42	96.65%	93.72%	86.60%

The factorial design plot, including the half-normal plot and Pareto chart of effects, are shown in Figure 5. The half-normal probability plot of the effects in Figure 5a shows the absolute values of the estimated effects from the largest effect to the smallest effect. From this analysis, it could be observed that the largest effect on turbidity removal efficiencies that is well displaced in the plot was the AB interaction (operation time and voltage), followed by B (voltage), AC interaction (operation time and size of electrode), BC interaction (voltage and size of electrode), A (operation time) and (C) size of the electrode. The sequence of the significant main effects with respect to decreasing influence on turbidity removal is found to be AB > B > AC > BC > A > C > ABC. The half-normal plot of the effects indicated that the single factor has a negative effect. By increasing the level, there is a significant reduction in turbidity in landfill leachates - another quick way to screen the significant factors is by analyzing the Pareto chart of the estimated effects. The Pareto chart is generated and tabulated, as shown in Figure 5b, to validate the results obtained from the half-normal probability plot of effects in Figure 5a. Pareto chart analyses revealed that all factors and interaction factors are statistically significant, and none of them was below the limit of Benferroni (3.46).

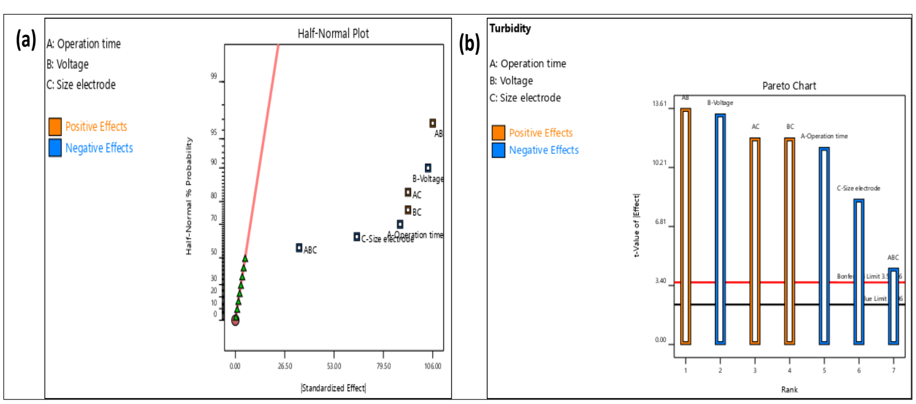

Figure 5. (a) Half-normal probability plot of the effects for the removal of turbidity and (b) standardized Pareto chart for turbidity

Model validation and multiple response optimization
An additional experiment is performed for each set of experiments to validate the models further. Optimized conditions and corresponding maximum COD removal are depicted using the ramp graph shown in Figure 6. Under the optimum conditions, three replicate experiments are conducted. The conditions with predicted and measured results are listed in Table 6. The table shows that the three responses are close to those estimated using factorial design. The model's validity is confirmed as the variation coefficients were around 5% for COD and turbidity removals. This method provided savings in terms of the time and size of electrodes.

Electrolysis time also significantly affects the efficiency of electrochemical coagulation methods in removing pollutants. It determines how much coagulant is produced and how much the cycle costs [16]. Increasing electrolysis time up to the optimum level increases pollutant removal efficiency but does not increase it beyond the optimum level. The fact is that the formation of coagulants at constant current voltage increases with an increase in electrolysis time, resulting in increased efficiency for removal [17]. The voltage applied to the electrodes is one of the most critical parameters that affect the efficiency and economy of the electrocoagulation cycle [18]. Figure 7 shows a sample of leachate landfill before and after the electrocoagulation process. A clear solution is obtained after using the electrocoagulation process by using parameters in this experime

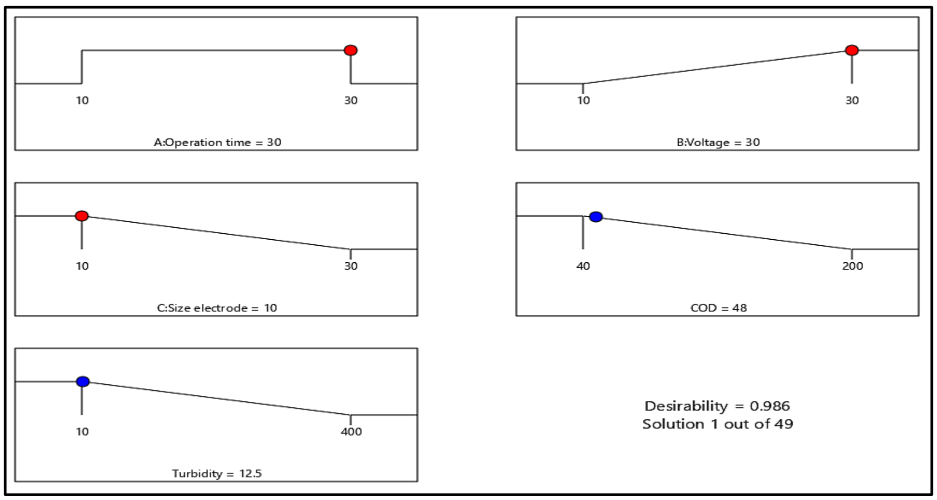

Figure 6. Suggestion value for optimization model

Table 6. Validation of the model

Removal Efficiencies	Factors			Predicted	Average Experimental Results
	Operation Time (min)	Voltage (V)	Electrode Size (m^2)		
COD (mg/l)	30	30	10	48.00	45.72
Turbidity (NTU)	30	30	10	12.5	11.87

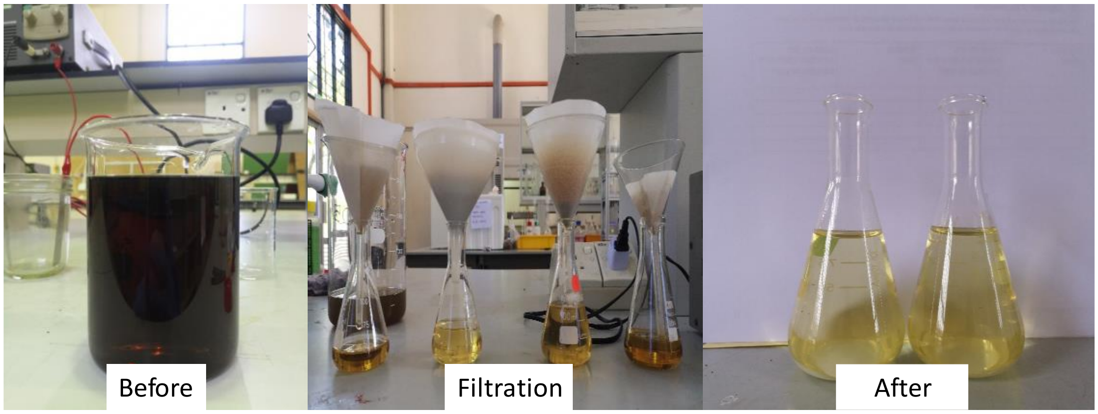

Figure 7. Sample leachate landfill before and after electrocoagulation

Conclusion

In this study, the FFD design of the experiment was used to observe the impact of voltage, operation time, and size of electrodes for the removal of COD and turbidity from landfill leachates by the electrocoagulation process. The screening experiment for the EC process was obtained by running 16 combinations of experiments provided by FFD design in the design of experiment software. The optimum condition was achieved by keeping the goal of operating time maximum, voltage maximum, size of electrode minimum, COD, and turbidity pollutant removal efficiencies in the "maximized" condition. The statistical analysis showed that the relevant p-value of each model was less than 0.001 (p-value < 0.001), indicating the corresponding significance of the model for all responses. The predicted values of COD and turbidity were 48 mg/l and 12.5 NTU, respectively. The maximum COD and turbidity removal experimental values were 45.72 mg/l and 11.87 NTU, respectively, at 30V, 30 min, and 10 cm electrode sizes. Therefore, this indicated that the electrocoagulation process gave good response for the treatment of leachate samples and agreed with the predicted values with less than 5% deviation.

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