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Factorial Design for Optimization and Performance Evaluation of Palm Oil Mill Effluent (POME) using Electrocoagulation

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Abstract: The palm oil industry needs a more effective method to treat palm oil mill effluent (POME) before being released into the river without polluting the environment. Thus, the best possible solution for treating POME is using electrocoagulation. This study focused on the factorial experimental design for optimization and performance evaluation in electrocoagulation treatment. This technique uses a direct current source between metal electrodes immersed in the effluent, which causes the dissolution of electrode plates into the effluent. At an appropriate pH, the metal ions can form a wide range of coagulated species and metal hydroxides that destabilize and aggregate the particles, which later precipitate and adsorb the dissolved contaminants. The results showed that the characteristics of untreated pollutants exceeded the Department of Environment (DOE) standard limit. Therefore, the pollutants need to undergo several treatments before being released into the river. After going through the electrocoagulation process, R^2 values of 0.7262 and 0.6566 shows a good agreement between experimental and predicted values of responses. Besides, the best removal for this process was recorded at a POME concentration of 10%. This study confirmed that the electrocoagulation process of POME could be treated even in high pollutants concentration, and the effluent discharge quality was achieved well below the standard limits of DOE.

Keywords: electrocoagulation; factorial design; palm oil mill effluent; regression equation

1. Introduction

Predominantly, Malaysia is one of the largest exporters in the world for palm oil. However, this agricultural product has added nearly 80 percent of industrial emissions containing high effluents discharged from the processing mills. Generally, the sludge from palm oil, known as palm oil mill effluent (POME), is brown, and this slurry contains 4 to 5 percent solids, mostly organic, 0.5 to 1 percent of residual oil, and almost 95 percent of water

[1,2]. Mainly, POME has highly contained liquid waste from sterilization and clarification in the palm oil milling process. This fluid refers to the effluent coming from the factory's final stages of palm oil processing. Since it contains a high volume of composition and nutrients, the effluent from palm oil mills can harm the environment. The discharge will result in land and water contamination if left untreated.

There are three significant POME sources: sterilizing the fresh fruit bunch (FFB), clarifying the extracted crude palm oil, and pressing the empty fruit bunch (EFB). POME has been identified as the primary cause of pollution in the water bodies due to the untreated effluent discharge to the river. The liquid contained a high concentration of biochemical oxygen demand (BOD_5), chemical oxygen demand (COD), and organic matter with complex composition in the presence of different sugar formations such as arabinose xylose, glucose, and mannose [3]. The biodegradation process of this organic matter is complicated; thus, the liquid is toxic to the ecosystem, especially the river biodiversity. The anaerobic pond method is commonly employed to treat POME in most palm oil industries. The process is cheap, but it is not adequate to cater to all the wastewater produced from the palm oil mill process. The treatment requires a more extended retention period, high maintenance cost in terms of workforce, and, most of all; it consumes a large area of land to accommodate the pond [4]. Due to the scarcity of land, the palm oil industry needs more effective treatment to treat POME to be released into the river without polluting the environment [5,6]. Thus, this paper evaluated the potential technologies focusing on electrocoagulation to minimize the contaminants from the POME.

Electrocoagulation is a treatment technique based on the coagulation method. Coagulation emphasizes a chemical concept using coagulants like alum and ferric sulfate. The process involves the dissolution of metal cations from the reactor anode with the simultaneous formation of hydroxyl ions and hydrogen gas at the cathode. The hydroxyl ions will react to the coagulant and form micro flocs when the particle is attached to the different ion charges (negative and positive). The concept applied for conventional coagulation treatment is almost the same, but electrocoagulation uses an electric supply to treat the water [7,8]. The electrocoagulation treatment method was tested successfully for treating phenolic wastewater [9], olive mill wastewater [10], sewage [11], textile wastewater [12], poultry industry wastewater [13], microplastics from wastewater [14], removal of lead and phosphate recovery from sludge anaerobic supernatant. Other than low management costs, this method is safe, natural, and environmentally friendly. This technique has the ability to treat many types of wastewater. It can also be the best possible solution for treating POME for a shorter period compared to the conventional method.

2. Materials and Methods

The palm oil mill effluent (POME) is collected at the study area from Palm Oil Mill Technology Centre (POMTEC), Labu, Negeri Sembilan, Malaysia. The POME sample was collected in the raw form before POME was transferred to the pond for the anaerobic process. A certain amount of POME is taken and kept in polyethylene containers and closed tightly. Then, the samples are brought to the laboratory and stored in the refrigerator at 5°C. This is to avoid disturbing the POME sample and any changes in the properties (decomposition) of the samples. The POME samples collected were then tested in the School of Civil Engineering, College of Engineering, Universiti Teknologi MARA, Shah Alam, Selangor, Malaysia.

The characteristics of untreated and treated POME from electrocoagulation were done in the laboratory. The parameters conducted were pH value, turbidity, biochemical oxygen demand (BOD_5), chemical oxygen demand (COD), ammonia-nitrogen (NH_3-N), phosphorus (P), nitrate-nitrogen (NO_3^--N), nitrite-nitrogen (NO_2^--N), total suspended solids (TSS) and total dissolved solids (TDS) for both untreated and treated POME [15].

The raw sample of POME parameters must be tested and analyzed in different dilutions to show the potential of electrocoagulation as an alternative to POME treatment. Therefore, the POME was diluted into four different sets of dilution. Four different sets of dilution samples were 1:100, 1:10, 1:5, and 1:3.333, with percentage concentrations of 1%, 10%, 20%, and 30%, respectively. These dilution sets suit the reactor design based on the experiment setup. Table 1 shows the details of the sample dilution prepared in this study.

Table 1. Details of sample dilution conducted in this study.

Percentage Concentration (%)	POME Volume (ml)	The volume of Distilled Water (L)	Total Volume (L)
1	0.2	19.8	20
10	2.0	18.0	20
20	4.0	16.0	20
30	6.0	14.0	20

2.1. Reactor configurations.

In this study, the electrocoagulation treatments were done in a fabricated electrocoagulation reactor. Figure 1 shows the schematic diagram of the electrocoagulation reactor that was used in this study. To set up the reactor, a fundamental concept of electrolysis is applied following Figure 2, which includes three units of direct current power supply (DC), a series of cathode and anode, and the electrolyte. The electrolyte is a medium that provides the ion transport mechanism between the anode and cathode necessary to sustain the electrochemical process (in this research is the POME samples).

The reactor was fabricated using acrylic with 600 mm x 300 mm x 300 mm. The acrylic thickness was 8 mm. Twenty (20) pieces of aluminum electrodes with a thickness of 1 mm were attached vertically inside the reactor. The dimension of the aluminum electrode was 250 mm x 284 mm. These electrodes represent the anode and cathode for the reactor, with the distance between the anode and cathode being 16 mm. In addition, the distance between each cell was 40 mm. The ten cells were divided into three sections, as shown in Figure 2. Sections 1 and 2 consisted of three sets of cells each, and section three consisted of four. All the cells were connected to a current power supply (DC) at 18 volts. The POME was poured into the reactor before applying the connection to the power supply. The POME liquid needs to be fully covered on the surface of the cells to allow the electrocoagulation process in the reactor.

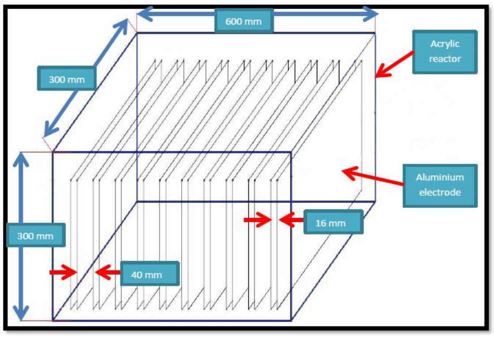

Figure 1. The schematic diagram of the electrocoagulation reactor.

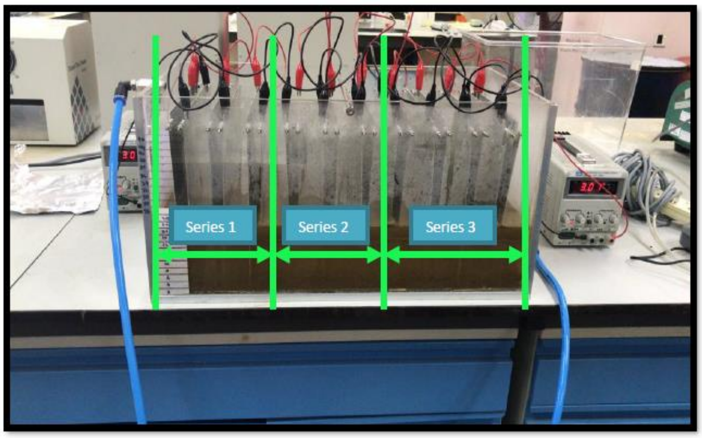

Figure 2. The electrocoagulation reactor in the Environment Laboratory in the School of Civil Engineering, College of Engineering, UiTM Shah Alam.

2.2. Electrocoagulation process.

Four dilutions were used for the POME sample, and each was analyzed in the fabricated reactor. The retention time for the electrocoagulation process in this study was set for 180 minutes in each dilution. For every 30-minute interval, 10 ml of treated effluent is collected in a beaker to calculate the percentage removal of contaminants. The treated effluent is collected at the outflow valve at the lower bottom of the reactor.

The electrocoagulation treatments were conducted at the same room temperature in the laboratory. Table 2 shows the detailed time interval of the electrocoagulation process performed in this study.

Table 2. Details of dilution and time intervals were conducted in this study.

Percentage Concentration (%)	**Time Interval (Minutes)**					
1	30	60	90	120	-	-
10	30	60	90	120	-	-
20	30	60	90	120	150	180
30	30	60	90	120	150	180

2.3. Electrocoagulation process.

The formula used to calculate the Removal Efficiency Percentage (REP) after the electrocoagulation process is described below. The value of the removal efficiency percentage is calculated using Equation 1 [16]:

$$\%\ \text{Removal} = \frac{c_0 - c_1}{c_0} \times 100 \qquad \text{Equation 1}$$

Where;
C_0 = Initial concentration of contaminant
C_1 = Final concentration of contaminant

2.4. Factorial design using design of experiment (DOE)

The relationship between the three-factor variables of Factor A (Duration), Factor B (Sludge Concentration), and Factor C (Parameter) with the response for the BOD_5, COD, ammonia nitrogen, and phosphorus reduction was analyzed by a full general factorial design by the DOE from Minitab software [17].

3. Results and Discussion

3.1. Characteristics of Palm Oil Mill Effluent (POME) Before and After Electrocoagulation Process.

The results of four different percentage concentrations of raw palm oil mill effluent (POME) taken from the Palm Oil Mill Technology Centre (POMTEC) before and after the electrocoagulation process are shown in this section.

3.1.1. pH.

Table 3 shows the characteristics of pH for untreated and treated POME from Palm Oil Mill Technology Centre (POMTEC). The pH results after electrocoagulation for POME concentration (1%, 10%, 20%, 30%) were 5.94, 7.06, 5.99, and 5.94, respectively. This showed that treated POME's pH after electrocoagulation is within the DOE standard limit [18], which is between 5.0 to 9.0. Neutralizing pH occurred due to the coagulant produced (OH^-) from the reaction of the cathode in the electrolysis process [19]. Hydrogen evolution at the cathode makes the electrode surrounded with alkaline, thus neutralizing the POME solution. Based on the result, the pH value obtained in raw POME is lower than the DOE standard [18] (5.0-9.0), with a pH of 3.50.

According to [20], the duration of treatment plays a significant role in neutralizing pH conditions in an aqueous solution. The longer retention time added to the treatment would result in a neutral state in the solution. Based on the experimental evaluation, 10% of POME concentration showed the best neutralization, as the final pH was 7.06. The electrocoagulation process succeeded in neutralizing the solution from acidic to neutral pH. As for 1%, 20%, and 30% of POME concentration, the result showed significant increments from acidic to neutral pH. This was reflected by the alkalinity produced during electrolysis which is sufficient to increase the pH value in the solution. This result indicated that the electrocoagulation treatment could neutralize the acidic condition of POME in the sample to become slightly neutral. The results of pH for all four treated effluent concentrations from the reactor were found in the range of DOE standard (pH 5 – 9) [18].

Table 3. The pH characteristics of untreated and treated POME collected from the Palm Oil Mill Technology Centre (POMTEC).

Parameters	**Untreated POME**	**Treated POME**				**DOE standard limit**
		POME Concentration (%)				
		1	**10**	**20**	**30**	
pH	3.5	5.94	7.06	5.99	5.94	5.00-9.00

3.1.2. Turbidity.

Table 4 shows the characteristics of turbidity for untreated and treated POME from Palm Oil Mill Technology Centre (POMTEC). The table shows that the turbidity before treatment was 4521.5 NTU. Meanwhile, after treatment for POME concentration (1%, 10%, 20%, 30%), it shows 362.5, 64, 412.5, and 461.62 of NTU, respectively. The results showed that the electrocoagulation process had removed a significant value of turbidity in the treated samples for POME concentration (1%, 10%, 20%, 30%) with percentage removal of 91.98%, 98.58%, 90.88%, 89.79%, separately The removal of turbidity for 30% of POME concentration was low due to the high concentration of POME in the sample. However, this result showed that the electrocoagulation treatment could remove a higher turbidity value in the sample if retention time is prolonged. It has been reported that the turbidity value has changed from 84.2 to 3.91 NTU after the electrocoagulation process using an aluminum electrode (removal of 95.36%) [21]. Moreover, the performance depends on significant parameters: a longer retention time will attain higher turbidity removal [12].

Table 4. The characteristics of turbidity for untreated and treated POME collected from Palm Oil Mill Technology Centre (POMTEC).

Parameters	Untreated POME	Treated POME				DOE standard limit
		POME Concentration (%)				
		1	10	20	30	
Turbidity (NTU)	4521.5	362.5	64	412.5	461.62	-

3.1.3. Biochemical oxygen demand (BOD_5).

Table 5 shows the characteristics of BOD_5 for untreated and treated POME from Palm Oil Mill Technology Centre (POMTEC). The concentration of BOD_5 in raw POME (15,800 mg/L) was very high compared with the DOE standard (100 mg/L) [18]. Meanwhile, results after electrocoagulation for POME concentration (1%, 10%, 20%, 30%) are 3,600.0 mg/L, 2,380.0 mg/L, 3,000.0 mg/L, and 3,399.66 mg/L, respectively.

According to [22], the high biochemical oxygen demand (BOD5) value indicated fecal contamination or an excessive volume of microorganisms in the water sample. Therefore, it can be denoted that the results of BOD_5 have reduced significantly after the electrocoagulation process. However, the effluent of treated POME for all percentage concentrations did not comply with the DOE standard limit [18]. Nevertheless, there are many microorganisms have been eliminated after the electrocoagulation process. The highest removal of BOD_5 was in the POME concentration of 10%. The lowest removal was observed from the POME concentration of 1%. This showed that the electrocoagulation reactor had effectively removed the microorganisms. It can be denoted that a greater amount of BOD_5 could be removed if an extension is imposed in the duration time [23].

It can be presumed that biological oxygen demand (BOD_5) is an important water quality parameter as it provides an index to assess the effect of effluent discharged on receiving water bodies. BOD_5 directly affects the amount of dissolved oxygen in rivers and streams. The higher the BOD_5 value, the greater the amount of organic matter or "food" available for oxygen-consuming bacteria [22,23]. The larger amount of BOD_5, the more oxygen will rapidly be depleted in the stream. This means less oxygen is accessible to higher forms of aquatic life. Depleting dissolved oxygen (DO) causes stress on aquatic organisms, making the environment unsuitable for any life forms.

Table 5. The characteristics of BOD_5 for untreated and treated POME collected from Palm Oil Mill Technology Centre (POMTEC).

Parameters	Untreated POME	Treated POME				DOE standard limit
		POME Concentration (%)				
		1	10	20	30	
BOD_5 (mg/L)	15,800.00	3600	2380	3000	3399.66	100

3.1.4. Chemical oxygen demand (COD).

Table 6 shows the characteristics of COD for untreated and treated POME from Palm Oil Mill Technology Centre (POMTEC). The result showed that COD for untreated POME is high, with a value of 40,013 mg/L. According to [24], the COD value at Seri Ulu Langat Palm Oil Mill was 98,010 mg/L. It showed that the COD value at POMTEC is lower than the COD value at Seri Ulu Langat Palm Oil Mill [24]. A high concentration of COD indicated that extensive pollutants existed in the sample, which would cause severe pollution in the water. The excessive COD levels disclosed a tremendous amount of oxidizable organic material present in the sample, which will decrease the dissolved oxygen (DO) in the water. A reduction in DO can lead to anaerobic conditions, harming aquatic life [25]. Chemical oxygen demand (COD) will be soaring if organic matter is also high in the water sample [23]. After electrocoagulation for COD for all POME concentrations, the result was 5500 mg/L, 3302.4 mg/L, 4785 mg/L, and 4486.22 mg/L, respectively. However, the treated POME shows the electrocoagulation process was not adequately removed most of the COD in the diluted POME samples within 180 minutes. This is because the aluminum hydroxide produced in the electrolyte is insufficient to remove all the pollutants in the samples in the given time frame. Thus, a long time needs to be imposed in the electrocoagulation process to reduce the COD value.

Table 6. The Characteristics of COD for Untreated and Treated POME collected from Palm Oil Mill Technology Centre (POMTEC).

Parameters	**Untreated POME**	**Treated POME**				**DOE standard limit**
		POME Concentration (%)				
		1	**10**	**20**	**30**	
COD (mg/L)	40,013.00	5500	3302.4	4785	4486.22	-

3.1.5. Ammonia-nitrogen (NH_3-N).

Table 7 shows ammonia-nitrogen characteristics for untreated and treated POME from Palm Oil Mill Technology Centre (POMTEC). The value of ammonia-nitrogen from POMTEC was 387.54 mg/L which is higher than the allowable limit by DOE [18]. Wastewater rich in ammonia-nitrogen will prevent natural nitrification, cause water hypoxia, contribute to fish toxicity, limit water purification efficiency, and potentially cause major harm to the water system [26]. Ammonia-nitrogen can form ammonia in water under some conditions, which is also highly soluble and can be easily used for algae growth. It can, however, be harmful to marine ecosystems at elevated concentrations. High quantities of ammonia in the rivers are mainly from direct discharges of toxins such as untreated or poorly treated effluent [26].

The results after treatment for POME concentrations of 1%, 10%, 20%, and 30% were 47.67 mg/L, 27.50 mg/L, 135.83 mg/L, and 143.87 mg/L, respectively. The initial characteristic of raw POME for ammonia-nitrogen was 387.54 mg/L. Table 7 shows that 10% of POME concentration gives the highest ammonia-nitrogen removal, and the lowest is from 30% of POME concentration. Slight removal from this sample was due to the low generation of aluminum hydroxide produced in the electrolyte. According to [27], aluminum ions and hydroxide concentration will accumulate when the electrolysis period increases. Furthermore, after treatment for all four concentrations, the result was below the allowable limit of DOE [18].

Table 7. The characteristics of ammonia nitrogen for untreated and treated POME collected from Palm Oil Mill Technology Centre (POMTEC).

Parameters	**Untreated POME**	**Treated POME**				**DOE limit**
		POME Concentration (%)				
		1	**10**	**20**	**30**	
NH_3-N (mg/L)	387.54	47.67	27.5	135.83	143.87	150

Therefore, the electrocoagulation process had sufficiently removed the ammonia-nitrogen, and this method can be considered the best solution for reducing ammonia-nitrogen in wastewater.

3.1.6. Phosphorus (P).

Table 8 shows the characteristics of phosphorus for untreated and treated POME from Palm Oil Mill Technology Centre (POMTEC). The result of phosphorus for untreated POME was 545.1 mg/L. Meanwhile, after treatment, the phosphorus results for all percentage concentrations were 20 mg/L, 0.6 mg/L, 2.5 mg/L, and 8.33 mg/L, respectively. High phosphorus concentrations (545.1 mg/L) from untreated POME must be due to the existence of this substance in the samples. The highest removal was from the POME concentration of 10%, and the lowest was 1%. However, phosphorus reduction gradually decreased in 180 minutes, showing that electrocoagulation could efficiently remove this contaminant.

Nutrients are essential for plant growth, but an overabundance of nutrients in water can have many harmful effects on the surrounding environment. Too much phosphorus can increase the growth of algae and large aquatic plants, resulting in decreased levels of dissolved oxygen. This process is called eutrophication [28]. Extreme phosphorus levels can also lead to algae blooms that produce algae toxins harmful to human and animal health. Algae feed on the nutrients, growing, spreading, and turning the water green. Algae blooms can create a bad smell and block the sunlight. After the algae have decayed, they decompose by bacteria, which consume the water's dissolved oxygen (DO). This scenario will deplete the DO, and the environment will turn hypoxic, creating a dead zone and leading to the death of aquatic life. Human activities can accelerate eutrophication by increasing the rate of nutrients entering the water bodies. Therefore, eliminating these pollutants is essential to retain the ecosystem's survival.

Table 8. The characteristics of phosphorus for untreated and treated POME collected from Palm Oil Mill Technology Centre (POMTEC).

Parameters	Untreated POME	Treated POME				DOE standard limit
		POME Concentration (%)				
		1	10	20	30	
P (mg/L)	545.1	20	0.6	2.5	8.33	-

3.1.7. Nitrate-nitrogen (NO_3-N) and nitrite-nitrogen (NO_2-N).

Table 9 shows the characteristics of nitrate-nitrogen and nitrite-nitrogen for untreated and treated POME from Palm Oil Mill Technology Centre (POMTEC).

Table 9. The characteristics of nitrate and nitrite for untreated and treated POME collected from Palm Oil Mill Technology Centre (POMTEC).

Parameters	Untreated POME	Treated POME				DOE standard limit
		POME Concentration (%)				
		1	10	20	30	
Nitrate-nitrogen (mg/L)	5.25	0	0.1	0.35	0	-
Nitrite-nitrogen (mg/L)	4.50	0	1.5	1.5	1.0	-

At 1% of POME concentration, the nitrate-nitrogen and nitrite-nitrogen gave the highest removal of pollutants after the electrocoagulation process, 0 mg/L for both contaminants. Before the electrocoagulation process, the value of these pollutants was 5.25 mg/L for nitrate-nitrogen and 4.5 mg/L for nitrite-nitrogen. This showed that electrocoagulation removes 100% of these pollutants at 1% of POME concentration. The results showed that aluminum hydroxide is adequate in the electrocoagulation process to degrade the nitrate-nitrogen and nitrite-nitrogen in the samples.

3.1.8. Total Dissolved Solid (TDS) and Total Suspended Solids (TSS).

Table 10 shows the characteristics of TDS and TSS for untreated and treated POME from Palm Oil Mill Technology Centre (POMTEC). From the results in Table 10, POME contains a high quantity of TSS and TDS. This could be attributed to the high solid content emanating from the palm fruit. The excessive solid content emerges from the leaching process involved in palm oil processing [29]. Following that, the palm oil mill effluents accumulated with solids are carried along with water during the process. TSS concentration after treatment in all four types of diluted POME samples is within the DOE discharge limit (400 mg/L) [18]. This is due to the acceptable production of aluminum hydroxide in the POME solution, thus removing the TSS. Furthermore, the TDS concentration in all four types is still higher after treatment. However, it decreased slightly after the electrocoagulation process was applied in the reactor.

From the result obtained, all the parameters before treatment did not comply with the DOE standard [18], higher than the allowable limit [30]. Thus, the POME from POMTEC needs to undergo further treatment before being released to the river to avoid contamination of the environment and deleterious biota [31]. However, some parameters do not comply with the DOE standard limit [18] after treatment. This is due to the short period in treating the pollutants. Therefore, if the electrolysis process were extended after 180 minutes, a higher amount of pollutants could be eliminated. Comprehensive treatment for electrocoagulation in terms of a higher incubation rate substantially reduces the number of pollutants in the POME sample. [12].

Table 10. The characteristics of TSS and TDS for untreated and treated POME collected from Palm Oil Mill Technology Centre (POMTEC).

Parameters	Untreated POME	Treated POME				DOE limit
		POME Concentration (%)				
		1	10	20	30	
TSS (mg/L)	4525	100	90	120	79.99	400
TDS (mg/L)	7864	3433.33	2996.67	6081.67	5772.76	-

3.2. Statistical analysis using full factorial design in Minitab software.

This full factorial design by the Design of Experiment (DOE) was used to predict the final concentration of the characteristic's removal. This experiment consisted of three factors used in the factorial design in DOE using Minitab software [17]. The period of experimentation is denoted as A, POME concentration as B, and the parameter is labeled as C. The A factor had seven levels: minutes of 0, 30, 60, 90, 120, 150, and 180, while the B factor had four levels, which were 1%, 10%, 20%, and 30% of sludge concentration samples. Lastly, the C factors had four levels which were BOD_5, COD, ammonia nitrogen, and phosphorus. Table 11 shows the percentage removal for every concentration and duration of the parameter for the factorial design of DOE [17]. It can be observed that POME concentration is high at the beginning of the experiment and reduced towards the end of the experiment. This shows that electrocoagulation successfully removes BOD_5, COD, ammonia-nitrogen, and phosphorus.

Table 11. Percentage removal for every concentration and duration of the parameter for the factorial design of DOE.

Percentage Removal (%)

Parameter (Factor C)	**POME Concentration (Factor B) (%)**	**Duration (Factor A) (Minutes)**						
		0	30	60	90	120	150	180
BOD$_5$	1	0	10	12	40	80	-	-
	10	0	55	60	69	77	-	-
	20	0	20	47	53	55	68	70
	30	0	20	30	43	60	65	70
COD	1	0	36.5	40	60	65	-	-
	10	0	30	43	61	75	-	-
	20	0	18	30	48	60	74	86
	30	0	20	30	31	50	74	86
Ammonia Nitrogen	1	0	70	80	82	85	-	-
	10	0	28	35	40	68	-	-
	20	0	8	20	25	40	57	59
	30	0	18	20	22	30	37	45
Phosphorus	1	0	18	88	90	92	-	-
	10	0	76	88	98	99	-	-
	20	0	36	38	70	90	98	88
	30	0	28	30	37	72	98	98

3.2.1. Regression analysis from the factorial design of DOE.

Figure 3 shows the factorial design analysis and the factors with percentage removal for the POME sample. Graph A (Figure 3) shows the mean of the percentage removal within the experiment period, starting with 0 minutes until 180 minutes. While graph B (Figure 3) shows the mean percentage removal for each concentration, beginning with 1% to 30% POME concentration. Lastly, graph C (Figure 3) shows the mean percentage removal for the four parameters. Each point in the graph represents the mean of the response (percentage removal) variable for each factor's various levels (minute, sludge concentration, and parameters).

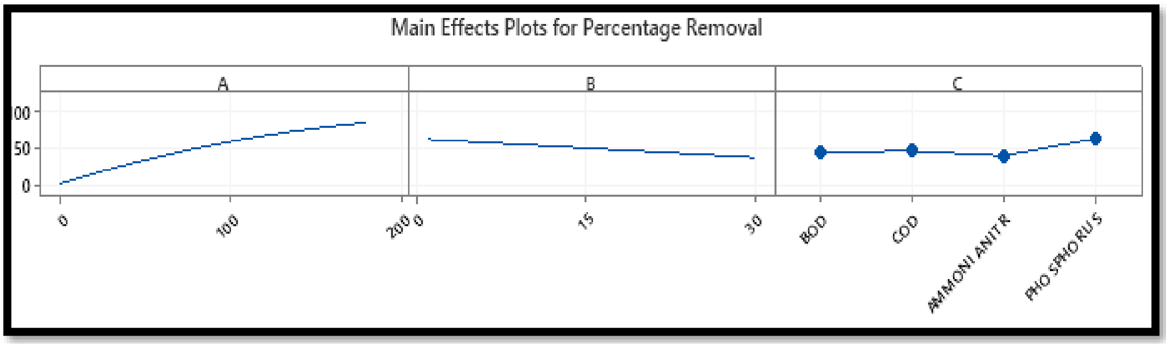

Figure 3. Factorial Design Analysis and the factors that affect the removal of each parameter from the POME sample.

Figure 3 shows the analysis of the factors affecting the percentage removal of pollutants. The plot points are the means of the response variable at the various levels of each factor, with a reference line drawn at the grand mean of the response data. The main effects plot is used for comparing the magnitudes of the main effects. The main effects of duration (A), percentage concentration (B), and parameter (C) have a significant impact on the percentage removal. In this study, the time interval significantly affected the removal percentage for every parameter. The mean of the percentage removal increased as the duration of the experiment was extended. It can be observed that ammonia-nitrogen concentration and duration can be the two main factors that affect ammonia-nitrogen removal [31].

As shown in Figure 3, the initial percentage concentration of 1% has a high removal percentage. The duration of 180 minutes gave a high percentage of removal of pollutants, indicating that the percentage of removal tends to increase when the duration increases. The mean percentage of removal increases in a polynomial manner by increasing the duration. In Figure 3, Graph B shows that the mean percentage removal from 1% to 30% reduced substantially as the concentration percentage soared. Meanwhile, graph C in Figure 3 indicates that phosphorus gives the highest removal percentage compared to the other pollutants. This means that phosphorus easily reacted to the aluminum hydroxide in the electrolyte, and therefore electrocoagulation was sufficient to eradicate the phosphorus. The three mentioned effects enhanced the reactor's removal of pollutants.

3.2.2. Regression Equation and Model Summary (R^2 value) of the study.

Figure 4 shows the interaction effect of duration, percentage concentration, and parameter on percentage removal. The interaction plot creates a single interaction plot for three independent variables (duration, percentage concentration, and parameters). An interactions plot is a plot of means for each level of an independent variable with the level of a second independent variable held constant. Interaction occurs when the response (percentage removal) at a factor level depends on other variables' levels. The greater the departure of the lines from the parallel state, the higher the interaction. Consequently, as mentioned earlier, the interaction shows significant relations between the independent variables on the response, as confirmed with Equation 2 evaluated in Minitab software [17].

$$\begin{aligned}\textbf{RESPONSE} = {} & 51.75 - 51.75\,(A\_0) - 21.03\,(A\_30) - 8.56\,(A\_60) + 2.56\,(A\_90) \\ & + 16.87\,(A\_120) + 27.77(A\_150) + 34.14\,(A\_180) + 8.06\,(B\_1) \\ & + 10.73\,(B\_10) - 6.47\,(B\_20) - 12.32\,(B\_30)\end{aligned} \qquad \text{Equation 2}$$

Where;

A: Duration

B: Percentage concentration

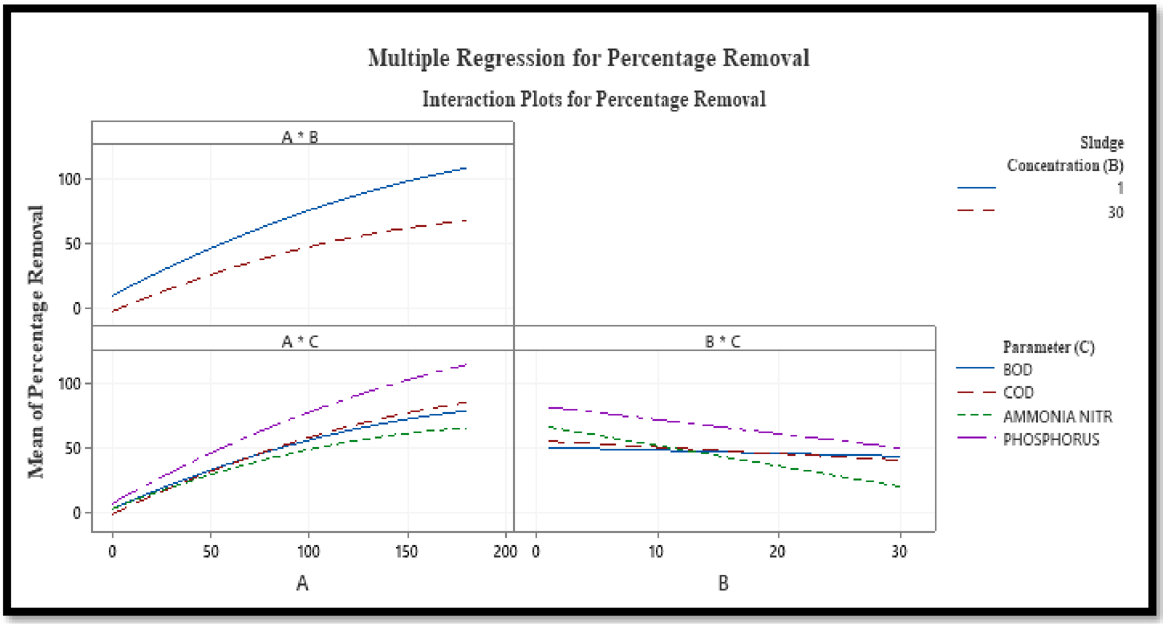

Figure 4. Interaction plot for percentage removal between the duration, percentage concentration, and parameter.

Minitab 17 is a software that developed the regression equations by the main effect plot [17]. Other studies reported that operation time and current density are the main effects that significantly influence the percentage removal for each parameter. The regression model explains that the removal efficiency is optimized to find the maximum removal level [32]. Meanwhile, in this research, the duration of time and percentage concentration is the significant factor for the regression model for the percentage removal of each parameter in developing the regression equation. To create a response surface regression model, a general polynomial model was applied [33] to the experimental observations of the response (percentage removal of pollutants); thus, a quadratic regression model was obtained as shown in Equation 2. "A" indicates the duration started from 0 minutes to 180 minutes. "B" indicates the concentration percentage started from 1% to 30%. Lastly, the response is the percentage removal.

Table 12. Model summary of the study.

R^2	R^2_{adj}	R^2_{pred}	S
0.7262	0.6976	0.6566	0.1709

The values of R-squared (R^2), adjusted R-squared (R^2adj), predicted R-squared (R^2pred), and the standard error (S) of the estimate are used to determine how well a regression model fits the data. The values are shown in Table 12. R^2 value (also called the coefficient of determination), which is the proportion of variance in the dependent variable, can be explained by the independent variables. Table 12 shows the R^2 value is 0.7262, which is the independent variables (duration, percentage concentration, and parameter) that explain 72.62% of the variability of the dependent variable (percentage removal). Besides, 27.38% (100% - 72.62% = 27.38%) of the variation is caused by factors other than the predictors included in this model.

R-squared seems like an easy-to-understand statistic that indicates how well a regression model fits a data set. However, it does not tell the entire story. In order to get the full picture, one must consider the R^2 value in combination with residual plots, other statistics, and in-depth knowledge of the subject area [34]. Adjusted R-Squared (R^2adj) is another crucial factor. The adjusted R-squared value is 0.6976, which indicates a 69.76% variation in the outcome variable. The low discrepancy between R-squared and Adjusted R Square values, 2.86% (72.62% - 69.76% = 2.86%), indicates a good model fit.

The standard error, S (0.1709) of a model fit, measures the model's precision. It is the standard deviation of the residuals. It shows how wrong it could be if the regression model is used to make predictions or estimate the dependent variable or variable of interest. As R^2 increases, the standard error will decrease. In other words, the estimates of a dependent variable (percentage removal) with this model will be wrong by 0.1709. The standard error is smaller, below 0.5 (50%). It is used to get a confidence interval for the predicted values [34]. Therefore, the regression is precise since the standard error is small.

3.2.3. Response surface plots.

This study investigated the interactions between dependent and independent process variables using response surface plots. Figure 5 and Figure 6 show the contour two dimensional (2D) and surface plot of 3 dimensional (3D) response (percentage removal) between duration and percentage concentration.

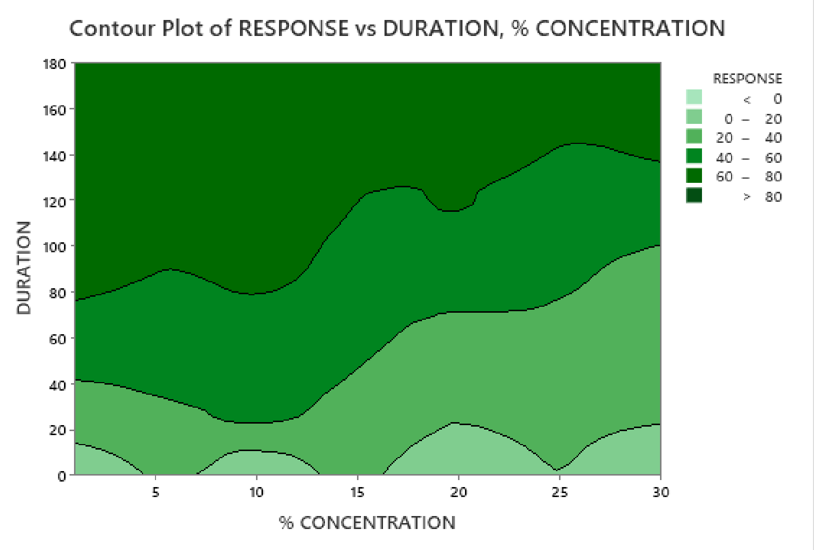

Figure 5. Contour plot for a response (percentage removal) between duration and percentage concentration.

The values for two-factor variables are represented on the y-time and x-percentage concentration axes in a contour plot. Homogeneous shaded zones, called contours, represent the value for a response percentage removal. This plot presents the overall distribution of the electrocoagulation process. As shown, an increased percentage of removal of pollutants was observed with increasing operation time and percentage concentration values. However, both factors increase an optimum zone for effective percentage removal within or over a dark shade. The duration is a critical process variable for electrocoagulation. This process starts with the neutralization of particles by ions released from the electrodes and coagulation [35 – 36]. An increase in the electrolysis period boosts the release of ion concentration, and in this way, the formation of hydroxyl flocks is improved [37 - 38]. This study's reaction periods longer than 80 minutes provided sufficient pollutant removal efficiencies (>80%).

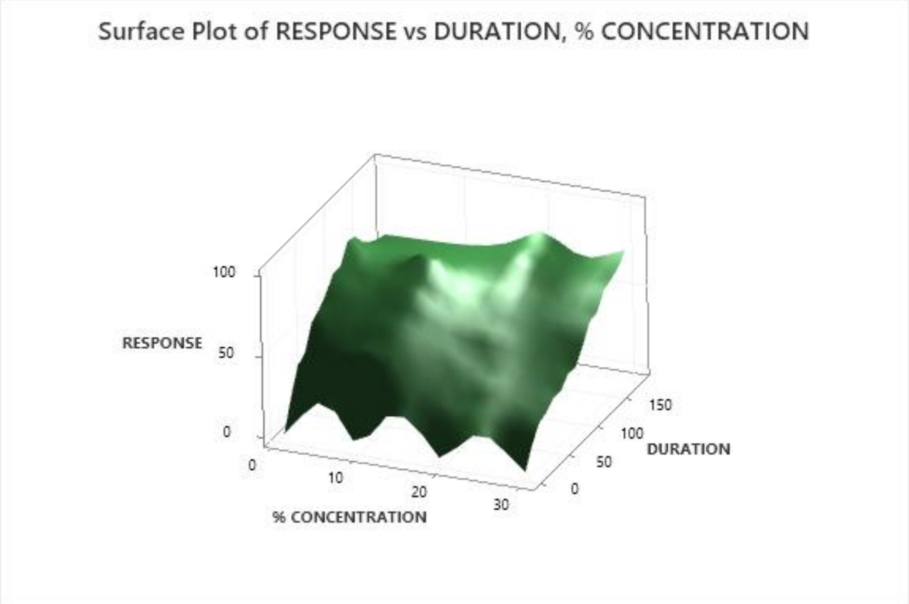

Figure 6. Surface plot for a response (percentage removal) between duration and percentage concentration.

Figure 6 shows the 3D response surface plot of the percentage removal model, indicating the percentage removal of pollutants on the z-axis display and the factor variables, which are duration and percentage concentration on the x and y-axis, respectively. Meanwhile, the y-axis represents the current density, and the x-axis represents the operation time [33]. An irregular surface defines the resulting data from Figure 6 with slightly higher plate zones at the right end of the duration plate. According to [38], the surface plot's uneven surface is found to increase slightly at the right end plate for both axes.

4. Conclusions

In conclusion, untreated POME contains a very harmful substance that can cause a significant environmental problem if this effluent is not treated properly before being released into the river. This study's regression analysis generated from the mathematical model equations has been evaluated to predict the pollutants' removal efficiency for COD, BOD_5, ammonia-nitrogen, and phosphorus. R^2 values of 0.7262 and 0.6566 show a good agreement between experimental and predicted values of responses. Besides, the best removal for this process was recorded at a POME concentration of 10%. This study also confirms that by the electrocoagulation process of POME wastewaters, high pollutants concentration can be treated using this method and is adequate to achieve the effluent quality below the DOE standard limits.

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Conflicts of Interest

The authors declare no conflict of interest. The funders had no role in the study's design; in the collection, analyses, or interpretation of data; in the writing of the manuscript, or in the decision to publish the results.

References

1. Kamyab, H.; Chelliapan, S.; Din, M.F.M.; Rezania, S.; Khademi, T.; Kumar, A. In palm oil mill effluent as an environmental pollutant.; Intechopen: London, United Kingdom, **2018**; https://doi.org/10.5772/intechopen.75811.
2. Mohammad, S.; Baidurah, S.; Kobayashi, T.; Ismail, N.; Leh, C.P. Palm oil mill effluent treatment processes—A review. *Processes* **2021**, *9*, 739. https://doi.org/10.3390/pr9050739.
3. Tanikkul, P.; Boonyawanich, S.; Pisutpaisal, N. Production of Methane from Ozonated Palm Oil Mill Effluent. *Int. J. Hydro. Energy* **2019**, *44*. 29561-29567, https://doi.org/10.1016/j.ijhydene.2019.08.210.
4. Khadaroo, S.N.B.A.; Poh, P.E.; Gouwanda, D.; Grassia, P. Applicability of Various Pretreatment Techniques to Enhance the Anaerobic Digestion of Palm Oil Mill Effluent (POME). *J. Environ. Chem. Eng.* **2019**, *7*, 103310. https://doi.org/10.1016/j.jece.2019.103310.
5. Ratnasari, A.; Syafiuddin, A.; Boopathy, R.; Malik, S.; Mehmood, M. A.; Amalia, R.; Prastyo, D. D.; Zaidi, N.S. Advances in pretreatment technology for handling the palm oil mill effluent: Challenges and prospects. *Bioresource Technology* **2022,** *344*, 126239. https://doi.org/10.1016/j.biortech.2021.126239.

6. Razman, K.K.; Hanafiah, M.M.; Mohammad, A.W.; Lun, A.W. Life Cycle Assessment of an Integrated Membrane Treatment System of Anaerobic-Treated Palm Oil Mill Effluent (POME). *Membranes* **2022,** *12,* 246. https://doi.org/10.3390/membranes12020246.
7. Mohamad, Z.; Razak, A.A.; Krishnan, S.; Singh, L.; Zularisam, A.W.; Nasrullah, M. Treatment of palm oil mill effluent using electrocoagulation powered by direct photovoltaic solar system. *Chemical Engineering Research and Design* **2021,** *177,* 578–582. https://doi.org/10.1016/j.cherd.2021.11.019.
8. Zhou, L.; Liu, L.; Qiao, W.; Gao, Y.; Zhao, Z.; Liu, D.; Bian, Z.; Wang, J.; Wang, Z.L. Improving Degradation Efficiency of Organic Pollutants through a Self-Powered Alternating Current Electrocoagulation System. *ACS Nano* **2021,** *15,* 19684−19691. https://doi.org/10.1021/acsnano.1c06988.
9. Zhao, C.; Zhou, J.; Yan, Y.; Yang, L.; Xing, G.; Li, H.; Wu, P.; Wang, M.; Zheng, H. Application of coagulation/flocculation in oily wastewater treatment: A review. *Sci. Total Environ.* **2021**, *765.* https://doi.org/10.1016/j.scitotenv.2020.142795.
10. Jovanović, T.; Velinov, N.; Petrović, M.; Najdanović, S.; Bojić, D.; Radović, M.; Bojić, A. Mechanism of the electrocoagulation process and its application for treatment of wastewater: A review. *Advanced Technologies* **2021**, *10,* 63–72. https://doi.org/10.5937/savteh2101063j.
11. Soler, P.; Faria, M.; Barata, C.; Garcia-Galea, E.; Lorente, B.; Vinyoles, D. Improving water quality does not guarantee fish health: Effects of ammonia pollution on the behaviour of wild-caught pre-exposed fish. *PLoS ONE*, **2021**, *16,* 1–17. https://doi.org/10.1371/journal.pone.0243404.
12. Al-Raad, A.A.; Hanafiah, M.M. Removal of inorganic pollutants using electrocoagulation technology: A review of emerging applications and mechanisms. *J. Environ. Manage.* **2021**, *300.* https://doi.org/10.1016/j.jenvman.2021.113696.
13. Eryürük, K.; Eryürük, S.; Un, U. T.; Ogutveren, U, B. A design of experiment approach of cattle slaughterhouse wastewater treatment by electrocoagulation method. *Desalin. Water Treat.* **2021**, *238,* 105–116, https://doi.org/10.5004/dwt.2021.27762.
14. Elkhatib, D.; Oyanedel-Craver, V.; Carissimi, E. Electrocoagulation applied for the removal of microplastics from wastewater treatment facilities. *Separation and Purification Technology* **2021,** *276,* 118877. https://doi.org/10.1016/j.seppur.2021.118877.
15. APHA. *Standard Methods for the Examination of Water and Wastewater*, 21st Edition.; Eaton, A.D.; Clesceri, L.S.; Greenberg, A.E.; America Public Health Association: Washington D.C., **2005**, https://www.scirp.org/(S(i43dyn45teexjx455qlt3d2q))/reference/ReferencesPapers.aspx?ReferenceID=267816.
16. Aniyikaiye, T. E.; Oluseyi, T.; Odiyo, J.O.; Edokpayi, J.N. Physico-chemical analysis of wastewater discharge from selected paint industries in Lagos, Nigeria. *Int. J. Environ. Res. Public Health* **2019**, *16, 1235,* https://doi.org/10.3390/ijerph16071235.
17. Minitab. Getting Started with Minitab Statistical Software. **2017,** https://www.minitab.com/content/dam/www/en/uploadedfiles/documents/getting-started/MinitabGettingStarted_EN.pdf.
18. DOE. Department of Environment. Environmental Requirement: A Guide for Investors. Ministry of Natural Resources and Environment, **2010**, October, 1-78, https://enviro2.doe.gov.my/ekmc/wp-content/uploads/2016/08/1403056822-A%20Guide%20For%20Investors%20-%202010.pdf.
19. Ahangarnokolaei, M. A.; Ayati, B.; Ganjidoust, H. Simultaneous and sequential combination of electrocoagulation and ozonation by Al and Fe electrodes for DirectBlue71 treatment in a new reactor: Synergistic effect and kinetics study. *Chemosphere* **2021**, *285.* https://doi.org/10.1016/j.chemosphere.2021.131424.
20. Nasrullah, M.; Wahid, Z.A.; Krishnan, S.; Sakinah, M.; Singh, L.; Fen, Y.W. High-Performance Electrocoagulation Process in Treating Palm Oil Mill Effluent Using High Current Intensity Application. *Chinese Journal of Chemical Engineering* **2019,** *22,* 208–217, https://doi.org/10.1016/j.cjche.2018.07.021,
21. Aoudj, S.; Khelifa, A.; Drouiche, N. Removal of Fluoride, SDS, Ammonia and Turbidity from Semiconductor Wastewater by Combined Electrocoagulation–Electroflotation. *Chemosphere* **2017**, *180,* 379-387, https://doi.org/10.1016/j.chemosphere.2017.04.045.
22. Abu Bakar, S.N.H.; Abu Hasan, H.; Mohammad, A.W.; Sheikh Abdullah, S.R.; Haan, T.Y.; Ngteni, R.; Yusof, K.M.M. A review of moving-bed biofilm reactor technology for palm oil mill effluent treatment. *Journal of Cleaner Production* **2018,** *171,* 1532–1545. https://doi.org/10.1016/j.jclepro.2017.10.100.
23. Bote, M. E. Studies on electrode combination for COD removal from domestic wastewater using electrocoagulation. *Heliyon* **2021,** *7.* https://doi.org/10.1016/j.heliyon.2021.e08614.

24. Tan, D.T.; Chin, S.K.; Poh, P.E.; Lee, Y.H. Preservation of thermophilic mixed culture for anaerobic palm oil mill effluent treatment by convective drying methods. *Int. J. Environ. Sci. Technol.* **2018**, *15,* 1211–1222. https://doi.org/10.1007/s13762-017-1486-9.
25. Li, D.; Liu, S. Water Quality Detection for Lakes. *Water Quality Monitoring and Management*, **2019**, 221–231. https://doi.org/10.1016/b978-0-12-811330-1.00008-9.
26. Kasmuri, N.; Lovitt, R.W.; Omar, M. Consumption of ammonia-nitrogen by AOB in immobilized batch culture. *J. Fundament. Appl. Sci.* **2018**, *9,* 257. https://doi.org//10.4314/jfas.v9i6s.2.
27. da Costa, J. G. R; Costa, J. M.; Neto A. F. A. Recent advances and future applications in electro-adsorption technology: An updated review. *J. Environ. Chem. Eng.* **2021**, *9,* 106355. https://doi.org/10.1016/j.jece.2021.106355.
28. Lemley, D.A.; Adams, J.B. Eutrophication. *Encyclopedia Ecology*, 2nd ed.; Fath, B.; Elsevier: Oxford, United Kingdom, **2019**, 86–90. https://doi.org/10.1016/B978-0-12-409548-9.10957-1.
29. Nwabanne, J.T.; Oguegbu, O.O.; Agu, C.M. Removal of Solids from Palm Oil Mill Effluent and Paint Wastewater Using Electrocoagulation Technique. *Int. J. Electrochem.* **2018**, 1–9. https://doi.org/10.1155/2018/4349639.
30. Cheng, Y.W.; Chang, Y.S.; Ng, K.H.; Wu, T.Y.; Cheng, C.K. Photocatalytic restoration of liquid effluent from oil palm agroindustry in Malaysia using tungsten oxides catalyst. *J. Clean Prod,* **2017**, *162,* 205–219. https://doi.org/10.1016/j.jclepro.2017.06.023.
31. Kasmuri, N.; Misni, M.Z. Ammonia-Nitrogen and Phosphate Removal in Leachate using Algae and Bacteria Mixture. *Int. J. Recent Technol. Eng.* **2019**, *8,* 7007–7012. https://doi.org/10.35940/ijrte.d5193.118419.
32. Ozyonar, F. Optimization of operational parameters of electrocoagulation process for real textile wastewater treatment using Taguchi experimental design method. *Desalination and Water Treatment* 2**016**, *57*, 2389–2399. https://doi.org/10.1080/19443994.2015.1005153.
33. Alaa N, G. Application of Response Surface Methodology to Optimize Nitrate Removal from Wastewater by Electrocoagulation. *International Journal of Scientific & Engineering Research* **2013**, *4,* 1410–1416. https://doi.org/10.14299/ijser.2013.10.003.
34. Dhakal, C.P. Interpreting the basic outputs (SPSS) of multiple linear regression. *Int. J. Sci. Res.* **2018,** 4–9, https://www.ijsr.net/archive/v8i6/4061901.pdf.
35. Ebba, M.; Asaithambi, P.; Alemayehu, E. Investigation on operating parameters and cost using an electrocoagulation process for wastewater treatment. *Appl. Water Sci.* **2021**, *11*. https://doi.org/10.1007/s13201-021-01517-y.
36. Igwegbe, C. A.; Onukwuli, O. D.; Ighalo, J. O.; Umembamalu, C. J. Electrocoagulation-flocculation of aquaculture effluent using hybrid iron and aluminium electrodes: A comparative study. *Chem. Eng. J. Adv.* **2021**, *6*, 100107. https://doi.org/10.1016/j.ceja.2021.100107.
37. Liu, Y.; Zhang, X.; Jiang, W. M.; Wu, M. R.; Li, Z. H. Comprehensive review of floc growth and structure using electrocoagulation: Characterization, measurement, and influencing factors. *Chemical Engineering Journal* **2021**, *417*, 129310. https://doi.org/10.1016/j.cej.2021.129310.
38. Veli, S.; Özbay, B.; Arslan, A.; Çebi, E. Optimization of process variables for treatment of food industry effluents by electrocoagulation. *Global Nest Journal* **2018,** *20,* 551–557. https://doi.org/10.30955/gnj.002640.