

Comparison of Palm Oil Mill Effluent Electrocoagulation by Using Fe-Fe and Al-Al Electrodes: Box-Behnken Design

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This present research aims to compare the COD removal of palm oil mill effluent (POME) by electrocoagulation using iron and aluminum electrodes. Response surface method coupled Box-Behnken design is applicable to investigate the effect of various process parameters (voltage, operating time and electrolyte concentration) and optimum condition. The optimum condition by using iron electrode is in 43 minutes, 20 volts and 0.06 M NaCl with the COD removal of 95.045%. By using electrode aluminum, optimum condition is 40.35 minutes, 19 volts and 0.1 M NaCl with the COD removal of 89.941%. Energy consumption of iron electrode and aluminum electrode are 0.189 and 0.209 kWh/mg decomposed COD with energy costs of Rp. 255 and Rp. 282/mg decomposed COD. This indicates that energy and cost consumption of electrocoagulation by using Al electrode are higher than those by using iron electrode. Based on the investigation during research, iron electrode is better for reducing COD while the aluminum is better in removing color in POME.

Keywords : Palm Oil Mill Effluent (POME), COD, electrocoagulation, electrode, Box Behnken Design

INTRODUCTION

The palm oil industry is one of industries that consume much enough water in its process and production. It is estimated that every ton of raw palm oil processed for production needs about 5.0 – 7.5 tons of water (Wu et al. 2009). About 50% of water used will be wastewater discharged into a pond of wastewater

treatment (Ahmad et al. 2010). This wastewater is known as palm oil mill effluent (POME). POME is brownish wastewater. It becomes brownish because of lignin and its group, tannin, humic acid, lipid, and fatty acid presence that come out during the hot steam process (Neoh et al. 2012). Physically, it is dark brownish gray, highly viscous and stinking. Raw POME highly contains colloidal suspension

that consists of about 96% water, 0.6 – 0.7% remaining oil and 5% total solids (Azmi et al. 2014). POME is generally brownish and contains a large amount of water, oil, suspended solid, dissolved solid and sand. The suspended components are mainly vegetative matters like cell walls, organelles, short fibers and water-soluble carbohydrates ranging from hemicelluloses to simple sugars (glucose, reducing sugars and pectin). They also cover nitrogenous compounds (from proteins to amino acids), free organic acids, lipids, as well as the mixture of minor organic material and mineral constituents (Foo et al. 2010).. Untreated POME has a pH range of 3.45 – 4.60, concentration of chemical oxygen demand (COD) 30,000 – 80,000 mg/L, BOD concentration 15,000 – 40,000, total suspended solid 1,500 – 50,000 mg/L and total solid 16,000 – 95,000 mg/L (Tetrattech 2010).

The common and the most famous treatment of POME is an open pond system. It consists of six until eight ponds; the process of every pond is different (Yulia 2015). The pond system covers sand and oil trap, cooling pond, acidification pond, anaerobic pond, facultative pond and aerobic pond. A sand and oil trap is a part of wastewater pre-treatment. Aerobic pond has a depth of the range of 1.0 – 1.5 m, whereas anaerobic pond depth typically ranges 5.0 – 7.0 m. An anaerobic pond could process large amounts of the solid and tend to need low cost (Lam et al. 2011) Open pond is commonly used by palm oil mills to treat POME through aerobic/anaerobic processes. In addition to its lacks such as long hydraulic

retention time (HRT) and large space need, effluent from the ponds also often fails to meet the discharge limits set by government (Bello et al. 2017)

Several other researchers investigated POME treatment such as anaerobic digestion (Singh et al. 2013), oxidation of aerobic active sludge (Vijayaraghavan et al. 2007), anaerobic and aerobic digestions (Chan et al. 2012), chemical flocculation and coagulation (Bhatia et al. 2007, Teh et al. 2014), membrane anaerobic system (Abdurrahman et al. 2011), up-flow anaerobic sludge fixed film (UASFF) reactor (Zinatizadeh et al. 2007), immobilized up-flow anaerobic sludge blanket (UASB) reactors (Abdurrahman et al. 2011), and adsorption (Mohammed et al. 2014). In Nasrullah work (2017) concluded that the above process still needed long time to remove pollutant substance in POME and needed many chemicals. Therefore, the process is more suitable to treat POME waste where its time treatment is short and the chemical impact of the process is more insignificant.

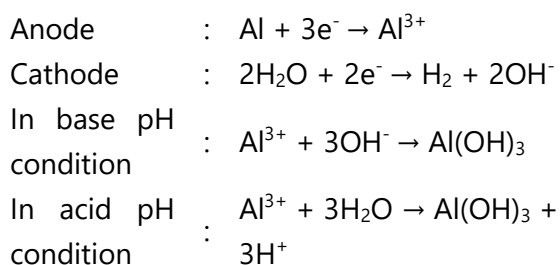
Another alternative to overcome problems arising from pond system and another process is to combine this system with other treatment systems. Treatment system used is electrocoagulation. Electrocoagulation process has been applicable in the olive-mill waste processing, canning (Kobya et al. 2013), surfactants, food processing, semiconductor, mechanical polishing, liquid organic fertilizer (Akyol et al. 2013), electroplating, livestock (Tak et al. 2015), textile wastewater (Behin et al. 2015), palm-oil mill effluent (Hanum et al. 2015, Nasution et al. 2014) winery wastewater

(Vepsilainen 2012) etc.

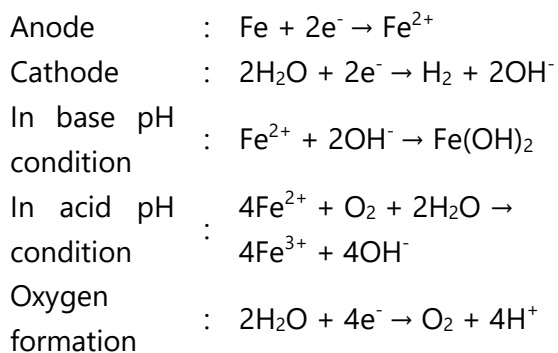
THEORY

Electrocoagulation is a coagulant production method with in situ process by applying an electric current through an anode that releases metallic ions into solution. A cathode will generate and recover hydrogen used as an energy source or reactants for other industrial applications. Floc formed has a large surface area that is able to adsorb dissolved organic compounds, and captures colloidal particles contained in solution (Mook et al. 2014).

The chemical reaction that occurs in the use of aluminum electrodes:



By using an iron electrode, the oxidation simplification and the reaction mechanism that occur are as follows.



Several parameters that influence the efficiency of electrocoagulation process in pollutant removal are as follows, (1) electrode generally used is iron, aluminum

or inert; (2) pH of solution influences the species formation from hydroxide metal in solution and zeta potential from a colloidal particle; (3) current density is a number of electrochemical reactions that occur in surface; (4) contact time and electrical charge applied will equal to coagulant resulted in electrocoagulation process; (5) electrode potential indicates particular reaction that occurs in electrode surface; (6) anion and electrolyte in pollutants such as sulfate and fluoride have the same property as hydroxide ion namely assisting pollutant precipitation; and (7) operation temperature influences the floc formation, chemical rate, and conductivity (Gumus et al. 2016). Hanum and Nasution (2015 and 2015) conducted the treatment of POME waste by electrocoagulation (EC). In the research, voltage and concentration of NaCl electrolyte solution used are relatively low. Based on the earlier research thus this research will use various electrode types, lower voltage, and contact time. Besides, energy consumption in both types of electrode will be calculated as economical consideration.

EXPERIMENT

Source of Wastewater, Characterizing, and Materials

Wastewater was collected from an anaerobic pond I at local palm oil mill PT Syaukath Seujahtera, Bireuen Regency, Aceh Province. This wastewater contained large amounts of organic compounds, had brown color, and smelled bad. It was put into a jerrycan and cooled in a refrigerator at a temperature of 4°C. The wastewater

characteristic after H₂SO₄ addition, such as pH, COD, TSS and TDS were 2; 7,000 mg/L (diluted by 10 times to 700 ppm); 4,720 mg/l (diluted by 10 times to 472 mg/L) and 1011 mg/l, respectively. COD in the pond was less than the COD in a fat pit that was usually in the range of 30,000 – 80,000 mg/l because the pond was the fourth stage in the wastewater treatment of oil palm factory.

Main equipment consisted of a reactor, electrodes, and power supply. Reactor EC was made of glass with a size of 14 cm × 12 cm × 15 cm. The electrode used consisted of two pairs of iron plates with a size of 15 cm × 10 cm × 0.5 cm. It was connected to DC power supply (Sanfix 30 A and 30 V). Chemicals used were NaCl and acetone (Merck). Research stages were started by cleaning electrode every time before and after running. Electrodes were sanded by using sandpaper until the surface of the electrode became slippery and shiny. Cleaning was continued with washing electrode by using acetone, and followed by rinsing with aquadest.

Research Procedure

The next stage was putting 1.3 L sample into a glass reactor. An amount of NaCl was added to the reactor in accordance with Box Behnken design (BBD). NaCl was added before electrical current was applied to the electrodes. In this research, sample kept agitated with magnetic stirrer at 100 rpm. Voltage set was adjusted to BBD. All runs were carried out in the room temperature namely 28°C. After the run was completed, the sample was filtered by using membrane with a pore size of 0.45 micrometer and the filtrate was collected

100 mL for COD analysis. Electrode was cleaned back by using ways mentioned above.

Experimental Design

Box-Behnken design was useful to adjust response surface, where formula was designed by a combination of 2^k factorial block with simple design. The designed result was typically very efficient to decide the number of experiments conducted. Response surface methodology (RSM) can figure out the relation between dependent process variables and the result. In order to find the relation, dependent variables for EC using electrode iron and aluminum were operation time (A), voltage (B), and electrolyte concentration (C). The experimental values of process variables for EC are shown in Table 1. Based on the dependent variables, Box-Behnken design conducts 17 experiments for both electrocoagulation processes.

Table 1. Input variable and code factor of response surface method with Box-Behnken for electrocoagulation process

Code Factor	Variable	-1	0	1
A	Time (minute)	15	30	45
B	Voltage (V)	10	15	20
C	Electrolyte concentration (M)	0.0	0.5	1.0

Chemical Oxygen Demand Analysis

Chemical oxygen demand was determined by referring to SNI.6989.2:2009 for organic and inorganic compound oxidized by Cr₂O₇²⁻ in closed reflux methods by using UV-VIS spectrophotometer model V-630. The

oxidant in the sample was determined by visible light 420 nm. Based on these results, the percentage of COD reduction was calculated from Equation 1.

$$\text{COD}_{\text{reduction}} (\%) = \frac{\text{COD}_0 - \text{COD}_t}{\text{COD}_0} \times 100 \quad (1)$$

COD₀ was initial COD and COD_t was remaining COD after electrocoagulation.

Energy Consumption

Electrochemical process consumed electrical energy. In this research, energy consumption in both electrocoagulation electrodes of iron and aluminum was determined in the optimum condition. The energy consumption was calculated by:

$$\text{Energy consumption (kWh/mg COD)} = \frac{I \times v \times t}{\Delta\text{COD} \times V} \quad (2)$$

I was a current intensity (A), v was voltage (V), t was a time of

electrocoagulation, V was the volume of solution (L), and ΔCOD was the COD reduction investigated in solution (Popescu et al. 2017).

RESULTS AND DISCUSSION

Response Analysis by Using Box – Behnken Design

This research focuses on the application of electrocoagulation processes in treating POME from a diluted anaerobic pond I. The dependent variables are voltage (10 – 20 V), operating time (15 – 45 minutes), electrolyte concentration (0.0 – 1.0 M), and electrode types (iron and aluminum), whereas a response variable in this treatment is COD reduction. By using the same variables, comparison of the COD removal is obtained in this research. This research applies analysis of variance (ANOVA) to investigate the model and the significant variables in the process. The result of COD reduction in both electrodes are shown in **Table 2** and **3**.

Table 2. Experimental design matrix by using iron electrode based on actual and predicted COD removal by BBD

Run	Time (min)	Volt (V)	NaCl (M)	COD removal (%)	
				Actual	Predicted
1	45	20	0.5	95.01	95.86
2	30	20	1.0	92.50	91.69
3	30	15	0.5	88.50	87.91
4	15	10	0.5	76.85	76.00
5	15	15	1.0	77.03	78.05
6	30	10	0.0	83.20	84.01
7	45	10	0.5	86.64	86.85
8	45	15	0.0	91.25	90.23
9	45	15	1.0	91.00	91.22
10	15	20	0.5	81.59	81.38
11	30	20	0.0	91.27	91.44
12	30	15	0.5	87.75	87.91
13	30	15	0.5	88.08	87.91
14	15	15	0.0	78.05	78.08
15	30	15	0.5	86.46	87.91
16	30	10	1.0	84.90	84.73
17	30	15	0.5	88.78	87.91

Table 3. Experimental design matrix by using aluminum electrode based on actual and predicted COD removal percentage by BBD

Run	Time (min)	Volt (V)	NaCl (M)	COD removal (%)	
				Actual	Predicted
1	45	20	0.5	89.68	89.85
2	30	20	1.0	89.53	89.13
3	30	15	0.5	84.02	84.97
4	15	10	0.5	70.44	70.27
5	15	15	1.0	73.36	72.91
6	30	10	0.0	83.70	84.10
7	45	10	0.5	88.69	87.84
8	45	15	0.0	87.01	87.46
9	45	15	1.0	87.01	87.24
10	15	20	0.5	78.78	79.63
11	30	20	0.0	89.53	88.92
12	30	15	0.5	85.24	84.97
13	30	15	0.5	84.31	84.97
14	15	15	0.0	74.22	73.99
15	30	15	0.5	85.02	84.97
16	30	10	1.0	81.98	82.59
17	30	15	0.5	86.28	84.97

The first run displays the highest value of POME COD reduction by an iron electrode with electrolysis time of 45 minutes, a voltage of 20 V, and sodium chloride concentration of 0.5 M. In such condition, the percentage of COD reduction obtained is 95.01%. Whereas the lowest COD percentage was in the fourth run at the time of 15 minutes, a voltage of 10 V and electrolyte concentration of 0.5 M that is 76.85%. By using aluminum electrode, the highest COD reduction obtained is 89.68% at the electrolysis time of 45 minutes, a voltage of 20 V and sodium chloride concentration of 0.5 M. The lowest COD removal with an aluminum electrode is 70.44% reported in electrolysis time of 15 minutes, a voltage of 10 V and NaCl concentration of 0.5 M.

As indicated in Table 2 and 3, the predicted response data are obtained from statistic model analysis by BBD in Design Expert. The predicted response is

from Equation 3 by using iron electrode and Equation 4 in using aluminum electrode. In the BBD matrix, electrolysis time is variable A (minute), voltage as variable B (volt) and NaCl concentration as variable C (M).

$$COD(\%) = 59.68 + 1.09 A - 0.03 B + 1.3 C + 0.01 AB + 0.03 AC - 0.05 BC - 0.01 A^2 + 0.01 B^2 - 1.15 C^2 \quad (3)$$

$$COD(\%) = 47.90 + 2.00 A - 0.41 B - 3.51 C + 0.02 AB + 0.03 AC - 0.17 BC - 0.01 A^2 + 0.05 B^2 - 0.57 C^2 \quad (4)$$

Analysis of variance (ANOVA) is useful as a parametric test to distinguish the average value of more than two data groups by comparing their variances and to find responses among all variables (Bezerra et al. 2008). Summaries of ANOVA for the usage of both electrodes to

Table 4. ANOVA of quadratic model for COD reduction with electrodes of Fe and Al

Electrode	Source	P-value	Remark
Fe-Fe	Model	< 0.0001	Significant
	A – Time	< 0.0001	
	B – Voltage	< 0.0001	
	C – NaCl concentration	0.5536	
	A²	< 0.0005	
	B ²	0.5403	
	C ²	0.6043	
	AB	0.1384	
	AC	0.6527	
	BC	0.8348	
	Lack of Fit	0.2512	Not significant
	R ²	98.29%	
	Adjusted R ²	96.08%	
Al-Al	Model	< 0.0001	Significant
	A – Time	< 0.0001	
	B – Voltage	< 0.0001	
	C – NaCl concentration	0.3644	
	A²	< 0.0001	
	B²	0.0213	
	C ²	0.7637	
	AB	0.0058	
	AC	0.6613	
	BC	0.3909	
	Lack of Fit	0.3887	Not significant
	R ²	98.90%	
	Adjusted R ²	97.48%	

determine significance of interaction between factor variable and the response variable is shown in Table 4. It predicts the value reduction of COD more than 90% by using both electrodes to approach the model. This R² value indicates that research result obtained is close to be true and significant by a quadratic model.

The effect of inter-variable interaction could be observed in **Table 4**. The probability p-value < 0.05 determines the interaction of significant or insignificant

variables. Values that are greater than 0.05 indicate the model terms are not significant. Significant variables in using iron electrode (Fe-Fe) are A (electrolysis time), B (voltage) and A² (quadrade of electrolysis time). For the Al-Al electrode, significant variables are A (electrolysis time), B (voltage), A² (quadrade of time electrolysis), B² (quadrade of voltage) and AB (interaction of time electrolysis and voltage). The significance implies that the variables have an important effect on COD

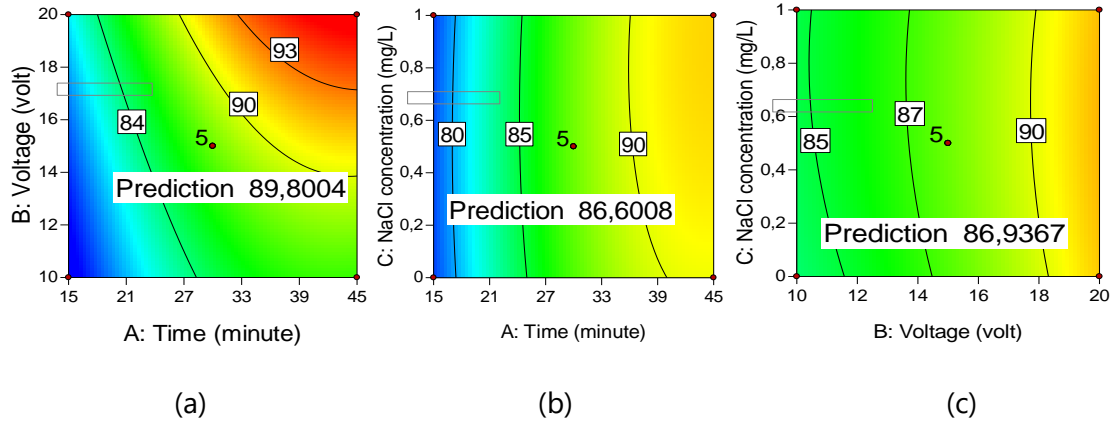


Fig. 1: Two-dimensional contour plots express the effect of time, voltage and electrolyte concentration on electrocoagulation by an iron electrode

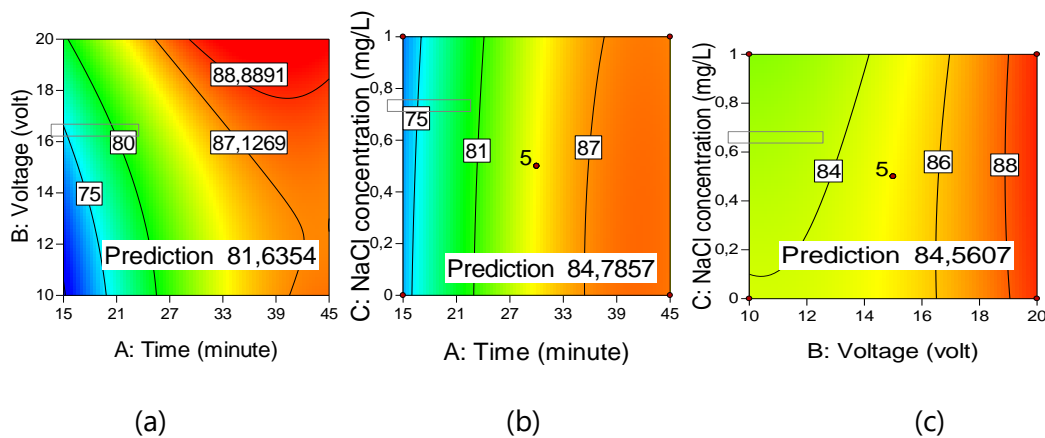


Fig. 2: Two-dimensional contour plots express the effect of time, voltage and electrolyte concentration on electrocoagulation by an aluminium electrode

reduction. P-value for lack of fit is above 0.05 value that indicates that the insignificance of lack of fit proves that model data are in good accordance with research data for the percentage of COD reduction (Moussa et al. 2016)

Effects of Variable Interaction in COD Reduction

The Interaction Effect of Electrolysis Time and Voltage

The analysis result of COD value in the effect of operation time and voltage by using two-dimensional response surface could be observed in the following **Figures 1** (a) and **2** (a) while time

electrolysis (X) and voltage (Y). Figures 1 (a) and 2 (a) show the effect of electrolysis time (A) and voltage (B) while the NaCl concentration is on the middle level (0.5 g/mL).

As the duration of the electrolysis increases, the highest removal of COD is obtained. While electrolysis increases from 15 to 45 minutes, the COD removal efficiency by using iron electrode increases to 93%. Figures 1 (a) and 2 (a) also indicates that as electrolysis time and voltage are longer, the COD removal efficiency increases. In the first 15 minutes, the COD removal was 76,85%. In the next 15 minutes and the higher voltage (15

volts), the efficiency of COD removal is 86 – 88%. By using aluminium electrode in process, COD removal also tends to increase from 75% to 88,88%.

Based on Figures 1 (a) and 2 (a), the higher voltage and time electrolysis enhance the rate of COD removal. The higher removal COD is obtained by using iron electrode. The iron could form bivalent or trivalent cations that depend on the pH and the potential of the solution. On the other hand, aluminum is only dissolved as trivalent cations (Akyol et al. 2015). By using Al electrode, aluminum hydroxide produces sludge. Al^{3+} ions are turned into hydrolysis reaction and aluminum hydroxide resulted produces more sludge with a consequently significant removal of color and COD (Khandegar et al. 2013). It is a deal that by increasing the voltage of the cell, the amount of hydrogen bubbles at the cathode increases, resulting in a greater upward flux and a faster removal of COD. As the bubble production increase, the organic matter settling much more better.

The Interaction Effect of Time Electrolysis and NaCl Concentration

A vital factor in electrocoagulation is time of electrolysis. By increasing operation time, pollutant in the wastewater could be contacted more and the chance in removal the pollutant in the wastewater is higher. Another factor that takes place in this research is a variation of electrolyte concentration. Both Figures 1 (b) and 2 (b) show two-dimensional contour plots by keeping the voltage as constant in 15 volts. As the electrolysis is getting longer, the COD reduction is

higher. In using aluminum electrode, in the range 0.0 – 1.0 M, the COD removal has no effect. In 45 minutes of electrolysis time, 15 volts of voltage without NaCl and with 1 M NaCl, the COD reduction is the same namely 87.01%. By using iron electrode with the same variable mentioned before, COD removal shows slightly decreased from 78.05% to 77.03%.

The aim of electrolyte concentration addition is to enhance the solution conductivity to decrease the energy consumption. Electrolyte solutions often used are NaCl, Na_2SO_4 , NH_4Cl , and $(NH_4)_2SO_4$. Because of low-cost and easy availability, NaCl has been selected as the best electrolyte and chosen as the electrolyte solution in this research (Behin et al. 2013, Gumus et al. 2016). In this research before starting the electrocoagulation process, the solution is in acid condition at pH of 2.0 by using H_2SO_4 . The chloride and nitrate ion existence could prevent the effect of sulfate ion existence by breaking passive layers formed. The chloride ion presence also much decreases the side effect of sulfate ion that causes salt precipitation in an electrode if the salt concentration is relatively high and layer formation occurs (31). Acid addition enables sulfate ion obtained from acid addition that aims to keep sample preserved. By adding electrolyte concentration, the effect in COD reduction is not significant (see the data in Tables 2 and 3). The NaCl absence in solution gives the higher COD reduction. It could be stated that the process in both electrodes in electrocoagulation is more effective at a lower concentration of NaCl (below 0.5 M

or without NaCl).

The Interaction Effects of Voltage and NaCl Concentration

In some research, the popular variable used in electrocoagulation is current density. Current density leads to the current per area of an electrode that determines the number of metal ions released from the electrodes (Akyol et al. 2013). In this research, voltage is used to control the current supply in the reactor. Generally, electrode dissociation is proportional to the applied voltage or current. The higher intake voltage results in the higher chance of electrode dissociation. However, optimum variables are necessary to block the excessive and large consumption of energy in the process. Figures 1 (c) and 2 (c) indicate the correlation of voltage and electrolyte concentration in COD removal efficiency.

The high intensity of voltage gives the higher reduction of COD. In Figures 1 (c) and 2 (c) up to 10 – 18 volts, COD reduction improves near to 90% in maximum voltage. In this case, electrolyte concentration is eligible as mentioned beforehand in section before. There is a high chance of energy wasting in heating the water and a decrease in current efficiency expressed as the ratio of the voltage or current consumed to produce a certain product to the total current consumption (32). In this research, high voltage using (20 V, 30 minutes, 1 M NaCl) in both electrodes increases final temperature. In electrode iron, the final temperature reaches 62°C while the final temperature by using electrode aluminum is 59°C. Even though the COD removal is

higher at the high voltage use, another side of energy use is also an important case in finding the optimum condition in this process.

Comparison of Using Iron and Aluminum Electrodes

This section is an overall view of the process variables and energy consumption by using both electrodes. Both electrodes have different excellence in electrocoagulation. The BBD can support to identify the significant factor in removing COD from the POME by electrocoagulation. In POME treatment, the higher COD removal is resulted mostly by using iron electrode. Using an iron electrode produces yellowish water that represents the oxidized iron. Using aluminum as electrode gave clearer wastewater of final effluent but the remained COD is higher than by using iron electrode. The overall COD reduction is indicated in Tables 2 and 3. The higher voltage by using iron electrode gives the higher final temperature of effluent while the final temperature of wastewater by using aluminum is slightly lower. Energy consumption for both electrodes is obtained at the optimum condition. This optimum condition is set by using response surface method with Box-Behnken design that refers to theoretical and experimental data. A higher desirability (closest to 1) determines the optimum condition. The desirability function or Derringer function is the most important and most recently used multi-criteria methodology in the analytic procedure optimization. This method is initially based on constructing a

desirability function for each response (Bezerra et al. 2008). The goal is to set the parameters in the range of process variable. Based on the response surface method of BBD, the optimum condition in using iron electrode is in 43 minutes of electrolysis time, 20 volts, and 0.06 M NaCl with the COD removal of 95.045%. By using electrode aluminum, optimum condition is 40.35 minutes, 19 volts, and 0.1 M NaCl with the COD removal of 89.941%.

In order to find the efficiency of the different processes performed, in this study the energy consumption per amount of degraded COD is calculated at the optimum operating variables. According to Equation (2), energy consumption by using iron electrode in 43 minutes of electrolysis time, 20 volts, and 0.06 M NaCl is 0.189 kWh/mg COD degraded. By using electrode aluminum, optimum condition in 40.35 minutes, 19 volts, and 0.1 M NaCl indicates that energy consumption 0.209 kWh/mg COD degraded. In Indonesian Rupiah, 1 kWh equals to Rp. 1,350. Based on this value, the energy cost in each electrode is Rp. 255 kWh and Rp. 282/kg COD mg degraded. This indicates that energy and cost consumption for electrocoagulation by using iron electrode is found lower than electrocoagulation by using aluminum electrode.

CONCLUSIONS

The effects of different electrodes, electrolysis time, voltage, and electrolyte concentration on COD removal are investigated in this research. The experimental conditions are optimized by

observing the effect of interactions among variables on COD removal efficiency by using a response surface methodology (RSM) with Box-Behnken design. Both high R^2 values in using iron and aluminum electrode are 96.08% and 97.48%, respectively. COD removal verified by ANOVA shows that the accuracy of the proposed quadratic model is acceptable (accuracy above 90%). As the time electrolysis and voltage increase, COD removal efficiency in both electrodes also improves. Based on the model from Box-Behnken design, the most influential factor is electrolysis time, voltage, and interaction of time electrolysis and voltage in using aluminum electrode. The optimum condition in using iron electrode is in 43 minutes of electrolysis time, 20 volts and 0.06 M NaCl with the COD removal of 95.045%. By using electrode aluminum, optimum condition is 40.35 minutes, 19 volts and 0.1 M NaCl with the COD removal of 89.941%. Based on the investigation during research, electrode iron is better for reducing COD while the aluminum is better for color removal of POME. Energy and cost consumption for electrocoagulation by using iron electrode is lower than that by using aluminum electrode. This research indicates that the electrocoagulation process by using iron electrode is the most successful process that removes COD in the POME.

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