



Optimizing electrocoagulation process for the treatment of biodiesel wastewater using response surface methodology

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Abstract

The production of biodiesel through a transesterification method produces a large amount of wastewater that contains high levels of chemical oxygen demand (COD) and oil and grease (O&G). Currently, flotation is the conventional primary treatment for O&G removal prior to biological treatments. In this study, electrocoagulation (EC) was adopted to treat the biodiesel wastewater. The effects of initial pH, applied voltage, and reaction time on the EC process for the removal of COD, O&G, and suspended solids (SS) were investigated using one factor at a time experiment. Furthermore, the Box-Behnken design, an experimental design for response surface methodology (RSM), was used to create a set of 15 experimental runs needed for optimizing of the operating conditions. Quadratic regression models with estimated coefficients were developed to describe the pollutant removals. The experimental results show that EC could effectively reduce COD, O&G, and SS by 55.43%, 98.42%, and 96.59%, respectively, at the optimum conditions of pH 6.06, applied voltage 18.2 V, and reaction time 23.5 min. The experimental observations were in reasonable agreement with the modeled values.

Key words: biodiesel wastewater; electrocoagulation; Box-Behnken design; response surface methodology

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Introduction

Biodiesel is one of the most promising alternative fuels in Thailand, particularly when its price is comparable with standard diesel. Biodiesel, the mono alkyl esters of long-chain fatty acids, is an alternative fuel for diesel engines produced by chemically reacting vegetable oil or animal fat with an alcohol. In Thailand, the government has promoted the production and the use of biodiesel as a substitute diesel fuel to reduce the importation of oil, enhance the energy security, and promote the use of alternative energy made from domestic crops. The government projects that the quantity of biodiesel sold in the market in 2012 will account for 10% of the total diesel sold (Gonsalves, 2006). At present, the existing capacity of biodiesel production is approximately 1.5 million liters per day with 43 biodiesel plants registered with the Department of Industrial Work (Department of Alternative Energy Development and Efficiency, 2008).

Over 90% of the biodiesel production in Thailand uses palm oil as the raw material by a transesterification reaction method using alkali catalysis. This method produces a high conversion of oil (triglycerides) to biodiesel (methyl esters) by a simple chemical reaction which occurs in a short time (Marchetti *et al.*, 2007). In the final process

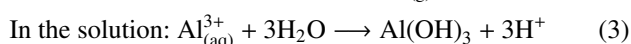
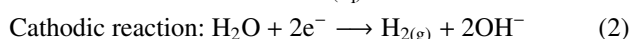
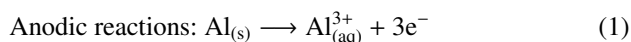
of biodiesel production, water is usually introduced into the biodiesel to remove impurities. This washing step is repeated 2–5 times dependent on the quantity of impurities in the methyl ester. A large amount of wastewater, 20–120 liters per 100 liters of biodiesel, is generated in this process (Suehara *et al.*, 2005; Marchetti *et al.*, 2007; Phukingngam *et al.*, 2008). The wastewater is basic (alkaline), with a high content of oil and grease, and a low content of nitrogen and phosphorus. As such, biological treatment of the biodiesel wastewater is expected to be very difficult (Suehara *et al.*, 2005).

In Thailand, typical treatments of oily wastewater employ a dissolved air floatation technique (DAF), an oil and grease (O&G) trap unit or other commercial O&G removal units to separate oil and grease prior to biological treatments. However, these conventional flotation techniques are not satisfactory for removing emulsified oils without chemical pretreatment (Moursy and Abo-Elela, 1982). Al-Shamrani *et al.* (2002) reported that the addition of aluminum or ferric salts, highly charged cations, can enhance the destabilization of an oil-water emulsion when applying with DAF. Saatci *et al.* (2001) studied the treatability of vegetable oil industry effluents using the DAF technique with lime, alum, or ferric chloride as chemical coagulants. Their results showed that ferric chloride was the most effective coagulant in reducing chemical oxygen

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demand (COD) and O&G. Although physicochemical methods appear to be effective in removing O&G from oily wastewater, they require handling chemicals, and their operations are relatively complicated.

Electrocoagulation (EC) has been widely and successfully introduced to treat numerous wastewaters including leachate from solid wastes, municipal wastewater, dying wastewater, and wastewater containing organic species such as phenol (Deng and Englehardt, 2007; Bukhari, 2007; Gürses *et al.*, 2002; Feng *et al.*, 2004). An EC reactor is composed of an electrolytic cell with a pair(s) of electrodes immersed in a liquid (wastewater) that serves as the electrolyte. The process of pollutant removal involves the application of an electric current to sacrificial electrodes which in turn leads to the dissolution of metal ions, such as iron or aluminum, from a sacrificial anode. Consequently, the metallic hydroxide, a coagulant formed by electrolytic oxidation in an aqueous phase, destabilizes colloidal suspension such as emulsified oil. Further, the destabilized colloids aggregate and form flocs (Bukhari, 2007). Given aluminum as an anode, the electrochemical reactions can be described as follows:



Finally, these colloid-adsorbed flocs can be removed by sedimentation or by floatation with H_2 bubbles produced at the cathode. In summary, the EC process involves three main mechanisms for pollutant removal, i.e., electrode oxidation, gas bubble generation, and floatation and sedimentation of formed flocs (Emamjomeh and Sivakumar, 2009).

EC not only provides a fast rate of pollutant removal and simplicity of operation; but no chemical additive is required, therefore, would produce less amount of sludge (Tezcan Ün *et al.*, 2006; Mouedhen *et al.*, 2008; Linares-Hernández *et al.*, 2008). These beneficial properties render EC more favorable than conventional physicochemical treatment processes. Recently, EC has also been successfully applied to remove oil and COD from the oily wastewater that comes from restaurants and the mechanical and metallurgical industries (Xu and Zhu, 2004; Tir and Moulai-Mostefa, 2008).

In this study, EC using an aluminum anode and a graphite cathode was used for the treatment of oily wastewater from the biodiesel production processes. The target removal pollutants were COD, O&G, and suspended solids (SS). Three important electrochemical factors were tested: initial pH, applied voltage, and reaction time. Response surface methodology (RSM) was used to create a set of designed experiments to obtain optimal multifactor operating conditions for the EC process. RSM is considered to be an effective means to design experiments that help to reduce the number of experimental trials, to evaluate the relative significance of variables and their interactions, and to build models (Gürses *et al.*, 2002; Tir and Moulai-Mostefa,

2008). Finally, a full quadratic model for prediction of pollutant removal efficiency was developed and verified with experimental data.

1 Materials and methods

1.1 Test wastewater

The test wastewater was obtained from a small-scale, commercial biodiesel production plant that employs an alkali-catalyzed transesterification process. This plant uses frying oil waste and crude palm oil as feedstock. The major source of the wastewater was from the methyl ester-washing stages. Characteristics of the biodiesel wastewater and analytical methods are shown in Table 1. The biodiesel wastewater contained high contents of COD and O&G due to the contamination by oil feed stock, soap, methanol and glycerol. Methanol concentration was found to be 10667 mg/L, while total residual O&G was 6020 mg/L. Since the alkali catalysts were used in the transesterification process, pH of the wastewater was expectedly high at 8.9.

Table 1 Characteristics of the biodiesel wastewater

| Parameter | Value | Analytical method/Instrument |
|----------------------|-------|---|
| pH | 8.9 | pH meter (Schott, USA) |
| COD (mg/L) | 30980 | Closed reflux ^a |
| O&G (mg/L) | 6020 | Gravimetric (hexane extraction) method ^a |
| Glycerol (mg/L) | 1360 | HPLC ^b (Shimadzu, Japan) |
| Methanol (mg/L) | 10667 | GC/FID ^c (Shimadzu, Japan) |
| SS (mg/L) | 340 | Gravimetric method ^a |
| Conductivity (μS/cm) | 350 | Conductivity meter (Schott, USA) |

COD: chemical oxygen demand; O&G: oil and grease; SS: suspended solids.

^a According to the standard methods for examination of water and wastewater (APHA, 1995); ^b High performance liquid chromatography;

^c gas chromatography/flame ionization detector.

1.2 Experimental apparatus

Figure 1 shows a diagram of the experimental apparatus. EC experiments were conducted in a 1.2-L monopolar batch reactor. An aluminum anode and a graphite cathode with flat and rectangular shapes were placed in parallel and vertically with a separation distance of 1.5 cm. The electrode gap was kept constant in all experiments. The

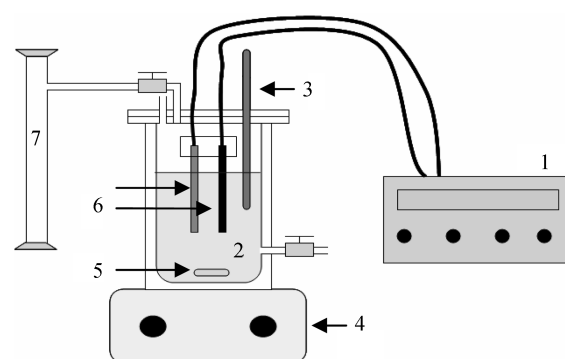


Fig. 1 Experimental apparatus. (1) DC power supply; (2) electrolyte; (3) thermometer; (4) stirrer plate; (5) magnetic bar; (6) electrodes; (7) gas collection equipment.

reaction surface area of each electrode was $10.5 \times 5.0 \text{ cm}^2$. The electrodes were connected to a digital DC power supply (0–60 V). The test volume of wastewater for each trial was 1 L. Duplicate runs were conducted for each testing condition to assure the reliability of experimentation. All experiments were conducted at the room temperature of 28°C . Gas production was measured by the water-replacement method. Sludge composition was characterized using a Fourier transform infrared spectrometer (FT-IR) (PerkinElmer, USA).

1.3 Experimental design

The Box-Behnken design, an experimental design for RSM, was used to create a set of designed experiments by MINITAB software (version 14). The Box-Behnken design was developed based on the combination of a two-level (full or fractional) factorial design with an incomplete block design. In general, a certain number of factors are put through all combinations for the factorial design in each block, while the other factors are kept at the central values. In this study, the Box-Behnken design for 3 factors, i.e., initial pH (x_1), applied voltage (x_2), and reaction time (x_3), involved three blocks. In each block, two factors were varied through the four possible combinations of high and low. The original factors of x_1 , x_2 , and x_3 were coded as given by Eq. (4):

$$X_i = \frac{x_i - x_{cp}}{\Delta x_i} \quad (4)$$

where, X_i is the coded level; x_{cp} is the original value of the centered point; and Δx_i is the value of variable change step. Table 2 shows the levels of original and coded factors using Box-Behnken design. The values of the original variables were selected based on the preliminary experimental results. Given the three main variables and three test levels, fifteen experiments were designated by MINITAB software.

To perform response surface regression analysis, experimental data were fitted to a full quadratic model, providing regression coefficients. The full quadratic model used in the response (Y_i) was described as the following:

$$Y_i = \beta_0 + \sum_{i=1}^4 \beta_i X_i + \sum_{i=1}^4 \beta_{ii} X_i^2 + \sum_{i=1}^3 \sum_{j=i+1}^4 \beta_{ij} X_{ij} \quad (5)$$

where, β_0 , β_i , β_{ii} and β_{ij} are the regression coefficients for intercept, linear, quadratic and interaction terms, respectively; and x_i , and x_{ij} are the independent variables. Y_i represents COD removal (Y_1), O&G removal (Y_2), and SS removal (Y_3).

Table 2 Original and coded factors

| Factor | Original factor (x) | Coded factor (X) | | |
|---------------------|-------------------------|----------------------|-----|----|
| | | −1 | 0 | 1 |
| Initial pH | x_1 | 4 | 6.5 | 9 |
| Applied voltage (V) | x_2 | 10 | 20 | 30 |
| Reaction time (min) | x_3 | 10 | 25 | 40 |

2 Results and discussion

2.1 Effects of operating parameters

2.1.1 Initial pH

It is known that initial pH and applied voltage (or current density) can influence the coagulant dosage rate and bubble generation rate, which in turn affect the pollutant removal efficiencies. To determine the effect of initial pH on the removal efficiencies of COD, O&G, and SS, the pH of biodiesel wastewater was adjusted with either H_2SO_4 or NaOH to 4, 6, and 9, respectively. In each batch experiment, the applied voltage and reaction time were kept constant at 20 V for 30 min. As shown in Fig. 2, the pollutant removal efficiencies increased as the initial pH decreased to the acidic level. At the pH value of 6, the highest removal efficiencies are achieved at 55.68% for COD, 97.83% for O&G, and 97.50% for SS. The residual COD is still high due to less significant removals of glycerol and methanol, which are the two main compositions of organic matters other than O&G in the biodiesel wastewater. The EC can remove glycerol and methanol by 3.53% and 16.86%, respectively. For removal mechanisms of O&G and SS, Gürses *et al.* (2002) contributed an increasing removal efficiency of colloidal particles in the pH range 4–7 to the formation of amorphous hydroxide precipitates and other aluminum hydroxo complexes with hydroxide ions and polymeric species. As pH increasing to 9, the decline of the removal efficiency is observed due to the less formation of the reactive flocs of aluminium hydroxide (Tir and Moulai-Mostefa, 2008). In the basic range, aluminium hydroxide ions may form negatively-charged ions such as $\text{Al}(\text{OH})_4^-$ and $\text{Al}(\text{OH})_5^-$, which allow less effective flocculation. Since hydroxide ions are produced along with H_2 bubbles at the cathode (Reaction (2)), it contributes to increase the final pH of the treated wastewater.

2.1.2 Applied voltage

The effect of applied voltage to the electrocoagulation cell was investigated by varying the voltage level from 10 to 30 V, which was equivalent to the measured current density range of 6.7–20.8 mA/cm^2 . Each experimental trial kept the initial pH at 6 and reaction time of 30 min.

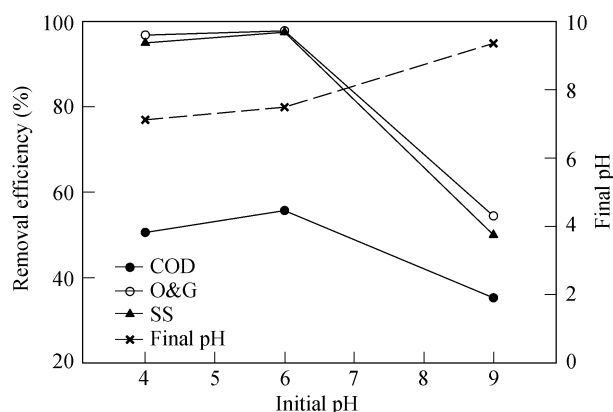


Fig. 2 Effect of initial pH on the removal efficiencies for COD, O&G, and SS. Applied voltage: 20 V; reaction time: 30 min.

Figure 3 shows variations of the removal efficiencies for COD, O&G, and SS as a function of applied voltage. A significant increase of the removal efficiencies could be observed as the applied voltage rose from 10 to 20 V. In the voltage range of 20–30 V, there is a tendency for a slight increase in removal. According to Faraday's law, the amount of coagulant or dissolved anodic metal is theoretically and directly proportional to the applied current to an electrolytic cell at a certain time. Thus, increasing applied voltage (or current density) results in an increasing amount of aluminum hydroxide flocs (Reaction (3)) for the removal of colloidal particles. At the same time, the generation rate of hydroxide ion is enhanced as well, resulting in a pH rise. The conductivity decreases from 480 at the beginning to 400–450 $\mu\text{S}/\text{cm}$ at the end of the experiments. As the applied voltage increases over 20 V, the final pH rose greater than 7.5. As shown previously, the basic condition is ineffective at removing colloidal particles.

2.1.3 Reaction time

Figure 4 shows the effect of reaction time on the pollutant removal efficiencies when it was varied from 10 to 40 min. The initial pH of 6 and applied voltage of 20 V were kept constant for all trials. All pollutant removal efficiencies increase with respect to the reaction time.

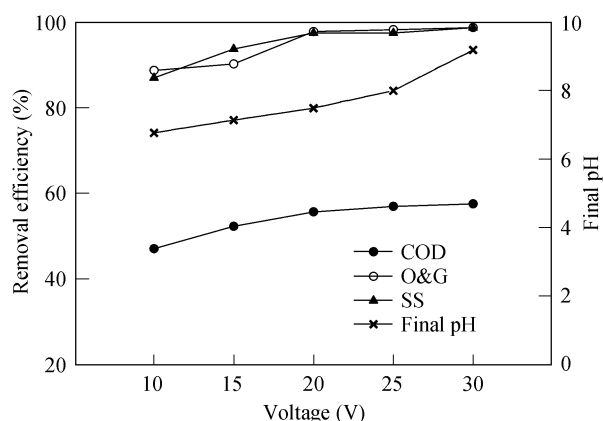


Fig. 3 Effect of applied voltage on the removal efficiencies for COD, O&G, and SS. Initial pH: 6; reaction time: 30 min.

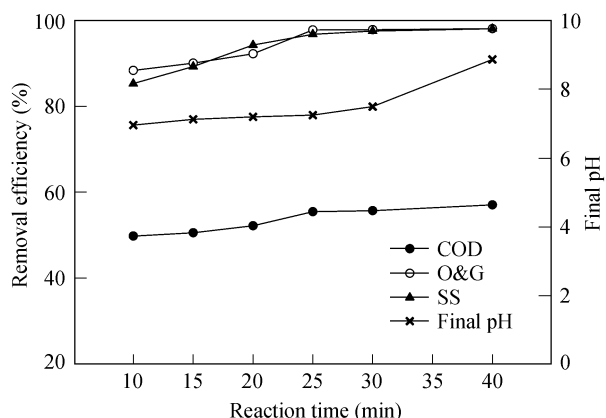


Fig. 4 Effect of reaction time on the removal efficiencies for COD, O&G, and SS. Initial pH: 6; applied voltage: 20 V.

However, the allowed reaction time longer than 25 min does not enhance the removal efficiencies. Our results are similar to other EC studies. Xu and Zhu (2002) and Tir and Moulai-Mostefa (2008) indicated that the optimal reaction time for treatment of oily wastewater is approximately 20 min. A final pH rise according to the reaction time is also observed due to the greater formation of hydroxide ion at the cathode. Additionally, the gas production is approximately 0.3 liter per liter of wastewater being treated.

2.2 Box-Behnken design experiments

Table 3 presents the Box-Behnken design with initial pH, applied voltage, and reaction time for fifteen experimental trials. To develop a response surface regression model, a full quadratic model (Eq. (5)) was applied to experimental observations of the removal efficiencies of COD (Y_1), O&G (Y_2), and SS (Y_3). Calibrating with the experimental results derived from the one factor at a time experiment, the quadratic regression models are given by Eqs. (6)–(8) for Y_1 , Y_2 , and Y_3 , respectively:

$$Y_1 = -14.0024 + 14.2979X_1 + 1.0149X_2 + 1.0688X_3 - 1.4142X_1^2 - 0.0207X_2^2 - 0.0107X_3^2 + 0.0866X_1X_2 - 0.0061X_1X_3 - 0.0122X_2X_3 \quad (6)$$

$$Y_2 = -105.595 + 35.345X_1 + 4.45X_2 + 3.99X_3 - 3.827X_1^2 - 0.071X_2^2 - 0.045X_3^2 + 0.185X_1X_2 + 0.071X_1X_3 - 0.064X_2X_3 \quad (7)$$

$$Y_3 = -89.854 + 33.7184X_1 + 4.2428X_2 + 3.3823X_3 - 3.7322X_1^2 - 0.0702X_2^2 - 0.03721X_3^2 + 0.1994X_1X_2 + 0.0803X_1X_3 - 0.0616X_2X_3 \quad (8)$$

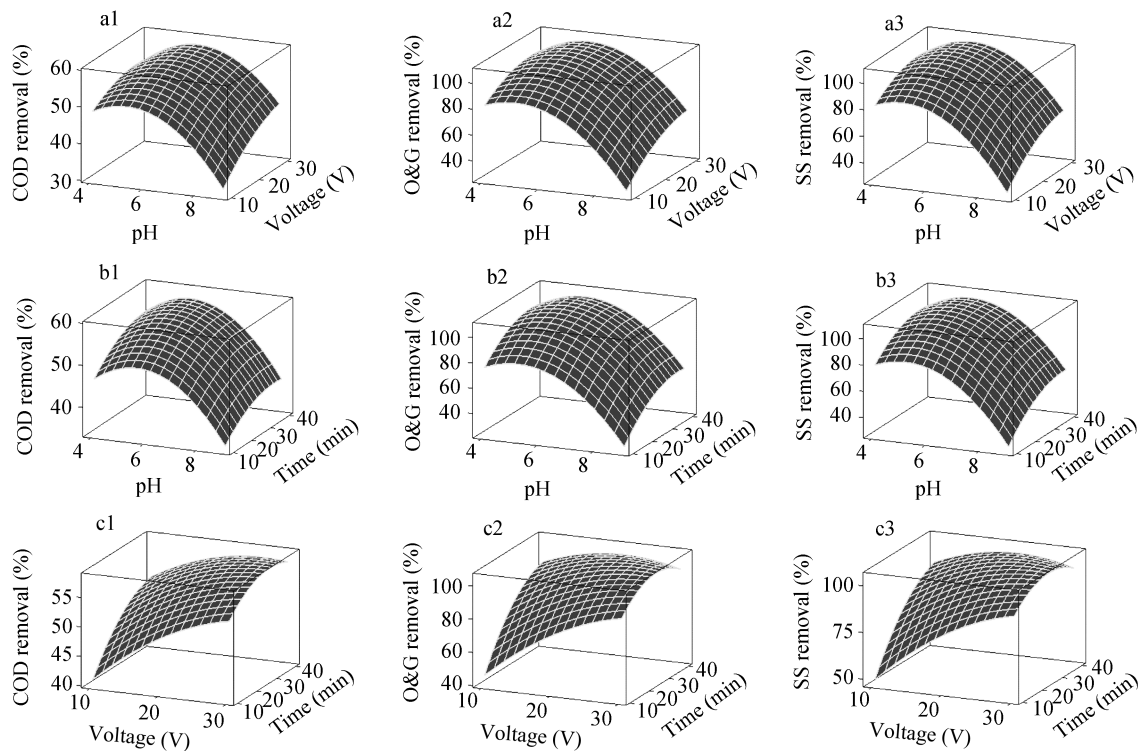
To verify the models, the set of experiments shown in Table 3 were conducted for fifteen runs. Table 3 shows that there was no observable difference in modeled and observed values. Furthermore, the influence of each independent factor on the models was tested for its level of statistical significance by analyzing the variance. The tested terms were liner terms (x_1 , x_2 , and x_3), square terms (x_1^2 , x_2^2 , and x_3^2), and interaction terms (x_1x_2 , x_2x_3 , and x_1x_3). These results show that the effects of all terms on COD, O&G, and SS removals are statistical significant at the 5% level.

Figure 5 shows the response surface plots for the variations of COD, O&G, and SS removal efficiencies according to initial pH, voltage, and reaction time. In each plot, two factors are varied, while the rest is kept constant. The plots are derived from the quadratic models of Eqs. (6)–(8).

It is obviously seen that the effects of pH, voltage, and reaction time on removals of COD, O&G, and SS exhibit the same tendency. The surface response plots offer the maximum removal efficiencies of 55.01% for COD, 97.77% for O&G, and 97.45% for SS at the optimum conditions for pH of 6.06, applied voltage of 18.2 V, and reaction time of 23.54 min (Table 4). As compared with the results obtained from one factor at a time experiments, the

Table 3 Box-Behnken design with three factors and its measured and predicted values

| Trial. | pH (x_1) | Voltage (x_2) (V) | Time (x_3) (min) | COD removal (%) | | O&G removal (%) | | SS removal (%) | |
|--------|-----------------|--------------------------|-------------------------|-----------------|-----------|-----------------|-----------|----------------|-----------|
| | | | | Experimental | Predicted | Experimental | Predicted | Experimental | Predicted |
| 1 | 6.5 | 30 | 40 | 57.81 | 57.29 | 98.92 | 97.63 | 98.86 | 97.35 |
| 2 | 6.5 | 30 | 10 | 53.65 | 53.45 | 90.03 | 89.19 | 92.05 | 91.46 |
| 3 | 4.0 | 20 | 40 | 54.20 | 54.40 | 96.01 | 97.87 | 96.88 | 97.37 |
| 4 | 4.0 | 10 | 25 | 48.81 | 48.48 | 83.64 | 82.16 | 83.75 | 82.63 |
| 5 | 4.0 | 30 | 25 | 52.59 | 53.04 | 95.85 | 97.32 | 95.63 | 96.47 |
| 6 | 6.5 | 20 | 25 | 55.21 | 55.40 | 98.34 | 98.43 | 98.30 | 97.88 |
| 7 | 9.0 | 10 | 25 | 32.03 | 31.61 | 29.07 | 28.25 | 29.55 | 28.63 |
| 8 | 9.0 | 30 | 25 | 44.46 | 44.84 | 59.80 | 61.91 | 61.36 | 62.42 |
| 9 | 6.5 | 10 | 10 | 40.32 | 40.89 | 43.36 | 45.58 | 47.73 | 49.16 |
| 10 | 9.0 | 20 | 40 | 41.15 | 41.41 | 57.31 | 58.54 | 59.09 | 59.37 |
| 11 | 6.5 | 20 | 25 | 55.73 | 55.40 | 97.67 | 98.43 | 97.16 | 97.88 |
| 12 | 4.0 | 20 | 10 | 46.66 | 46.44 | 75.91 | 75.55 | 79.38 | 79.03 |
| 13 | 9.0 | 20 | 10 | 34.51 | 34.36 | 26.58 | 25.57 | 29.55 | 28.98 |
| 14 | 6.5 | 10 | 40 | 51.82 | 52.06 | 90.86 | 92.42 | 91.48 | 92.01 |
| 15 | 6.5 | 20 | 25 | 55.21 | 55.40 | 98.92 | 97.63 | 98.30 | 97.88 |

**Fig. 5** Effects of pH, voltage, and reaction time on COD, O&G, and SS removal. (a) The reaction time was kept constant at 25 min, (b) the applied voltage was kept constant at 20 V, and (c) the initial pH was kept constant at 6.5.

optimum operating conditions are pH 6, applied voltage of 20 V, and reaction time of 25 min. This implies that the quadratic regression models reasonably optimize the operating conditions and predict the EC process efficiency for the biodiesel wastewater treatment.

2.3 Energy consumption

Typical costs in wastewater treatment with the EC process are the expenditure on energy consumption and mass loss of electrodes. Chemical addition is only used for the purpose of pH adjustment. Under the optimum conditions, the EC treatment for 1 m³ of biodiesel wastewater requires the power consumption of 5.57 kWh, which is close to that obtained from other studies (Sanchez-Calvo *et al.*, 2003;

Table 4 Comparison of optimum conditions and removal efficiencies obtained from one factor at a time experiments and the Box-Behnken design optimization

| | One factor at a time experiments | Box-Behnken design optimization |
|---------------------|-------------------------------------|------------------------------------|
| Optimum condition | | |
| pH | 6 | 6.06 |
| Voltage (V) | 20 | 18.2 |
| Reaction time (min) | 25 | 23.54 |
| COD removal (%) | 55.43 | 55.01 |
| O&G removal (%) | 96.75 | 97.45 |
| SS removal (%) | 97.76 | 97.77 |

Tir and Moulai-Mostefa, 2008). Tir and Moulai-Mostefa (2008) reported the power consumption for treating the

oily wastewater with the EC process, ranging from 0.115 to 11.38 kWh/m³. The mass loss of an aluminum electrode for a liter of biodiesel wastewater being treated is 0.13 g.

2.4 Sludge characteristics

Under the optimum conditions, the sludge mass production rate was found to be 1.18 g/L of the treated wastewater (on a dry basis). The characterization of the EC-generated sludge was performed with FT-IR, and spectra show that esters are the dominant functional groups of the sludge composition. They include methyl linoleate, ethyl palmitate, ethyl myristate, butyl stearate, and methyl elaidate. These ester compounds were possibly left from the transesterification of glyceride with alcohol to ester.

3 Conclusions

This study demonstrates that the EC process using an aluminum anode and graphite cathode is effective in reducing O&G and SS by more than 95% in biodiesel-processing wastewater. However, the COD removal is achieved by 55% due to less significant removal of glycerol and methanol. Therefore, the EC process is possibly suitable for a primary treatment for biodiesel wastewater. It still requires a further biological treatment process. Chavalparit and Meanseta (2006) demonstrated that pretreatment of oily wastewater from a crude palm oil mill with EC processing can enhance the biodegradability of oil emulsions in the wastewater. The pretreatment with EC followed by a biological treatment process seems feasible and competitive compared with evaporation or pure physicochemical treatments. It requires less energy consumption, short process time, no chemical addition, and less sludge production.

Using the Box-Behnken design to create a set of experimental runs can reduce the number of runs needed to optimize the operating conditions in comparison with the one factor at a time experiment method. Box-Behnken provides sufficient data to fit the quadratic models for pollutant removals. The optimization of the models provides the optimum conditions at an initial pH of 6.06, 18.2 V of applied voltage, and 23.5 min of reaction time. These optimum conditions are in excellent agreement with those obtained by the one factor at a time experiments. Therefore, RSM could be effectively adopted to optimize the operating multifactors in complex EC processes.

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