Polishing of treated palm oil mill effluent (POME) from ponding system by electrocoagulation process

Mohammed J. K. Bashir, Tham Mau Han, Lim Jun Wei, Ng Choon Aun and Salem S. Abu Amr

ABSTRACT

As the ponding system used to treat palm oil mill effluent (POME) frequently fails to satisfy the discharge standard in Malaysia, the present study aimed to resolve this problem using an optimized electrocoagulation process. Thus, a central composite design (CCD) module in response surface methodology was employed to optimize the interactions of process variables, namely current density, contact time and initial pH targeted on maximum removal of chemical oxygen demand (COD), colour and turbidity with satisfactory pH of discharge POME. The batch study was initially designed by CCD and statistical models of responses were subsequently derived to indicate the significant terms of interactive process variables. All models were verified by analysis of variance showing model significances with Prob > F < 0.01. The optimum performance was obtained at the current density of 56 mA/cm^2, contact time of 65 min and initial pH of 4.5, rendering complete removal of colour and turbidity with COD removal of 75.4%. The pH of post-treated POME of 7.6 was achieved, which is suitable for direct discharge. These predicted outputs were subsequently confirmed by insignificant standard deviation readings between predicted and actual values. This optimum condition also permitted the simultaneous removal of NH_3-N, and various metal ions, signifying the superiority of the electrocoagulation process optimized by CCD.

Key words | central composite design, electrocoagulation, optimization, POME, post treatment

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INTRODUCTION

Elaeis guineensis, commonly recognized as oil palm, is a versatile monoecious oil bearing plant originates from West and Central Africa. Its distribution is typically restricted to the tropical regions of the world, e.g. Malaysia and Indonesia (UNEP & GEAS 2011). In Malaysia, oil palm plantations cover approximately three million hectares of the cultivated area, an increase of almost 70 times since the 1960s (Tabassum *et al.* 2015). The extraction of palm oil has generated various types of waste by-products, namely solid waste such as oil palm shell, empty fruit bunches, fibres, palm oil clinker and palm oil fuel ash, accompanied by inevitable liquid waste production, i.e. palm oil mill effluent (POME). Alas, POME inherits high strength wastewater characteristics (high chemical oxygen demand (COD) and biochemical oxygen demand (BOD) concentrations). The fresh POME is usually hot, viscous, dark brown in colour, and acidic with pH between 4.0 and 5.0 (Chan *et al.* 2015). It is estimated that 1.5 m^3 of water is needed to process a ton of fresh fruit bunch and half of this amount becomes POME (Mohammed *et al.* 2014). In this regard, 60 million tons of POME was generated in Malaysia from 241 palm oil mills in the year 2010 and can engender a serious environmental menace if the untreated POME is indiscriminately discharged into the natural water bodies (Othman *et al.* 2014). As cost is of concern, virtually all palm oil mills in Malaysia adopt a ponding system in treating POME. Nonetheless, the bottlenecks of this method are a large treatment area and long retention time requirements since the setup usually consists of a de-oiling tank, acidification, anaerobic and facultative ponds with hydraulic retention times of 1, 4, 45, and 16 days, respectively (Chan *et al.* 2015). In the interim, the treatment of POME is very critical to conserve the environment due to the perpetual emissions of biogases from the ponding system which insidiously fortify the impact of global warming, besides failing to comply to the discharge standard most, if not all, of the time.

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Owing to the limitations experienced by the ponding system, researchers had rendered enormous efforts to offer an economical, practical and efficient alternative in treating POME (Saeed *et al.* 2015; Taha & Ibrahim 2014; Bala *et al.* 2015; Cheng *et al.* 2015; Parthasarathy *et al.* 2016). Typically, the oxidation process has been exploited as a part of an integrating system with other wastewater treatment methods primarily to synergistically enhance the treatment efficiencies when dealing with wastewaters loaded with refractory organics (Li *et al.* 2010; Saeed *et al.* 2014). Nevertheless, one promising technique for treating hard-to-treat wastewater streams is based on electrochemical technologies. Electrochemical processes (electrolysis and electrocoagulation) have been effectively used for removing pollutants in various industrial wastewaters (Kobya *et al.* 2003; Bashir *et al.* 2013). Ideally, the electrocoagulation process is an effective wastewater treatment technique that is capable of completely mineralizing a wide range of organic substances. According to Kobya *et al.* (2003), removal mechanisms in the electrocoagulation process include coagulation, precipitation and flotation. Electrocoagulation utilizes aluminium or iron anodes to produce aluminium or iron hydroxide flocs by reaction at the anodes followed by hydrolysis.

As such, this study emphasized employing the electrocoagulation process using aluminium electrodes for further polishing of biologically treated POME using a ponding system in order to satisfy the discharge limit decreed by Environmental Quality Regulations (2000). This auxiliary post-treatment via the electrocoagulation process application was statistically optimized using a central composite design (CCD) module in a response surface methodology (RSM) programme. Instead of introducing various electrolytes to spur the electrocoagulation process, the pH of POME collected from the ponding system was fine-tuned simultaneously with the current density and contact time, targeting to achieve maximum treatment efficiencies of COD, colour and turbidity, besides arriving at a satisfactory pH prior to the discharge.

MATERIALS AND METHODS

Characteristics of treated POME by ponding system

The POME used in this study was originally from Tian Siang Oil Mill (Air Kuning) Sdn. Bhd. located in the District of Batang Padang, Perak, Malaysia. The POME samples were collected from the last treatment pond of a ponding system that would be eventually siphoned into the vicinity water bodies. The collected samples were transported to the environmental laboratory and stored at 4 °C in order to minimize the biological and chemical changes. The characteristics of partially treated POME from a ponding system were analysed based on *Standard Methods for the Examination of Water and Wastewater* (APHA 2012) and presented in Table 1.

Setup of the laboratory-scale electrocoagulation system

Batch experiments were conducted for all studies using beakers containing 500 mL of characterized POME each. A pair of aluminium electrode plates were used as an anode and cathode in electrocoagulation system with a dimension of 12×4.5 cm. Exactly half of the electrode plates (6 cm) were vertically submerged into the POME solution giving the contact surface area of 27 cm^2 each. The parallel electrode plates were positioned at the centre of every beaker and separated with 3 cm distance giving way to the 300 rpm stirring impact to spur the reaction between electrodes. The electrodes were replaced when >10% of electrode material was lost. The current was supplied by laboratory DC power with a constant voltage set at 12 V. When every batch experiment conclusion had reached, the concentrations of COD, colour and turbidity were analysed and their respective removal efficiencies were calculated using Equation (1):

$$\text{Removal efficiency } (\%) = \left(\frac{C_i - C_f}{C_i}\right) \times 100\% \quad (1)$$

where C_i and C_f refer to the initial and final concentrations of COD, colour and turbidity.

Experimental design and statistical analysis

In this study, the Design-Expert® Version 7.0 (Stat-Ease, Inc., Minneapolis, MN, USA) software was employed to

Table 1 | Characteristics of partially treated POME from ponding system

Parameter	Unit	Average value
COD	mg/L	2030.0
NH_3-N	mg/L	100.0
Colour	PtCo	3910.0
Turbidity	NTU	809
BOD_5	mg/L	7.0
Suspended solids	mg/L	511.0
pH	–	8.5
Temperature	°C	25.0

provide a platform for RSM to analyse and optimize the treatment of POME from ponding system. The most widely exploited module of RSM, i.e. CCD, was used to evaluate the correlation between the process variable and the response. Guven *et al.* (2008) substantiated that CCD is an effective design tool for sequential experimentation as it permits reasonable volume of information to test the lack of fit when a sufficient number of experimental values is present. In this study, the three significant process variables that to be ultimately optimized were current density (A), contact time (B) and pH (C) with each process variable numerically varied from -1 to $+1$ coded value. The respective actual value ranges were 10–60 mA/cm^2, 10–90 min and 3.0–5.0, all determined from preliminary experiments and supported by the literature (Bashir *et al.* 2009, 2013; Mohajeri *et al.* 2010). The design by CCD had culminated in a total of 20 batch experiments, inclusive of six repeated batches, that occurred at the intermediate point of every process variable to assess the random error (Table 2). The monitored responses were the simultaneous percentage removals of COD, colour and turbidity together with pH at the end of each run. Subsequently, every response was statistically modelled using a quadratic model via correlation with process variable terms as demonstrated in Equation (2):

$$Y = \beta_0 + \sum_{j=1}^{k} \beta_j X_j + \sum_{j=1}^{k} \beta_{jj} X^2{}_j + \sum_{i} \sum_{<j=2}^{k} \beta_{ij} X_i X_j + e_i \qquad (2)$$

where Y is the response, x_i and x_j are the process variables, β_0 is the constant coefficient, β_i, β_{ii} and β_{ij} are the interaction coefficients of linear, quadratic and second-order terms, respectively, k is the number of process variables and ϵ is the random error component.

The quality of the fitted model was verified by the coefficient of determination (R^2) value and its statistical significance was attested by F-test from analysis of variance (ANOVA) based on the probability (P-value) of 95% confidence level (Bashir *et al.* 2009). The models that described their respective responses' interaction were then used to predict the optimum condition of process variables targeting on maximum removal efficiencies of COD, colour and turbidity in concert with a satisfactory pH of post-treated POME for discharge.

Table 2 | Batch experiments as designed by CCD and their respective response values

	Process variables			Responses			
Run	Current density (mA/cm^2)	Time (min)	pH	COD (%)	Colour (%)	Turbidity (%)	pH
1	10.0	90	5.0	61.5	93.8	97.3	8.0
2	35.0	70	4.0	70.7	96.4	99.8	6.7
3	10.0	10	5.0	32.4	51.3	12.2	6.1
4	47.5	50	4.0	75.2	97.2	99.8	7.0
5	10.0	10	3.0	36.8	54.2	26.9	4.2
6	60.0	10	5.0	58.4	67.6	15.1	5.9
7	60.0	90	5.0	66.6	95.4	92.9	7.9
8	35.0	50	4.0	66.1	97.1	99.9	7.4
9	35.0	50	4.0	66.2	97.3	99.9	7.4
10	35.0	50	4.0	66.5	97.3	99.9	7.4
11	10.0	90	3.0	67.8	96.1	97.5	7.4
12	35.0	50	4.0	66.7	97.3	99.9	7.4
13	60.0	10	3.0	67.6	83.2	83.2	5.2
14	35.0	30	4.0	71.1	88.6	68.5	6.9
15	35.0	50	4.0	66.3	97.2	99.9	7.4
16	35.0	50	4.0	66.3	97.4	99.9	7.4
17	35.0	50	3.5	74.3	97.5	99.9	7.3
18	22.5	50	4.0	70.9	96.6	99.1	6.9
19	60.0	90	3.0	7.42	98.1	98.9	5.9
20	35.0	50	4.5	72.3	96.8	99.5	7.2

RESULTS AND DISCUSSION

As a matter of fact, the performance of a ponding system in treating POME is unveiled in Table 1. The POME released to the surrounding water sources was still bearing high concentrations of COD, colour, turbidity, suspended solids and ammonia–nitrogen, violating the discharge limits set by the Environmental Quality Regulations (2000) of Malaysia. From this viewpoint, post-treatment of POME from a ponding system is seriously entailed to not only satisfy the decree, but also to protect the health of environment receiving the discharge POME.

Statistical significance of responses' models

The application of the electrocoagulation process as a post treatment technique of POME was optimized by CCD of RSM in this study. The response results of batch experiments designed by CCD via statistically varying process variables are shown in Table 2. The CCD module can offer an empirical design in relating response to process variable based on the estimation of the parameter (Bashir *et al.* 2009). By applying the factorial regression analysis on the

experimental data shown in Table 2, responses and process variables could be related by polynomial equations. The quadratic models acquired for every response upon omitting the statistically insignificant model terms are presented in Equations (3)–(6):

$$\text{COD removal } (\%) = 33.12 + 0.61A + 1.17B - 3.50C - 0.01AB - 0.01B^2 \quad (3)$$

$$\text{Colour removal } (\%) = 76.90 - 0.45A + 2.37B - 11.99C + 0.25AC - 0.16BC - 0.02B^2 \quad (4)$$

$$\text{Turbidity removal}(\%) = 33.98 + 1.84A + 2.12B - 12.23C - 0.01AB - 0.30AC + 0.24BC - 0.02B^2 \quad (5)$$

$$\text{Treated pH solution} = 1.77 + 0.01A + 0.10B + 0.61C \quad (6)$$

A, B and C are the model terms that represent the process variables of current density, contact time and pH, respectively. Table 3 elucidates the ANOVA analysis of four response quadratic models.

In all cases, the adequacies and significances of response quadratic models were confirmed as pointed out by the Prob > F <0.01. The coefficient of determination (R^2) for all models indicated satisfactory adjustment of the quadratic models to the experimental data. Myers *et al.* (2009) suggested that a good fit model should have R^2> 0.8, as manifested in this study. According to Myers *et al.* (2009), the R^2 value of closer to 1.0 is statistically desirable. Next, the coefficients of variance of all models were below 10%, demonstrating good reproducibility of models (Bashir *et al.* 2010). Adequate precisions of >4.0 were observed for all models, which indicate that adequate signals for the models can be used to steer the design space (Azmi *et al.* 2015). Accordingly, the derived models were adequate to navigate through the design space defined by CCD and able to predict the responses (Mohajeri *et al.* 2010). On another note, the dispersion of data values around their respective means were highlighted by the calculated standard deviation values of all below 10%, a typically acceptable variation. In this regard, the actual values of responses could be viewed scattering around their respective predicted responses by the derived models, as shown in Figure 1, evidencing the variations were insignificant for all cases.

Table 3 | ANOVA results for validating response surface quadratic models

	Response			
ANOVA parameter	**COD**	**Colour**	**Turbidity**	**pH**
Model				
F-value	36.7	13.9	65.0	17.6
P-value (Prob > *F*)	<0.01	<0.01	<0.01	<0.01
Model lack-of-fit				
F-value	2.41	212.2	13.6	1767.5
P-value (Prob > *F*)	0.173	<0.01	0.01	<0.01
Model R^2	0.9291	0.8652	0.9743	0.8629
Model coefficient of variance	5.4	10	7.1	6.0
Model adequate precision	22	12	26	16
Mean (%)	65	86	84	6.9*
Standard deviation (%)	3.5	8.9	5.9	0.4*

*pH has no unit for mean and standard deviation.

Impact of process variable interactions on responses

The fitted models (Equations (3)–(6)) were subsequently used to access the interactions of process variables (namely current density, contact time and pH) impacting on removal of colour, turbidity and COD together with the consequential pH of post-treated POME by the electrocoagulation process. The conspicuous interaction of process variables leading to a synergistic effect can be viewed in a 3D surface plot shown by steep curvature and vice versa. Figure 2 in this study presents the respective statistically predicted responses of treated POME via the electrocoagulation process resulted from the interactions among the process variables. The removal of colour, turbidity and COD generally increased with the reduction of initial pH from 5.0 to 3.0. Among these, the removal of colour (Figure 2) exhibited that the largest changes occurred at minimum current density and maximum contact time, i.e. from 40% at pH 5.0 to 63% at pH 4.0 and further increased to 88% at pH 3.0. Likewise, Wu *et al.* (2015) found that the total organic carbon (TOC) removal was higher in acidic solution than in neutral or basic solutions in the electrocoagulation process.

On account of current density and contact time, progressive removal of colour and turbidity could be observed at the increase of current density, peaking at the contact time between 50 and 70 min for all interaction cases. In this region, virtually complete removal of colour and turbidity could be identified within the studied pH range as shown in Figure 2, respectively. Focusing on COD removal, the

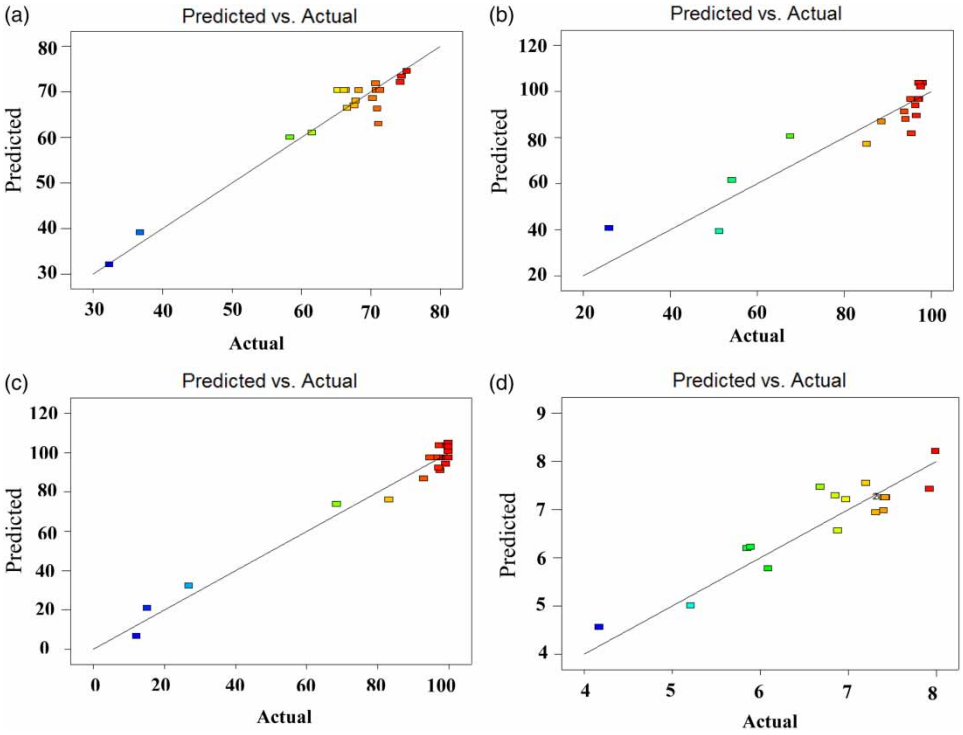

Figure 1 | Predicted versus actual values plots: (a) COD, (b) colour, (c) turbidity, (d) pH.

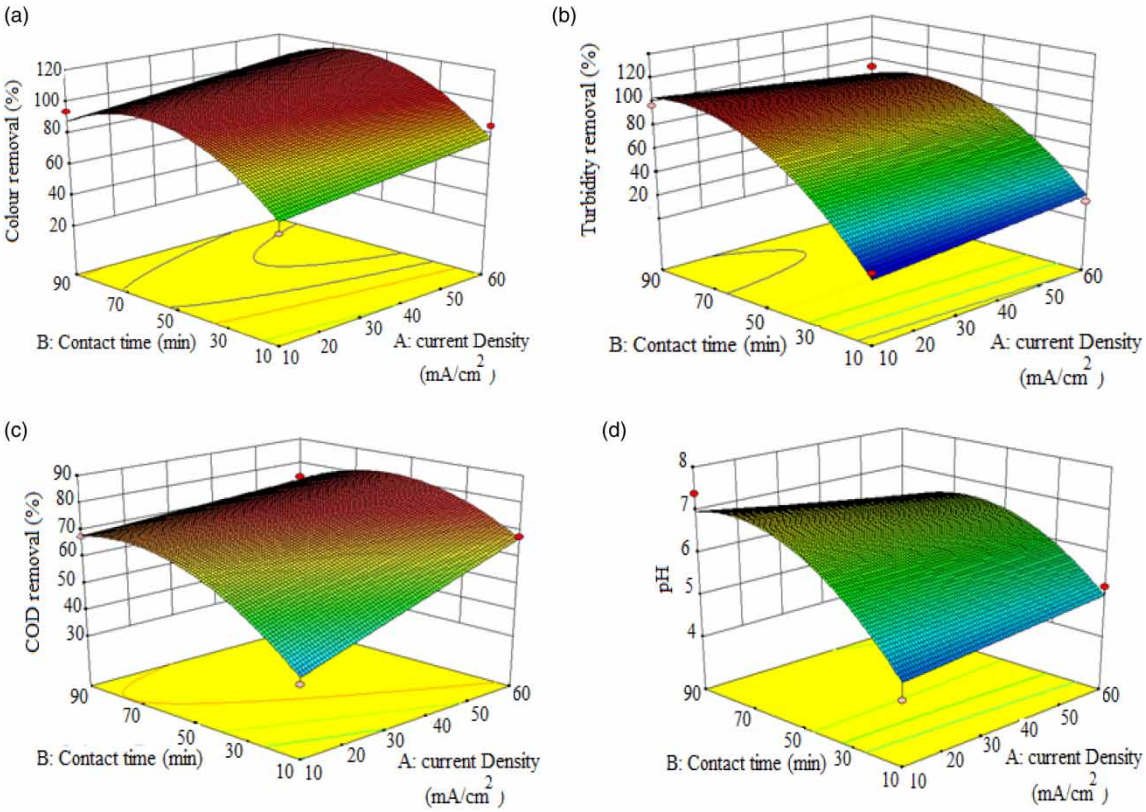

Figure 2 | 3D surface plots for colour (a), turbidity (b) and COD (c) removal efficiencies, and pH (d).

favourable effect of low pH was seen intensifying noticeably with the increasing of current density at the contact time of between 50 and 70 min, enhancing the removal of COD via the electrocoagulation process to the tune of about 80% (Figure 2(c)).

In the electrocoagulation process, aluminium electrode as a sacrificial anode was dissolved to produce Al^{3+} upon the application of an electrical current. The metal ions produced are hydrolyzed in the electrochemical cell to produce metal hydroxide ions. Equations (7)–(10) present the chemical reactions at the anode and cathode (Kobya *et al.* 2003; Xiangdong *et al.* 2011).

At anode:

$$Al \rightarrow Al^{3+} + 3e^{-} \quad (7)$$

$$2Al + 6H_2O + 2OH^{-} \rightarrow 2Al(OH)^{4-} + 3H_2 \quad (8)$$

At cathode:

$$3H_2O + 3e \rightarrow 3/2H_2 + 3OH^{-} \quad (9)$$

Overall reaction:

$$2Al + 6H_2O \rightarrow 2Al(OH) + 3H_2 \quad (10)$$

As indicated in the previous reactions, the flocks are formed as metal hydroxides in the electrocoagulation process. Considering the environment, almost neutral post-treated POME by electrocoagulation process was plausibly produced by prolonging the contact time, particularly at the initial pH range between 3.0 and 4.0 (Figure 2(d)). This was due to the continuous release of hydroxide ion (OH^{-}) by cathode into the POME solution as shown by Equation (9) when the electrocoagulation process advances with time, elevating the initial pH of solution (Bashir *et al.* 2009).

Optimization of process variable interactions in electrocoagulation process

The process variable interactions as discussed in the previous section were served as an insight in narrowing down the ranges of process variables whilst finding the optimum condition for the process to take place. As such, the contact time and pH programmed in CCD of RSM were limited within 50–70 min and pH 3.0–4.0, respectively. The current density, on the other hand, was goaled at minimum in providing economically feasible post-treatment of POME without having to compromise the practicability of the electrocoagulation process. In the case of responses, the colour, turbidity and COD removal were targeted at maximum with the pH of post-treated POME set within the range of pH 6.0–8.0 to fulfil the discharge standard and, at the same time, defuse the deleterious impact on the environment upon the POME discharge. The optimum condition was found at the current density of 56 mA/cm^2, contact time of 65 min and pH of 4.5. This optimum condition would impart complete removal of colour and turbidity with COD removal of 75.4%. The pH of post-treated POME was predicted as pH 7.6 which is suitable for discharge to the natural environment (Environmental Quality Regulations 2000). To validate the accuracy of predicted outputs, the electrocoagulation process, tweaked to achieve optimum condition, was implemented and the actual results are shown in Table 4. The standard deviations calculated between predicted and actual values for each response were subsequently found to be negligible, signifying the reliability of the CCD tool in optimizing the electrocoagulation process.

Researchers had laboriously contributed to the literature vis-à-vis the use of the electrochemical process for POME treatments. Vijayaraghavan & Kamala Nalini (2012) reported that with initial COD concentration of 15 g/L, current density of 37.6 mA/cm^2, sodium chloride as an add on electrolyte of 3% and contact time of 4 h had resulted in 82% of COD removal with residuals BOD_5 1265 mg/L, TOC 1024 mg/L, chlorine 86 mg/L and temperature 61 °C. Extended treatment of POME in their study had overall increased the post-treated solution temperature which was unsuitable for immediate discharge, particularly in bulk quantity. In the Fenton oxidation process, Saeed *et al.* (2014) confirmed that 92.1% of COD and 85.1% of colour were removed with the final solution pH attained at 2.9 amidst the optimum POME treatment condition, namely 4.57 g/L of H_2O_2, 1.88 g/L of Fe^{2+} ions, 30 min of contact time, 120 rpm of agitation speed and pH range within

Table 4 | Comparison of predicted optimized and laboratory determined responses

Response	Predicted value	Actual value	Standard deviation
COD (%)	75.4	69.6	4.10
Colour (%)	100	97.8	1.56
Turbidity (%)	100	99.6	0.28
pH	7.59	7.48	0.08

3.0–5.0. The very acidic POME solution after the treatment required additional conditioning process prior to the discharge to avert untoward occurrences arising from surrounding natural biochemical processes. Also, the use of the electro-Fenton process to enhance the later bioremediation of POME had been documented by Ramesh Babu *et al.* (2010). In their study, 48.4% of 6,700 mg/L COD concentration was successfully oxidized via the electro-Fenton process before being treated by a biological oxidation process which removed 86.1% of residual COD concentration in POME, as well as 85.2% of BOD. The superiority of their fabricated, combined treatment process was as well capable of removing high concentrations of TOC and total nitrogen at the same time. By using the optimum condition of the electrocoagulation process determined in this study, other pollutants were as effectively removed from POME as shown in Table 5.

Ammonia–nitrogen concentration was noted flopping from 100 to 65 mg/L in the POME after the treatment; a removal efficiency of approximately 35% was successfully achieved in this case. High removal efficiency of ammonia removal was also reported by Liu *et al.* (2009) in the electrolytic degradation using Ti/IrO_2 anode. Li & Liu (2009) found that the ammonia was transformed into nitrogen gas in their mechanism study involving electro-oxidation with RuO_2/Ti anode. On a different note, among the pollutants, suspended solids encompassed the highest removal efficiency, virtually 100%; preponderantly due to the complete removal of turbidity when the optimum condition of the electrocoagulation process was applied. Metal ions presented in POME also could be neutralized and deposited by the cathode via reduction during the electrocoagulation process.

In order to examine the released amount of aluminium from electrodes during the electrocoagulation process, the concentration of aluminium before and after treatment were examined as presented in Table 5. The slight increase in aluminium concentration from 32.3 to 32.9 mg/L was caused by the ionization of anode aluminium electrode. Upon the application of a direct current, electrocoagulation involves the generation of coagulants *in situ* by dissolving sacrificial anodes, as presented in Equation (7) (Xiangdong *et al.* 2011).

Table 5 | Other responding parameters to the optimized process variables of electrocoagulation system

Parameter	Initial concentration (mg/L)	Final concentration (mg/L)	Removal efficiency* (%)
NH_3-N	100	64.5	35.4
Suspended solids	511	3.50	99.4
Aluminium	32.3	32.9	−1.77
Chromium	0.51	0.11	77.8
Iron	66.8	34.7	48.0
Nickel	0.85	0.14	83.3
Copper	0.33	0.18	45.6
Zinc	27.1	12.9	52.3

*Calculation of removal efficiency is based on Equation (1).

Operation cost analysis

Cost analysis of biologically treated POME via electrocoagulation was carried out. Energy consumption is the main concern of electrocoagulation treatment. In this study, the operation costs were estimated for an applied current density of 56 mA/cm^2 which is the current density for which the optimum energy consumption was obtained. Therefore, the energy consumption needed was calculated by Equation (11).

$$\text{Energy consumption (kWh/m}^3) = \text{current density} \times \text{anode surface area} \times \text{voltage} \times \text{time} \times 1000\text{L/sample size}$$
$$= 56 \times 27 \times 12 \times 10^{x-6} \times \frac{65}{60} \times \frac{1000}{0.5} = 39.3\,\text{kWh/m}^3 \quad (11)$$

The result indicated that the specific energy consumption needed at optimum conditions was 39.3 kWh/m^3. Prices of electricity are mainly dependent on the particular country. If the present unitary electricity cost in Malaysia, which is around 0.218 MYR ($kWhh^{-1}$), is considered then the energy cost by electrocoagulation would be 39.3 $kWh/m^3 \times 0.218$ (electricity cost per kWh in Malaysia ringget) = 8.56 MYR/m^3 which is equal to 1.9 $US\$/m^3$.

In addition to the cost of energy needed, a pair of aluminium electrode plates costing 0.3 MYR was purchased and used in this study.

CONCLUSIONS

The CCD of RSM was used to optimize the electrocoagulation process for post-treatment of POME from the ponding system. The interaction of process variables, namely current density, contact time and initial pH were simultaneously optimized. The responses targeted maximum removal of COD, colour and turbidity with a satisfactory pH of

discharge POME programmed in the CCD module. The derived response statistical models showing significant terms of interactive process variables were verified by ANOVA with each possessing Prob > F <0.01 and R^2 value of closer to 1.0. The optimum electrocoagulation process condition predicted 100% removal of colour and turbidity with COD removal of 75.4%. The pH of post-treated POME of 7.6 was achieved at optimum condition, suitable for direct discharge to the natural environment. Significant simultaneous removal of ammonia–nitrogen, suspended solids and various metal ions also materialized at the optimum condition indicating practicability of the electrocoagulation process optimized by CCD for post-treatment of POME. Upon these findings, the electrocoagulation process at a specified level may be used as a post-treatment technology that is effective and efficient before discharging to meet the requirements.

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