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Pretreatment of palm oil mill effluent by electrocoagulation and coagulation

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ABSTRACT: Palm oil mill effluent (POME) is wastewater generated from the palm oil milling process. It is regarded as a highly polluting wastewater as it has a high chemical oxygen demand (COD), and contains high levels of oil and suspended solids. Pretreatment of POME is desirable before a subsequent biological or other treatment. This paper reports a study of POME pretreatment by (1) electrocoagulation and (2) conventional coagulation with $Al_2(SO_4)_3$. At an optimal condition with a current density of 20 A/m^2 for 5 min at 313 K and pH 5, the percentage of oil, COD, suspended solids, and total solids removal by electrocoagulation were 72%, 64%, 53%, and 43%, respectively. The electrical consumption under this condition was in the order of 0.10 kWh/m^3. The addition of $NaNO_3$ improved the electrocoagulation, and it may also be beneficial for a subsequent biological treatment. At high current densities and/or long electrocoagulation time, POME treatment produced bubbles, which decrease the efficiency of oil removal. This effect did not occur in conventional coagulation. However, chemical coagulation limits oil removal from POME due to the effect of pH. Overall, electrocoagulation should cause less environmental damage than conventional chemical coagulation by $Al_2(SO_4)_3$ as it treats POME without the need to add sulphate ions.

KEYWORDS: oil-in-water, aluminium sulphate, oil removal, electrochemistry

INTRODUCTION

Electrocoagulation is a technique used to treat wastewater to recover valuable chemicals. The main advantages of electrocoagulation over other conventional techniques, such as chemical coagulation or adsorption, are the delivery of reactive agents in situ without generating secondary pollution, and using compact equipment. Several studies have reported the potential of electrocoagulation in treating a variety of different pollutants in wastewater including the removal of suspended solids[1–3], dyes[4–6], heavy metals[7,8], breaking up oil-in-water emulsions[9,10], and the removal of complex organics[11,12] and bacteria, viruses, and cysts[13]. Particularly when prevention of secondary pollutants is desirable, electrocoagulation has been shown to be a suitable treatment method when biological treatments fail[14].

Palm oil mill effluent (POME) is a highly polluting wastewater with typically a biochemical oxygen demand of 25 g/l, a chemical oxygen demand of 50 g/l, oil and grease of 8 g/l, suspended solids of 20 g/l, and total solids of 40 g/l[15]. POME also contains macromolecules, such as polysaccharides, lipids, proteins, and a number of monocyclic and polymeric aromatic molecules[16]. Anaerobic digestion is often employed to convert carbon in POME to methane, which can then be further employed as a renewable energy source. However, oil is generally difficult to degrade biologically and the high content of oil in POME can cause sludge and granule flotation in an upflow bioreactor[17]. Removal of oil from POME before anaerobic treatment is desirable as the oil can be recovered for sale as low-grade oil and the POME then becomes more suitable for subsequent anaerobic treatment. A conventional method for breaking up oil-in-water emulsions is the addition of hydrolysing metal salts, especially aluminium sulphate, to wastewater. However, the chemical coagulation method not only decreases the POME pH but also generates a secondary pollutant, namely sulphate, which is known to be an important inhibitor of anaerobic digestion[18]. The sulphate can be reduced to sulphide by sulphate-reducing bacteria[19,20] which suppress methane production by competing with methanogens for common organic and inorganic substrates[21]. In addition, the sulphide produced can be toxic to bacteria[22–24]. For these reasons, electrocoagulation in which the anodes (aluminium or iron electrodes) corrode to release active coagulants, i.e., $Al^{3+}_{(aq)}$ and $Fe^{3+}_{(aq)}$, into a solution without generating sulphate may be more suitable for breaking up oil-in-water emulsions in POME than other methods. Besides oil, electrocoagulation may be able to remove other water pollutants such as suspended and total solids.

In this study, the performance of electrocoagulation in the pretreatment of POME was investigated. The pretreatment of POME by chemical coagulation with aluminium sulphate was also investigated in order to compare the electrocoagulation technique with the conventional chemical method. The operational parameters investigated were the current density and electrocoagulation time. The effect on the process efficiency of environmental factors such as pH and temperature were also considered.

MATERIALS AND METHOD

Palm oil mill effluent

POME was obtained from Suksomboon Palm Oil Co., Chonburi, Thailand, which produces approximately 160 m^3 of wastewater per day. The POME was a mixture of wastewater from sterilization of fresh oil palm fruit brunches, wastewater from clarification of palm oil, and effluent from hydrocyclone operations. The POME had been retained in a holding tank in order to separate floatable oils from water and emulsified oils. Table 1 shows the properties of POME after this primary treatment.

Experimental apparatus and procedure

A bench-scale electrochemical system was used in this study. The system consisted of a DC power supply, a power control and measurement units, an electrochemical reactor, and a magnetic heater and stirrer. The electrochemical reactor was a 2-l beaker (12.5 cm in diameter and 17 cm in height) with a set of five pairs of electrodes made of aluminium. The dimension of the electrodes was 20 × 4 × 0.10 cm (length × width × thickness). The electrodes were connected vertically with a gap of 1 cm and to the DC power supply in monopolar parallel mode, and the electrochemical reactor was operated in a galvanostatic mode. In the monopolar mode, each electrode acts as either anode or cathode, but not simultaneously. In the arrangement of monopolar electrodes with cells in parallel, the electric current is divided between all the electrodes in inverse proportion to the resistance of the individual cells. In the present study, the effect of initial pH on the efficiency of the electrocoagulation method was investigated by adjusting the pH of the POME to a desired value with NaOH. The initial pH imposed was not kept constant, but was recorded at the end of each assay.

Table 1 Characteristics of pre-settled POME and POME before and after electrocoagulation.

Property	pre-settled	before	after
pH	4.39–4.60	4.39	4.98
Conductivity (mS/cm)	12.5–13.3	13.3	19.0
COD (g/l)	39.8–73.2	73.2	26.1
TS (g/l)	37–68	67.7	38.3
SS (g/l)	10–21	10.0	4.7
Oil content (g/l)	2.40–3.73	3.00	0.83
−(Zeta potential) (mV)	16.0–20.5	17.4	22.1

Before and at the end of each run, the electrodes were washed thoroughly with water, dipped in HCl solution (5% v/v) for at least 15 min, and rinsed again with water. Runs were conducted at 313 K and 343 K. During the runs, the reactor unit was stirred at 150 rev/min by a magnetic stirrer (IKA, C-MAG HS, Germany). The experiments were carried out in a batch mode with a liquid sample of 1.5 l. The electrodes were immersed vertically to a depth of 11 cm. The setup resulted in a total electrode working area of 440 cm^2. Current densities of 10, 20, 40, 60, and 80 A/m^2 were applied. For each current density, electrocoagulation times of 2, 5, 10, and 15 min were used. After the current was applied the system was left to settle for 60 min. A sample was then pipetted from the middle portion to analyse the water properties.

Jar test experiment

The initial POME of 1.5 l was transferred into a 2-l beaker and $Al_2(SO_4)_3$ stock solution was then added in steps of 0.5 g/l to obtain a final concentration in the range of 0–5 g/l. The suspension was stirred at 150 rev/min for 5 min and at 30 rev/min for 30 min. The POME was subjected to settling for 60 min before a sample was pipetted from the middle portion of the supernatant.

Analytical method

Analysis of total solids (TS), suspended solids (SS), and chemical oxygen demand (COD) was carried out on the wastewater. The analytical methods followed the Standard Methods of the American Public Health Association[25]. The pH was measured by a pH meter (Schott, Lab 850, Germany) and the conductivity by a conductivity meter (Milwaukee, EC59, Italy). The zeta potential was determined by Laser Doppler Electrophoresis using a Zetasizer 3000 (Malvern Instruments Ltd., UK). Oil content was analysed by a Soxtec 2043 (Foss, Denmark).

The removal efficiency was determined as $(C_0 - C)/C_0$, where C_0 and C are the concentrations of water pollutants (i.e., oil, SS, TS) before and after electrocoagulation, respectively.

RESULTS AND DISCUSSION

In this study, each treatment was repeated twice and the results agreed to within 10%. The main parameter to investigate further was the oil content, which is the main obstacle in the biological treatment of POME. After the POME samples had been retained in a holding container, the floatable oils were removed from the samples leaving most of the oil in the form of emulsified oil. Dispersed oil droplets have been reported to be stabilized by natural emulsifying agents such as tree bark present in POME[26]. The POME in our study was very dark. The dark colour in POME has been attributed to the high content of polyaromatic compounds such as phenols and tannins[27, 28].

Effect of current density and electrocoagulation time

In the first experiments, electrocoagulation of POME was performed at 313 K without adjusting the pH or adding supporting electrolytes. The effect of the electrocoagulation treatment was measured by the final oil content in the POME. The electrocoagulated POME separated into three layers. The top layer contained floating bubbles, fine solids, and oils. The middle layer was liquid containing remaining suspended solids and emulsified oil. The bottom layer was settled as flocks.

The optimal oil-removal efficiency was achieved with a current density of 20 A/m^2 (Fig. 1a). The drop in the oil-removal efficiency at a current density of 80 A/m^2 corresponded to the generation of abundant gas bubbles observed during the electrocoagulation. Previous studies have reported that current density directly influences the number and size of bubbles[29] and that the mass of bubbles produced is proportional to current density[30]. The decrease of the oil-removal efficiency while increasing current density was mainly attributed to the size of gas bubbles whose specific area decreases with the diameter of gas bubbles produced during electrolysis. A possible reason for the decrease of the oil-removal efficiency with increasing current density is that the specific surface area, defined as surface area per unit volume, of the gas bubbles produced during electrochemical process increases with the current density. The increase in the specific surface area then increases the number of small colloidal particles attached to the gas bubbles. During the electrochemical process, the gas bubbles carrying small colloidal particles float to the top. The particles can interfere with the oil-removal process by reducing the probability of oil droplet collisions and the probability of attachment of oil droplets to flocks. An attachment of oil droplets to flocks followed by an immediate flock removal has been reported previously to be an efficient strategy for separating oil from an oil/water emulsion[31].

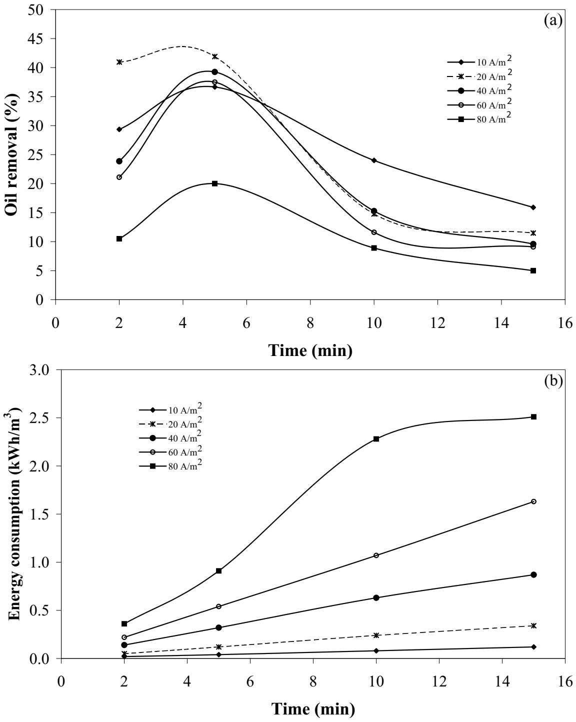

Fig. 1 (a) Oil-removal efficiency and (b) energy consumption as a function of current density and electrocoagulation time (313 K, pH 4, no addition of supporting electrolyte).

The maximum oil-removal efficiency of 42% was obtained with a current density of 20 A/m^2 and electrocoagulation time of 5 min. For current densities of 20, 40, and 60 A/m^2, the oil-removal efficiency was reduced when the electrocoagulation time was increased from 10 to 15 min. As for the case of the high current density of 80 A/m^2, the drop of the oil-removal efficiency at the longer electrocoagulation times corresponded to an increase in gas bubble generation. The generated gas bubbles could be clearly seen to ascend from the bottom to the top, carrying fine solids with them. The height of the bubble layer was more than 5 cm and even spilled over the beaker at times.

The occurrence of gas bubbles with an upward motion seems to set a limit on the use of electrocoagulation for the pretreatment of POME. In the literature, when electrocoagulation was applied to remove oil in a system with low suspended solids and phenolic compounds, the performance of electrocoagulation was highly satisfactory[32, 33].

Before treatment in this study, the POME was acidic (pH 4) and the pH remained between 4 and 5 during the electrocoagulation treatment. An observable physical change was the colour of POME which became darker when a high current density of 80 A/m^2 was applied. Previous studies on the electrocoagulation of olive mill wastewater (OMW) also reported an observable increase in colour intensity of the OMW which was shown to be due to the oxidative polymerization of phenols and tannins originally present in the OMW[27, 28, 34].

The electricity cost for the process was in the range of 0.02 to 2.5 kWh/m^3 with the cost increasing as the current density and the electrocoagulation time were increased (Fig. 1b). At the optimal condition of a current density of 20 A/m^2 and the electrocoagulation time of 5 min, the electrical consumption was 0.12 kWh/m^3. The cost of this pretreatment was relatively low in comparison with the 219 kWh/m^3 for the complete treatment of OMW with 99.54% oil removal reported by Un et al[35].

Effect of pH

An adjustment of pH is typically performed by adding NaOH or Na_2CO_3. The final pH of each assay increased approximately 0.5 ± 0.1. It is generally known that pH increases during an electrochemical process since the process leads to the formation of metal hydroxide as a net final product. The generated metallic ions, i.e., $Al^{3+}_{(aq)}$ in this study, undergo further spontaneous reactions to produce corresponding hydroxides (here, aluminium hydroxide). Aluminium hydroxide starts to precipitate at pH 6 and has its lowest solubility at pH 6.2[36]. When the initial pH was adjusted to 5, the oil-removal efficiency increased slightly from 42 to 45% (Fig. 2). However, a further increase of pH to 6 and 7 reduced the efficiency of oil-removal and also resulted in an increase in the colour intensity of POME. All zeta potential values under the investigation were well below zero (Fig. 2b), indicating that the amount of Al^{3+} released from the electrochemical process with the current density of 20 A/m^2 and electrocoagulation time of 5 min was not sufficient to neutralize the surface charges. It was noted that the zeta potential values became more negative at steady-state pH beyond 6. The increase of surface charge negativity at increasing pH, due to the adsorption of anions from the liquid phase of the suspension[33], was used to explain the decrease of the oil-removal efficiency by increasing the initial pH to 6 and 7.

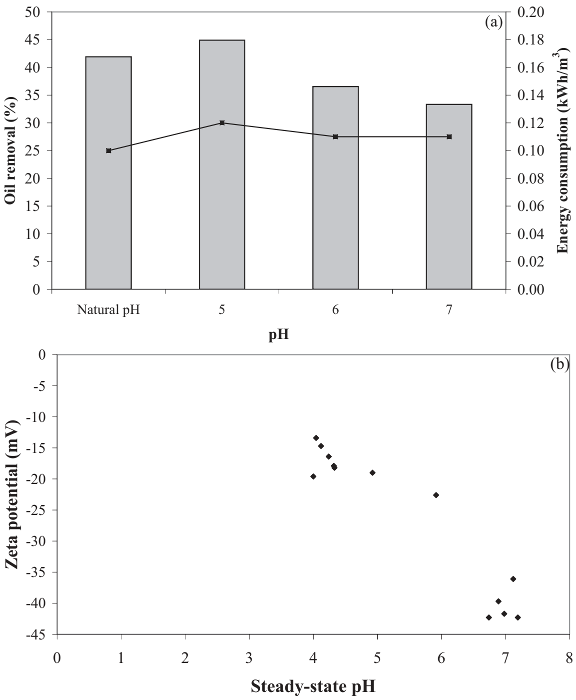

Fig. 2 (a) Influence of pH on oil-removal efficiency and energy consumption (current density: 20 A/m^2, electrocoagulation time: 5 min, temperature: 313 K, no supporting electrolyte added). (b) Zeta potential as a function of steady-state pH.

Effect of temperature

POME is typically discharged at temperatures of 353–363 K which makes treatment at both mesophilic (298–318 K) and thermophilic (324–343 K) conditions feasible. The effect of temperature on electrocoagulation was investigated in the range of 313 and to 343 K at steps of 10 K. An increase in temperature did not lead to an increase in treatment efficiency (Fig. 3). The temperature can affect the electrocoagulation process in many ways such as rate of reactions, solubility of metal hydroxides, liquid conductivity, and kinetics of gas bubbles, or small colloidal particles. Despite this, previous studies have reported an insignificant effect of temperature on electrocoagulation performance[35, 37]. However, the role of

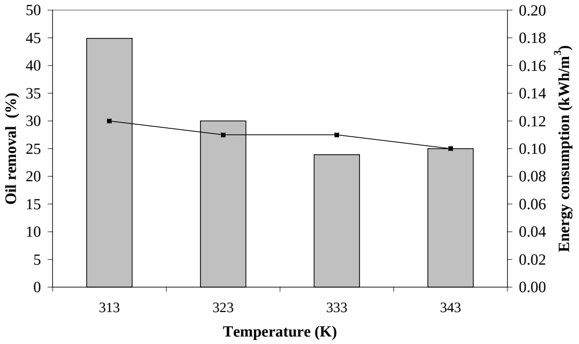

Fig. 3 Oil-removal efficiency and energy consumption as a function of temperature (current density: 20 A/m^2, electrocoagulation time: 5 min, pH: 5, without an addition of a supporting electrolyte.

temperature is not straightforward because there are many competing mechanisms in an electrocoagulation process. In the present study, bubble formation was one of the major characteristics of electrocoagulation treatment of POME and an increase in temperature might be expected to have a negative effect due to a corresponding increase in kinetic energy and random motion of the gas bubbles. The increase in the random motion of the gas bubbles and small colloidal particles could interfere with the processes of oil droplet coalescence and attachment of oil onto flocks.

Effect of supporting electrolyte

The influence of electrolyte dosage on the removal of oil from POME was investigated. Sodium nitrate was chosen as the supplementary electrolyte because it does not interfere with the electrochemical reactions[30] and it can serve as a nitrogen source for the working microorganisms in the subsequent biological treatment. The added concentration of $NaNO_3$ was such that the ratio of COD:N was equal to 100:2 (w/w) which is a desirable value for anaerobic bacteria[38]. For an initial COD concentration in the order of 70 g/l, the added concentration of $NaNO_3$ was 8.5 g/l (0.1 M). This was an amount comparable to values used in previous studies on the effect of electrolyte dosage[39]. With a current density of 20 A/m^2 for 5 min electrocoagulation, at 313 K and pH 5, the addition of $NaNO_3$ resulted in a substantial increase of the oil-removal efficiency from 45 to 72%. The electrical consumption under this condition was in the order of 0.10 kWh/m^3. The addition of $NaNO_3$ increased the POME conductivity from 13 to 19 mS/cm. The increased conductivity can facilitate the passage of current. Yildiz et al[40] reported that the addition of Na_2SO_4 resulted in the delivery of more iron to the medium. The present study showed that the addition of $NaNO_3$ is beneficial to the pretreatment by electrocoagulation. It is also expected that this addition could also be beneficial to a subsequent biological treatment.

The properties of POME before and after the electrocoagulation treatment with the addition of $NaNO_3$ and with a current density, electrocoagulation time, temperature, and pH of 20 A/m^2, 5 min, 313 K, and 5, respectively, are shown in Table 1. The table shows the electrocoagulation treatment increased the pH of the POME. This is expected because the treatment generated hydroxide ions. The increase in conductivity was mainly due to the addition of $NaNO_3$. It can be seen that the oil content, COD, SS, and TS were reduced by 72%, 64%, 53%, and 43%, respectively. The average zeta potential indicates that the surfaces of the remaining suspended particles in POME remained negative after the electrocoagulation process.

Comparison with alum coagulation

Chemical coagulation of POME was performed using aluminium sulphate in jar test experiments. The effect of $Al_2(SO_4)_3$ dosage was first studied by varying the weight dosage without a pH control. In contrast to the electrocoagulation, the alum coagulation resulted in a pH reduction of the POME. This reduction occurs because aluminium sulphate is acidic. At the lower dosages, the oil and SS removal increased with the dosage (Fig. 4). However, when the $Al_2(SO_4)_3$ dosage was higher than 2 g/l (equivalent to 11.7 mM of Al^{3+}), the pH dropped below 4 and the oil and SS removal also dropped significantly. In another jar test study, the pH was held at 4.64 and $Al_2(SO_4)_3$ dosage was varied between 0–12 g/l (equivalent to 70.2 mM of Al^{3+}). As shown in Fig. 5(a), both oil and SS can be almost completely removed. In another jar test study, the $Al_2(SO_4)_3$ dosage was fixed at 2 g/l (equivalent to 11.7 mM of Al^{3+}), and the pH was varied between 3–9. It was found that the oil could be removed almost completely at all pH except 3. As shown in Fig. 5(b), pH had an effect on the removal efficiency.

In a previous study, Cañizares et al[33] examined the effect of both coagulation and electrocoagulation on destabilizing synthetic oil-in-water emulsions. They found that the oil-removal efficiency was independent of the dosing technology. In the present study, the efficiency of electrocoagulation was compared with the efficiency of chemical coagulation. The amount of iron released into solution by electrolytic oxidation of the anode was estimated from Faraday's

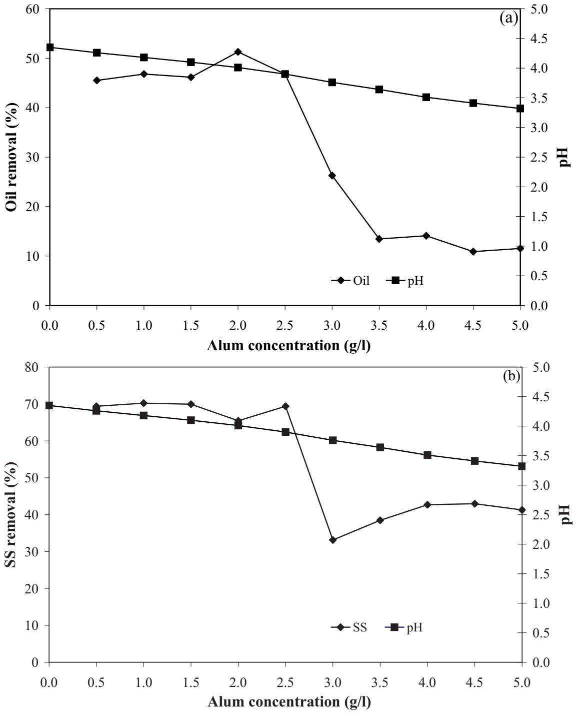

Fig. 4 (a) Oil-removal efficiency and pH as a function of alum concentration (b) SS removal efficiency and pH as a function of alum concentration.

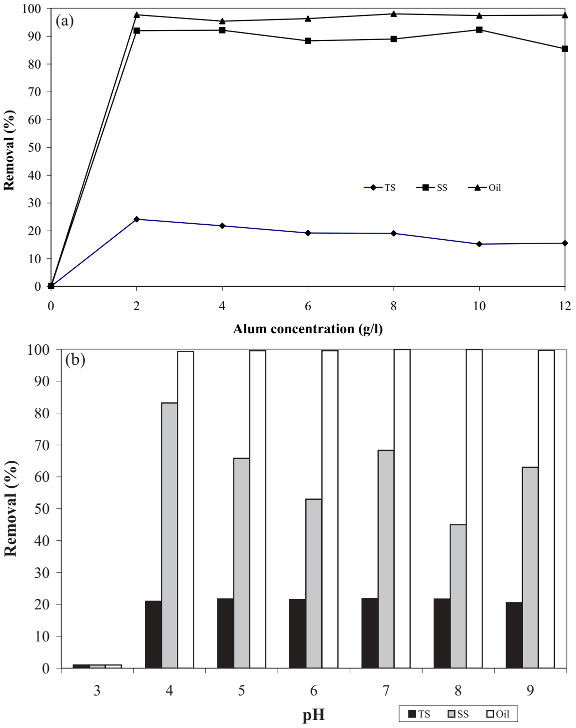

Fig. 5 (a) Oil, SS, and TS removal efficiency as a function of alum concentration held at pH 4.64 (b) Oil, SS, and TS removal efficiency as a function of pH with a fixed alum concentration at 2 g/l.

law:

$$w = \frac{ItM_{\mathrm{Al}}}{ZF}, \quad n_{\mathrm{Al}^{3+}} = \frac{JAt}{ZF},$$

where w is mass of the aluminium dissolved, $n_{\mathrm{Al}^{3+}}$ is the number of moles of aluminium dissolved, I is the current, J is the current density, A is the total working area, t is the electrocoagulation time, M_{Al} is the molecular mass of Al, Z is the number of electrons involved in the redox reaction (+3), and F is Faraday's constant.

At the best condition with a current density of 20 A/m^2 and an electrocoagulation time of 5 min, the electrocoagulation was calculated to produce a dose of 9.12 mmol of Al^{3+}. In 1.5 l POME, the dose was equivalent to a [Al^{3+}] of 6.1 mM. For the chemical coagulation method and an equivalent dose of $\mathrm{Al_2(SO_4)_3}$, i.e., 1.0 g/l, the oil-removal efficiency was comparable with the electrocoagulation. However, the efficiency of the two techniques became clearly different at higher aluminium doses. For electrocoagulation, it was found that the bubbles generated during the process set a limit on the efficiency of the oil-removal process. This limit was due to the small colloidal particles in the POME which caused interference with the processes of oil droplet coalescence and the attachment of oil onto flocks. This bubble effect did not occur in the coagulation process. However, chemical coagulation also had a limit in removing oil from POME due to the pH effect. A pH control was necessary for coagulation by $\mathrm{Al_2(SO_4)_3}$. If the pH was lower than 4, the removal efficiency of oil and SS dropped significantly and at a pH of 3 no oil and SS was removed by the process. It should also be noted that the TS removal efficiency of the coagulation by $\mathrm{Al_2(SO_4)_3}$ was lower than that of the electrocoagulation. This suggests that electrocoagulation can remove both suspended and dissolved solids whereas chemical coagulation can mainly remove suspended solids. A possible explanation is that the underlying mechanism for solid removal by alum coagulation is the surface charge neutralization of suspended solid particles. However, the underlying mechanism of electrocoagulation appears to be more complex. The generated $\mathrm{Al}^{3+}_{(\mathrm{aq})}$ ions can undergo further spontaneous reactions to produce corresponding hydroxides and/or polyhydroxides. These hydroxides/polyhydroxides/polyhydroxymetallic compounds have strong affinity with dispersed/dissolved substances as well as the counter ions to cause coagulation/adsorption/complexation[10,36,41,42]. At the equivalent dose of [Al^{3+}] of 6.1 mM, the amount of sludge produced during the electrocoagulation was approximately double that of the chemical coagulation of POME, i.e., 29 kg of sludge produced per m^3 of POME by electrocoagulation and 12.5 kg/m^3 by chemical coagulation.

The operating costs of the two methods were compared at the same oil-removal efficiency of 45%. The costs were estimated from the chemical and equipment prices in January 2010. The main operating costs of the electrocoagulation are electrical energy and electrode consumption costs, while those of the chemical coagulation are chemical and transportation costs. The electrical energy cost was estimated based on the required electrical energy of 0.12 kWh/m^3 of POME and the electrical charging rate of 3 baht/kWh. The electrode consumption cost was estimated based on the electrode consumption rate of 6.1 mmol of Al^{3+}/m^3 of POME (equivalent to 0.16 kg/m^3) and the market price of thin aluminium sheet (1 mm thickness) of 40 000 baht/ton. The chemical cost was estimated based on the required $Al_2(SO_4)_3$ dose of 1.0 g/l (equivalent to 1.0 kg/m^3 of POME) and the market price of commercial alum (16% $Al_2(SO_4)_3$) of 4500 baht/ton). The transportation cost for the chemical delivery was estimated to be 300 baht/ton of the commercial alum. The disposal cost was 1000 baht/ton of sludge. Based on these prices, the operating costs of the electrocoagulation are 0.36 baht/m^3 of POME for electrical energy, 6.4 baht/m^3 of POME for electrodes, and 29 baht/m^3 of POME for sludge disposal. The operating costs of the chemical coagulation are 28 baht/m^3 of POME for chemicals, 1.88 baht/m^3 of POME for transportation, and 12.5 baht/m^3 of POME for sludge disposal. Even though the cost estimation is only preliminary, the electrocoagulation is estimated to be cheaper than the conventional chemical coagulation.

CONCLUSIONS

The efficiency of electrocoagulation as a pretreatment of POME has been found to be satisfactory. With an equivalent dose of aluminium ions ([Al^{3+}]) of 6.1 mM, the oil-removal efficiencies of both electrocoagulation and chemical coagulation were comparable. The electrocoagulation technique was a relatively more environmentally friendly process as it could avoid the addition of approximately of 9.2 mM sulphate ions into POME. A combination of low current density and low electrocoagulation time was preferable. The generation of gas bubbles at high current densities and/or long electrocoagulation time set a limit on the efficiency of oil removal by electrocoagulation.

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