

## **Removal color from Palm Oil Mill Effluent (POME): Electrocoagulation Method vs Microfiltration using Nanofiber Membrane**

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Palm oil trees have long been introduced in Malaysia since before the establishment of the country. Nowadays, Malaysia is known as the second major exporter of palm oil and palm-based products, which totals up to 17.95 million tonnes of oils and fats around the globe in 2017. However, the rapid growth of palm oil plantation led to the increment of wastewater due to palm oil processing. Hence, various methods for treatment of palm oil mill effluent (POME) were implemented. In this study, two methods were chosen namely electrocoagulation and microfiltration. In electrocoagulation method, 2V to 10V of potential difference were applied with iron as electrode while for microfiltration, nylon 6,6 nanofiber membrane was used and were fabricated using electrospinning method. The results show that by using electrocoagulation method, 94.2% removal of color was observed for 10V and 13.4% for 2V while for microfiltration, 13.5% was obtained. Chemical oxygen demand (COD) reduction using electrocoagulation method was observed to be higher with 69.9% reduction for 10V while for microfiltration, 68.5% COD reduction was recorded. Hence, electrocoagulation is seen to be more efficient in treating palm oil-based wastewater compared to microfiltration.

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**Keywords:** palm oil; effluent; electrocoagulation; microfiltration; nanofiber

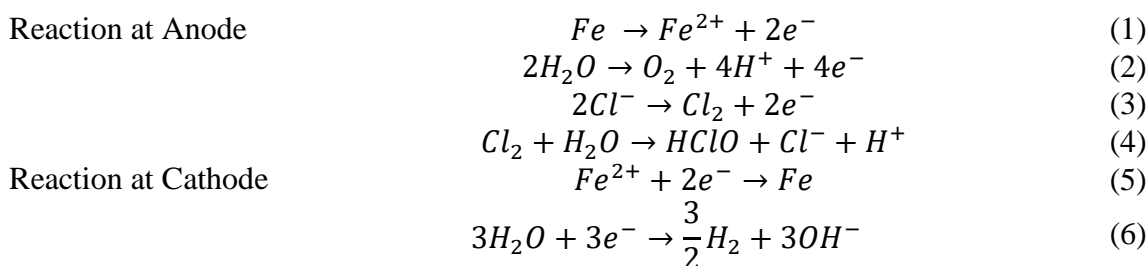
### **1. INTRODUCTION**

Palm Oil Mill Effluent (POME) is known to be the major pollutants of water embodiment since most of the effluent is released to the nearby river hence disrupted the main source of water in Malaysia [1]. It was reported that for every metric tonne of Crude Palm Oil (CPO) produced, approximately 2.5-3.75 tonnes of POME were emitted [2]. Thus, more than 45 million metric tonnes of POME were released to the environment every year given that palm oil produced yearly in Malaysia could reach

17.95 million metric tonnes [3]. POME is defined as a viscous brown liquid with pH ranging from 4-5 and consists of fine suspended solids [4]. Raw POME from mill usually found to be in brownish color resulted from the presence of humic acid and fulvic acid-like component [5] and the mixture is buildup of colloids where the temperature is around 353 K to 373 K. POME comprises mainly water (95-96%), total solids (4-5%) and oil (0.6-0.7%). The high amount of organic compound causes it to have high value of biological oxygen demand (BOD) and chemical oxygen demand (COD).

Up to present, various methods such as biological treatment [6], adsorption [7], Fenton-oxidation [8] and membrane technology [9,10] have been used to treat POME. One of the common methods is open ponding system in which was implemented to almost 85% of palm oil mill in Malaysia [11]. Apart from open ponding system that relies on settling of the particle and size of the tank, anaerobic digestion was also well known for its efficiency in converting organic compounds as well as capturing renewable biogas that could be converted into source of energy [6]. However, the use of anaerobic digestion plant was reported to be time consuming, cost-intensive and has complexity of operation [4,12]. Others are relatively new and yet to be implemented in real-life plant.

Recent studies show that electrocoagulation method is an excellent alternative to palm oil-based wastewater treatment. The basic principle of electrocoagulation is almost similar to coagulation and flocculation except there is no addition of coagulants as the ionization of the electrode material replace the coagulants. Electrocoagulation is a process of neutralization of the particles charge present inside the wastewater which repels with each other [13]. By neutralizing the charge, suspended particles would coagulate together to form bigger masses hence ease the settling process. Most authors agreed that electrocoagulation method is a process that stands on three clear stages, which are (i) electrolytic oxidation on electrode surfaces (ii) destabilization of contaminants that forms coagulant in aqueous phase and (iii) sedimentation of destabilized particles or flocs [14–17]. The reactions that occur at cathode and anode of the electrocoagulation system are well explained by Equation (1) – (6) [18].



Apart from electrocoagulation, filtration using membrane technology is also a promising approach in treating POME. In general, membrane filtration can be categorized into four types; which are microfiltration (MF), ultrafiltration (UF), nanofiltration (NF) and reverse osmosis (RO) [19,20]. These membranes were classified based on pore sizes, molecular weight cut-off and trans-membrane pressure [21]. Pore sizes for MF ranges between 0.1-10  $\mu\text{m}$ . Most contaminants such as suspended particles and large colloids that have size between 0.08-2  $\mu\text{m}$  are suitable for separation using MF [22]. UF consists of pores with size 0.001 – 0.1  $\mu\text{m}$ , NF with pore size 0.005-0.0007  $\mu\text{m}$ , and RO with lower than 0.001  $\mu\text{m}$  [23–25]. UF is suitable for separation of carbohydrate, protein, viruses and macro-nutrients that present in water [26–28]. As for NF and RO, they are more suitable for desalination and

removal of dissolved constituents [26]. In removing constituents in POME, usage of NF and RO may be too inefficient due to large particles and colloids that presents in the wastewater. The narrow pore size of NF and RO induced fouling at faster rate in which would render low productivity [29,30]. Thus, selection of MF in removing colloids and small particle is seen to be more logical and has better productivity [31]. MF membrane such as nanofiber membrane (NFM) for example has great potential to be applied for wastewater treatment due to its better fouling problem handling, porous structure compared to conventional membrane and high surface area relative to its volume [32–34]. Moreover, it also has a low production cost which makes it preferable to be used in membrane technology.

In this research, comparison between electrocoagulation and MF using nylon 6,6 NFM is investigated based on their effectiveness in removing color and reducing COD of POME. After extensive reading on past research, currently no available topic has discussed on the comparison between electrocoagulation and MF in degradation of POME. In this research, the significance of oxidized iron electrodes used during electrocoagulation of POME is also discussed and compared with pure iron electrodes in which has not been reported in past researches. Thus, the aims of this research will be to determine the influential parameters on electrocoagulation method and to select the most efficient method in removing color as well as COD of POME.

## 2. EXPERIMENTAL

### 2.1. Materials

POME was obtained from FELCRA Nasaruddin palm oil mill, Seri Iskandar, Perak and was used as feed solution. Sodium chloride (NaCl) was purchased from Sigma-Aldrich, USA and used as electrolyte in feed solution for electrocoagulation setup. Pure iron plates with dimension 5.0 cm × 10.0 cm × 0.2 cm was acquired as cathode and anode during electrocoagulation. Oxidized iron electrodes were prepared by exposing pure iron electrodes in room temperature of 27°C after soaked with tap water for 72 hours. Chemicals used for NFM preparation were formic acid (98-100%) obtained from Merck, USA, acetic acid from Merck, USA and nylon 6,6 pellets from Sigma-Aldrich, USA.

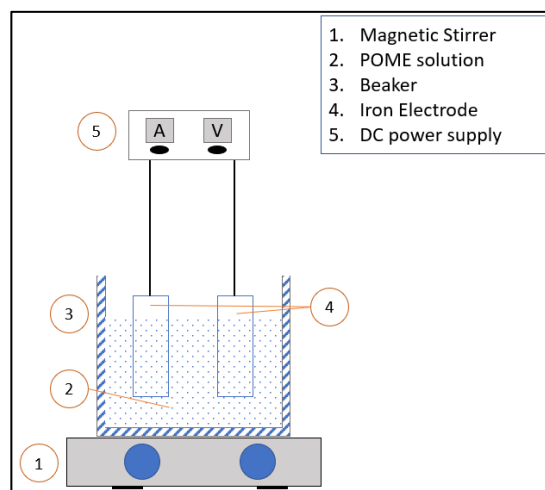
### 2.2. Pre-treatment of feed solution

POME solution obtained was filtered from grains and silt using a 100 microns strainer. POME solution was then diluted with distilled water with a ratio of 1:10 of POME to distilled water.

### 2.3. Electro-coagulation setup

Direct current (DC) was supplied to two iron electrodes from a DC power supply. 400 ml of POME solution was measured and poured into a beaker. To provide electrolyte for electro-oxidation of the cathode and anode, 1g of NaCl was added into the POME solution. The beaker was placed on a magnetic stirrer to provide consistent contact of wastewater to the electrodes surfaces. Voltage and

electrocoagulation time are parameters manipulated during experimental work. 5 different voltage were applied which are 2V, 4V, 6V, 8V and 10V to observe the effect of voltage on removal efficiency. POME was observed every 15 minutes until 60 minutes for every voltage during electrocoagulation to study the effect of time towards removal efficiency. The configuration of the setup is illustrated in Fig. 1.

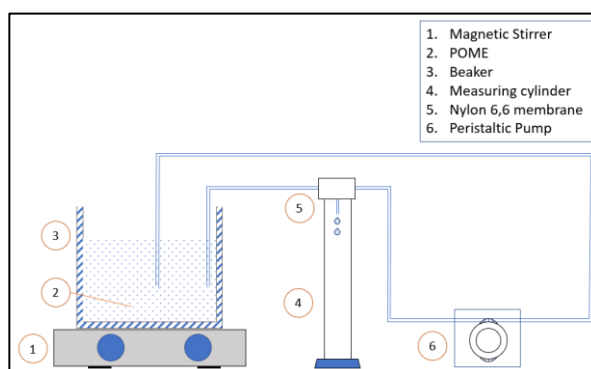


**Figure 1.** Electrocoagulation setup of pre-treated POME solution using 2V, 4V, 6V, 8V and 10V of potential difference and 27°C.

#### 2.4. Nylon 6,6 Nanofiber Membrane Preparation

Nylon 6,6 NFM was synthesized using electrospinning method. First, membrane solution was prepared by dissolving nylon 6,6 pellets (14 wt.%) in a mixture of acetic acid and formic acid at 1:1 ratio. Next, 5 ml syringe was used and attached to a syringe pump which later will inject the solution to a rotating collector. The flow rate was set at 0.4 ml/h and the distance from needle tip to a metal screen collector was 15 cm. 20 kV of voltage was applied and the rotation of collector was maintained at 500 rpm.

#### 2.5. Microfiltration Setup



**Figure 2.** Microfiltration setup using nylon 6,6 nanofiber membrane with pressure maintained at 0.1 bar and 27°C

A 8cm × 4cm × 3cm rubber casing was used to hold nylon 6,6 NFM in place and mounted on a 500 ml measuring cylinder. Permeate was collected every 15 minutes to measure the permeability. Treatment time for microfiltration ends after permeate reached steady state. Permeate obtained after steady state is achieved was sent for UV-Vis and COD analysis. A peristaltic pump was used to drive the pressure difference and maintained the flow of solution at 0.1 bar. The configuration of the membrane setup is illustrated in Fig. 2.

## 2.6. Membrane Characterization

Nylon 6,6 NFM was characterized based on membrane morphology, surface roughness, pore size and porosity. Membrane morphology was observed by using Field Emission Scanning Electron Microscope (VPFESEM, ZeissSupra55 VP). Average surface roughness was determined with Atomic Force Microscopy (AFM) using Universal Scanning Probe Microscope (USPM, Model: NanoNavi E-Sweep). Pore size measurement was taken using ImageJ software and membrane porosity was determined using dry wet method.

## 2.7. Methods and parameters

The degradation of POME was measured based on percentage of color removal and COD reduction by using UV-Vis spectroscopy. To determine the percentage of color removal, two parameters were taken into consideration for electrocoagulation process; time and voltage. The color of POME was measured for every 15 minutes until 60 minutes and the voltage were set at 2V, 4V, 6V, 8V and 10V. For COD removal, only voltage will be varied from 2V, 4V, 6V, 8V and 10V in order to study the effect of voltage towards reduction of COD in palm oil based wastewater. COD measurements were taken at initial condition where no treatment method was applied and later were compared to the treated samples after 60 minutes for each voltage. For microfiltration, the permeate was collected and tested for color using UV-Vis and COD removal once it has reached steady state condition. The COD reduction is determined based on equation (7).

$$COD\ reduction\ (\%) = \frac{COD_0 - COD_t}{COD_0} \times 100\% \quad (7)$$

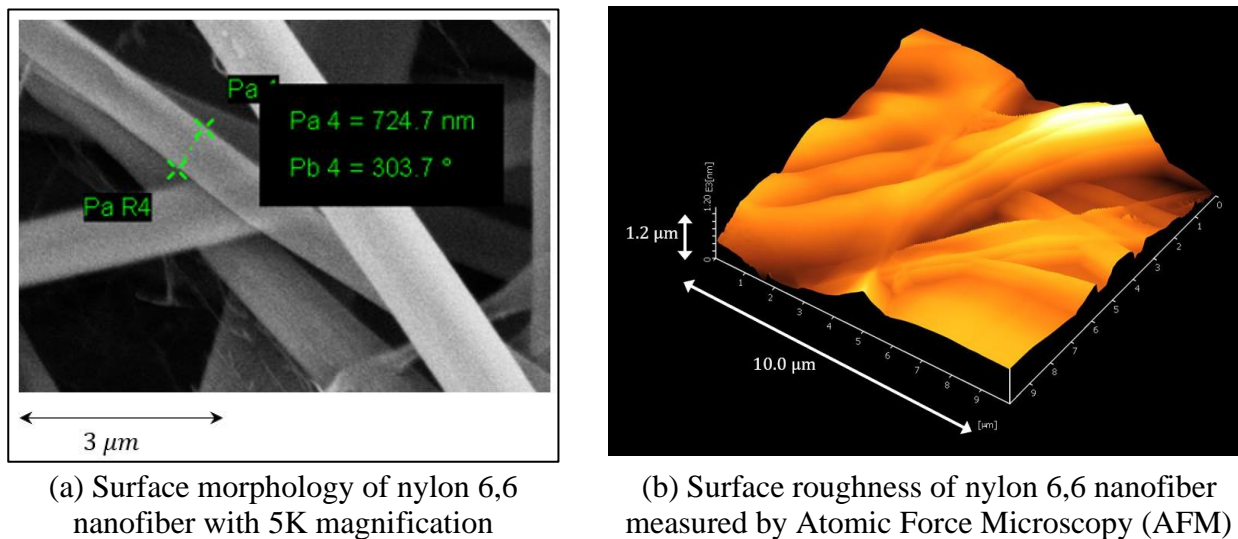
Where  $COD_0$  is initial COD of wastewater (mg/l) and  $COD_t$  is COD of treated wastewater at selected voltage at 60 minutes.

## 3. RESULTS AND DISCUSSION

### 3.1. Characterization of nylon 6,6 nanofiber membrane

The surface morphology of nylon 6,6 NFM is shown in Fig. 3(a). Based on the figure, non-uniform and bead-free fibers with cylindrical morphology were obtained. The diameter of the fibers is

approximately  $724.7 \pm 45.0$  nm. As for the surface roughness of nylon 6,6 NFM (Fig.3 (b)), it is 165.6 nm, respectively. Surface roughness plays an important role in controlling the fouling in membrane separation. Membrane that has rougher surface tends to get fouled easier due to more number of valleys present at the surface hence ease the accumulation of particles [35]. Based on Table 1, nylon 6,6 NFM has pore size and porosity of 0.2  $\mu\text{m}$  and 71.3% which also correlated with literatures [31,35,36].



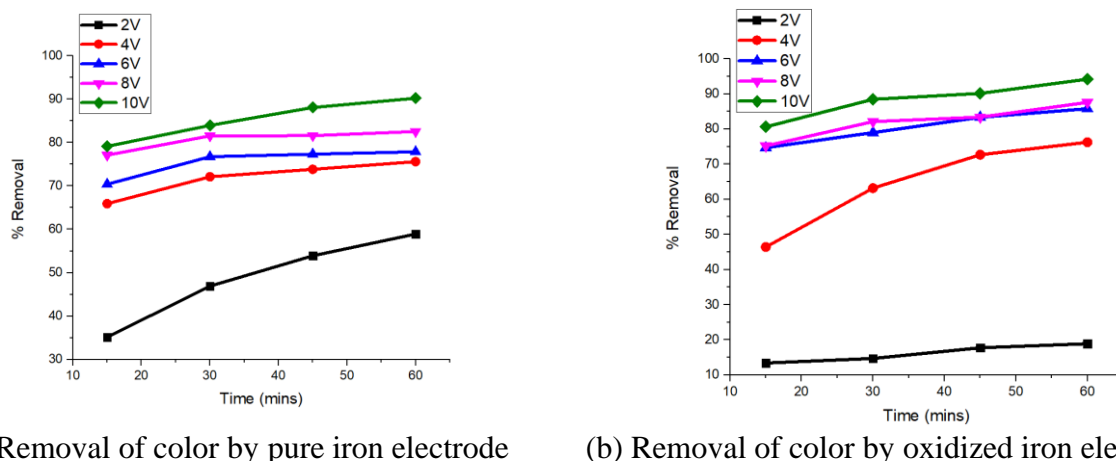
**Figure 3.** Characterization of nylon 6,6 nanofiber by FESEM and AFM

**Table 1.** Nylon 6,6 nanofiber membrane characteristics analyzed by ImageJ software, FESEM and AFM.

Fibre diameter (nm)	Pore size ( $\mu\text{m}$ )	Porosity (%)	Surface Roughness (nm)
$724.7 \pm 45.0$	0.2	71.3	165.6

### 3.2. UV-Vis Spectroscopy Analysis

Based on Fig. 4, the time and voltage applied to the electrocoagulation setup has greatly affected the color removal. The longer the time of electrocoagulation process, the higher the percentage of color removal. This is proven by the trend shown by both pure iron electrode and oxidized electrode. As the voltage is applied to the electrodes and the reaction time is prolonged, more metal atoms will turn into ions. In this experiment, more  $\text{Fe}^{3+}$  and  $\text{Fe}^{2+}$  ions were formed.



(a) Removal of color by pure iron electrode

(b) Removal of color by oxidized iron electrode

**Figure 4.** Performance of different electrodes for different voltage and time using electrocoagulation method at 27°C and continuous stirring.

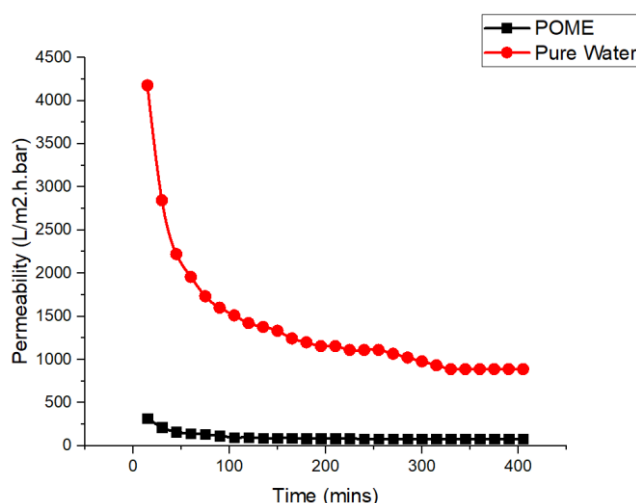
Voltage applied is found to be linearly proportional to percentage of color removal. At high potential difference, the rate of anode dissolution would also increase [37][38,39]. However, when higher voltage is applied, the increment of color removal between the first 15 minutes until 60 minutes is not as high when compared to low voltage. Based on Table 2, the removal percentage is 35.2% for pure iron at 2V and 15 minutes, while at 60 minutes, the removal percentage is 58.9%, respectively. This makes up a difference of 23.7%. The difference in percentage of color removal between 5 minutes and 60 minutes are 7.9%, 5%, 5.5%, 11.1% for 4V, 6V, 8V and 10V, respectively. A same trend can also be observed for oxidized iron electrode at which the percentage differences are significant with 29.8% for 4V compared to 6V, 8V and 10V which stand at 11.1%, 12.4% and 13.6% respectively. This is due to the presence of leftover complex organic molecules inside the wastewater, hence retards the mineralization rate [40]. Further contact time of electrocoagulation and excess current applied may not be as efficient as more energy will be used and consumed the electrode, hence increase the operational cost [41].

**Table 2.** Percentage removal of color between electrocoagulation and membrane method using UV-Vis spectroscopy analysis.

Time (mins)	% Removal										
	Pure Iron Electrode					Oxidized Iron Electrode					Membrane Treatment
	2V	4V	6V	8V	10V	2V	4V	6V	8V	10V	
15	35.2	65.9	70.4	77.1	79.1	17.7	46.4	74.7	75.2	80.6	13.50
30	46.9	75.6	76.7	81.6	83.9	18.9	63.2	79.0	82.1	88.5	
45	53.9	72.1	77.3	81.5	88.1	14.7	72.7	83.3	83.4	90.1	
60	58.9	73.8	75.4	82.6	90.2	13.4	76.2	85.8	87.6	94.2	

In microfiltration process, the performance was based on permeability of pure water and POME. According to Fig. 5, pure water has higher permeability with 4178 (L/m<sup>2</sup>.h.bar) during initial period of

the experiment and reached steady state after 330 minutes where the permeability maintained at 889 (L/m<sup>2</sup>.h.bar). Nevertheless, the permeability of POME is lower compared to pure water. The initial permeability started at 320 (L/m<sup>2</sup>.h.bar) and reached steady state after 240 minutes with 80 (L/m<sup>2</sup>.h.bar). This indicates that POME contains large amount of solutes and colloidal particles which unable to penetrate through the membranes pores [10]. This leads to fouling phenomena where the membrane pores were blocked by large particles, hence reducing membrane flux. Besides that, lower permeability may indicates the colloids and color removal of POME. However, based on Table 2, the color removal was observed to be only 13.5%. This is due to the presence of smaller particles i.e. humic acid and fulvic acid in POME [5]. Humic acid has average diameters of approximately 300-600 nm [42]. NFM only able to filtrate large particles and smaller particles such as humic acid and fulvic acid which causes the brownish color of POME still able to pass through the membrane, hence causes low removal of color in MF.



**Figure 5.** Comparison of membrane permeability between pure water and POME as feed with constant pressure at 0.1 bar and 27°C.

### 3.3. COD Analysis

In electrocoagulation process, electrodes are the key element as the reaction occurs mainly on the electrode surface. Thus selection of electrode is important to determine the efficiency of metal oxidation hence improving the overall efficiency of the process. Iron metals are easily obtained due to its low cost and readily available in market. This section focuses on the COD reduction between two types of electrode, pure iron and oxidized iron. Table 3 shows the percentage of COD reduction between electrocoagulation and membrane treatment. At 60 minutes and 2V, pure iron electrode exhibits 20.2% of COD reduction while oxidized iron shows 13.7% reduction. By increasing the voltage to 4V, pure iron electrode could reach more than half COD reduction compared to oxidized iron which only manage to reduce to 47.3%. The trend is valid from 2V until 8V only. This implies that the higher the voltage applied, the higher the COD reduction. This phenomenon can be best explained by the increment of ion production in anode and cathode of both electrodes. Fe<sup>3+</sup> and Fe<sup>2+</sup> ions were formed on anode surfaces



as current was applied [43]. The metal ions are coagulants in which would actively react with colloids that have opposite charges and bind them together [44]. Thus along with the agglomeration of the particle, organic molecules which have lower diameter or size tends to traps between the colloids [13]. Apart from that, with addition of NaCl, chloride ion ( $\text{Cl}^-$ ) would form during salts dissolution in water.  $\text{Cl}^-$  is one of the strong oxidizing agent which help to disintegrate or oxidize organic particles that present in wastewater [45]. However at 10V, oxidized iron electrode has shown a higher COD reduction with a difference of 0.5%. This is explained by Rossmeis et al (2005) that water can dissociate to form adsorbed O atoms, and only after the surface has oxidized sufficiently to make this step slower, it will trigger the evolution of oxygen mechanism [46]. In this experiment, 10V was seen to be the threshold for the oxygen evolution to occur more in oxidized iron electrode compare to pure iron electrode.

For MF, COD reduction is at 68.5% in which almost the same as electrocoagulation method at 10V. This is due to the ability of NFM to trap more organic matters that would help in COD reduction. More interconnected pores were formed during electrospinning in which create higher surface area [47]. In addition, NFM has less pressure drop compared to conventional filters [48]. However, despite having almost similar COD reduction as electrocoagulation, microfiltration requires longer time to achieve steady-state condition.

**Table 3.** COD reduction in percentage between electrocoagulation and membrane treatment of POME.

Voltage	COD Reduction (%)		
	Pure iron	Oxidized iron	Microfiltration
2V	20.2	13.7	68.5
4V	55.6	47.3	
6V	68.9	60.8	
8V	68.9	62.6	
10V	69.4	69.9	

#### 4. CONCLUSION

Both electrocoagulation and microfiltration methods exhibit excellent potential treatment for POME. Selection of electrode is crucial in determining the binding agent that may be produced during electrocoagulation. A good selection of electrode should poses excellent removal color of wastewater as well as COD. Iron for instance is largely used as electrode due to its trivalent ions production when given current is applied. For oxidized iron, the efficiency at lower voltages were slightly lower compared to pure iron but performs better at 10V, and maybe onwards. Time and voltages both are linearly equal to the color removal efficiency as well as COD reduction. Microfiltration on the other hand removed COD efficiently at 68.5%, however has lower percentage of color removal (13.5%) as compared to electrocoagulation (94.2%). In respect with time, microfiltration requires at least 240 minutes to reach maximum efficiency of filtration while electrocoagulation only requires 60 minutes to obtain 94.2% of color removal. Hence, electrocoagulation method is proven to be more efficient and reliable in treating POME compared to microfiltration.

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