# The Study of Semi-Continuous Chemical Physical Treatment System for Pollutant Removal in Palm Oil Mill Effluent

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### Abstract

Effluent from palm oil mill processing is a major concern due its properties and characteristics that highly contributed to water pollution. This study reported on POME treatment by a semi-continuous system consisting of coagulation, treatment sedimentation and filtration processes in the presence of a by-product coagulant (copperas combined with *lime). The treatment was conducted for different types* of filter medias (FMs): a) FM 1: sand+activated carbon and b) FM 2: sand+ceramic ring) and various retention times (RT) from 1/2 to 2 h. The treatment resulted in different removal efficiencies of turbidity, colour, chemical oxygen demand (COD) and total suspended solids (TSS) for secondary and tertiary POME samples. With semi-continuous treatment, the pollutants in the secondary POME sample decreased, resulting in the removal efficiency of 57.34% for turbidity, 95.16% for colour, 82.08% for COD and 83.23% for TSS.

Meanwhile, the results of the tertiary sample were 54.81%, 81.08%, 78.05% and 68.51% for turbidity, colour, COD and TSS removal respectively. The statistical analysis of ANOVA (p-value<0.05) indicated that the data was statistically significant with the FM 1 as a media filter. As a result, a semi-continous chemical-physical treatment system may provide information in efficient POME treatment.

**Keywords:** Copperas, POME, coagulation, sedimentation, filtration.

# Introduction

Malaysia is one of the world's largest palm oil exporters with approximately 28% and 33% of the global palm oil production and exports respectively<sup>25</sup>. The palm oil tree or *Elaeis guineensis* originated from West Africa and the tree developed into an agricultural crop. The oil palm plantation in Malaysia was introduced in the early 1870s. In 2020, Malaysia had planted about 5.90 million hectares of land with palm oil trees, producing 19.86 million tonnes of palm oil and 2.32 tonnes of palm kernel oil<sup>25</sup>.

Palm oil production is commonly carried out from fresh fruit bunch (FFB) by a mechanical process. Typically, a palm oil mill has many operational units as shown in figure 1. This includes sterilisation, stripping, digestion and pressing, clearing (clarification), cleaning (purification), drying and storage. Oil extraction from oil palm fruits requires no additional chemicals. Thus, the effluent from a palm oil mill is a non-toxic waste. In Malaysia, wet palm oil milling is typically done using hot water to leach out the oil.

This process uses a large amount of water, thus producing a large amount of wastewater<sup>18</sup>. Palm oil mill effluent (POME) production is known as one of the biggest producers of palm oil processed by a palm oil mill. The production rate is about 0.67 m<sup>3</sup> per tonne of FFB processed<sup>26</sup>. It is estimated that POME contributes about 30% of the total biological oxygen demand (BOD) load discharged on the Malaysian aquatic environment. Hence, POME cannot be discharged directly to either land or watercourse. This is because POME will significantly affect the soil and vegetation systems. Besides, the effluent can also reduce water quality for aquatic lives when POME is discharged directly without treatment<sup>29</sup>.

POME can also pollute water which will reduce the quality of water sources (e.g. rivers and lakes). Irenosen et al<sup>13</sup> stated that the effluent should be treated so that the organisms present in the watercourse can tolerate the effluent.

Significant increases in crude palm oil (CPO) production have always been a threat to the environment as oil contains high organic and nutrient content if discharged directly as raw or partially treated POME into nearby watercourses. Thus, effective treatment methods are very important to discharge effluent with acceptable quality. Various treatment methods and technologies have been used in treating POME. A wastewater treatment facility is the most crucial component of a palm oil mill system. This facility is used to treat effluent from the mill, which is produced in a large volume during CPO production. POME treatment methods include a ponding system that consists of aerobic, anaerobic and facultative ponds, adsorption, biological treatment, coagulation-flocculation and membrane technology.

Bello and Abdul Raman<sup>5</sup> stated that the ponding system use might require long retention time (RT) and a large treatment area even though this system is economic compared to other treatment systems. Taheran et al<sup>38</sup> stated that membrane technology offers a high removal rate, stable effluent quality, modularity, free from the addition of chemicals and the ability to integrate with other wastewater treatment systems.



Figure 1: Oil processing in a palm oil mill

The application of membrane ultrafiltration for POME treatment removed about 88% chemical oxygen demand  $(COD)^{35}$ , 80% total suspended solids  $(TSS)^{35}$  and 57.0%  $COD^{41}$ .

However, the main drawback of membrane technology is the blocking of membrane surface which affects the maintenance and capital cost and also affects the membrane<sup>35</sup>. Furthermore, it is still challenging to comply with strict regulations using membrane technology. As a result, there is a need to study the treatment that can be used so the pollutant can be reduced before being discharged.

In this present study, a semi-continuous laboratory scale wastewater treatment system consisting of chemical and physical methods (coagulation, sedimentation and filtration) was developed and applied for POME treatment. The aim of this study is to improve the effluent quality of POME and meet the standard discharge limit. Copperas, a by-product from titanium manufacturing industries, was used as coagulant integrated with calcium hydroxide (lime) for chemical treatment followed by physical treatment using dual-media filtration. These treatment steps were implemented to identify the potential in removing turbidity, colour, COD and TSS and also determine the effect on the final pH.

Coagulation-flocculation process or chemical treatment is one of the preferred purification methods for urban and industrial wastewater<sup>31</sup>. Coagulation is used as a pretreatment to remove a significant amount of colloidal and particulate organic matter in residual water. It is an easy process due to its simple operation, relatively simple design and consumption of low energy<sup>2</sup>. The most commonly used metal coagulants for pretreatment are aluminium and ironbased coagulants<sup>39</sup>. These coagulants are simple, easy to handle, cheap and have good wastewater removal efficiency<sup>16</sup>.

A study has been conducted on the treatment of rubber effluent using iron (II) sulphate giving a percentage removal more than 90% of BOD, COD, SS and  $NH_3^{27}$ . Aziz and Wan Kamar<sup>4</sup> also stated that iron (II) sulphate can improve colour by removing about 73% colour from domestic wastewater sample.

In physical treatment, water was filtered into a bed of granular materials in granular filtration via adsorption

approaches. It will seperates molecules or ions from aqueous solution and adsorbs them on solid surfaces of the granular materials<sup>1</sup>. Filtration was the most common method for removing dissolved particulate matter of water or wastewater treatment and purification<sup>32,33</sup>. Rapid sand, slow sand and diatomaceous earth are the three types of granular filtration methods used today<sup>37</sup>. A few study stated that the used of sand as a filter is considered a cost-effective option<sup>17,20,34</sup>.

Application of slow sand filter was a simple and easy-to-use method that enables raw water to flow through a medium<sup>12,34</sup> and it removed any nonsettled floc<sup>6</sup> while rapid sand filter is a form of physical filtration and does not generate a large biological layer compared to slow sand filter<sup>12</sup>. Filtration is one of the environmentally friendly wastewater treatment methods among other physico-chemical processes. Several studies have been conducted on the use of sand filters in the treatment of wastewater (remove >99% COD and TSS)<sup>11</sup> and seawater treatment (remove micro-algae)<sup>33</sup>.

Thus, a combination of chemical and physical treatment were integrated for the removal of pollutant from POME. A complete coagulation-floccution (chemical treatment) process using a suitable coagulant will then pass into a granular filter (physical treatment), which removes any residual debris.

According to previous study, the combination of coagulation/flocculation and dual-media filter may reduce particulate fouling on membranes<sup>10,15,24,28</sup> and dual-media filter can work effectively trappig larger and smaller particles along the media<sup>43</sup>. Few studies have been conducted on the application of a combined treatment for petrochemical effluent (50.58% SS and 10.42% COD removal)<sup>40</sup>, for algal cultivation (78±18% particles removal)<sup>10</sup> and for seawater treatment<sup>21,22</sup>. This research was focused on the application of the combination of chemical

and physical treatment for the removal of turbidity, colour, COD and TSS and will also determine the effect on the final pH.

# Material and Methods

Sample and preparation: POME was obtained from a local palm oil mill. An adequate quantity of POME was collected from the outlet of an anaerobic pond (as the secondary POME sample) and also from the final discharge of the mill (as the tertiary POME sample). The samples were transferred into a plastic container, labelled and sealed before being transported to the laboratory. POME was kept at a temperature less than 4 °C prior to use to avoid biodegradation due to microbial action. The coagulants used in this study were a combination of copperas with lime. Copperas or known as ferrous sulphate heptahydrate (FeSO<sub>4</sub>.7H<sub>2</sub>O) is a by-product from the titanium dioxide industry supplied by Venator Material Corp., Teluk Kalung, Terengganu. The combination of these coagulant is in the ratio of 80:20 (Copperas:lime). Both coagulants were prepared separately in 2% concentration.

The characterisation of secondary and tertiary POME samples involved the measurement of pH, turbidity, colour, COD and TSS of raw and treated POME. The pH and turbidity of POME were tested using pH and turbidity (ORION AQ3010 Model) meters respectively. The laboratory experiments involved the tests for colour (Method 8025), COD (Method 8000) and TSS. The characterisation was conducted before and after conducting the experiments using the semi-continuous treatment system. All data was presented in average of three values.

**Semi-continuous treatment system (laboratory scale):** Figures 2 and 3 illustrate the schematic diagram and the setup of the semi-continuous treatment system respectively representing a small-scale laboratory wastewater treatment system.



Figure 2: Schematic diagram of a small-scale treatment system

The treatment unit consists of a raw wastewater tank, a coagulant dosing tank, a coagulation-flocculation, sedimentation and a filtration media. The size of each tank was the same which was 20 cm x 10 cm x 10 cm with the dosing flowrate 0.13 L/min.

For the small-scale laboratory wastewater treatment system (as shown in figure 3), the process started as the raw POME (secondary and tertiary POME samples) was pumped concurrent with the coagulation-flocculation tank. In the coagulation-flocculation tank, the raw POME sample was mixed by mechanical agitation with a stirrer at a speed of 150rpm for 20 minutes. The partially treated wastewater then flowed into the second container where sedimentation occurred for about 30 minutes. The treated wastewater from the settling unit flowed into the empty tank and then it was pumped to the filtration unit. In filtration, two different filter medias (FMs) were used which were a) FM 1: sand+activated carbon and b) FM 2: sand+ceramic ring.

Sand size in the range from 0.5 to 1.5 mm was used as first filter media. The use of sand filter in each filter media is cost-effective and does not require any addition of chemical as stated by Kumar et al<sup>17</sup>. After the treatment was complete, the samples were collected for analysis. The experiments were conducted to determine the final pH and the reduction of turbidity, colour, COD and TSS.

**Removal efficiencies:** After conducting the treatment using the semi-continuous system, both samples were tested

according to the parameters studied (i.e. the final pH, turbidity, colour, COD and TSS). Then, the removal efficiencies of all parameters were calculated. The formula used is presented in equation 1:

$$R, \% = (1 - C_f / C_i) \times 100$$
(1)

where R is the percentage removal and  $C_i$  and  $C_f$  are the initial and final concentrations of the wastewater quality studied respectively.

#### **Results and Discussion**

**Characteristics of POME:** Table 1 presents the values of secondary and tertiary POME samples. The tests were carried out on the samples before conducting the semicontinuous treatment for characterisation purposes.

**Semi-continuous POME treatment system:** The semicontinuous treatment in laboratory scale was performed to determine the efficiency of coagulant used with the help of the FM, followed by the study on the effect of RT in removing turbidity, colour, COD and TSS.

**Effect of different filter types:** The semi-continuous treatment was initially carried out using different filter media (granular filtration) for treating the secondary and tertiary POME samples. Granular filtration can eliminate pollutants from the water matrix as the water flows through granular materials<sup>30</sup>.



Figure 3: The setup of laboratory-scale POME treatment

Table 1						
Results of the characterisation tests for secondary and tertiary POME samples						
Parameter	Unit	Secondary POME	Tertiary POME	Discharge		
		sample	sample	limit		
pH	-	7.96	8.15	5.0-9.0		
Turbidity	NTU	143.33	107.33	-		
Colour	Pt-Co	2075	444	-		
COD	mg/L	738.67	322	-		
TSS	mg/L	656	308	200		

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The process was done without changing the pH value and the dosage of 2,000 mg/L of 80:20 copperas:lime. The influence of both FM1 and FM2 for the removal of turbidity, colour, COD and TSS from secondary and tertiary POME samples is shown in figures 4 and 5 respectively.

Figure 4 shows the percentage removal efficiency for the semi-continuous treatment system using different filter media for the secondary POME sample. Higher percentage removal was obtained after filtration using FM 1, with 57.35%, 95.16%, 82.08% and 83.23% for turbidity, colour, COD and TSS respectively. The final pH of the sample after completing the treatment was 8.71.

Meanwhile, for FM 2, the removal efficiencies were 50.79%, 87.65%, 69.22% and 84.91% for turbidity, colour, COD and TSS respectively with the final pH of 8.69. Activated carbon with numerous micropores (0–2 nm), mesopores (2–50 nm) and macropores (> 50 nm)<sup>42</sup> achieved higher removal compared to ceramic ring with 1–5  $\mu$ m pore size<sup>36</sup>. Pollutant removal is higher for smaller pore size.

Figure 5 illustrates the percentage removal for the tertiary POME sample. Both filer media could remove the pollutants in the tertiary POME sample. FM 1 achieved the percentage

removal of 54.81%, 81.08%, 78.05% and 68.51% for turbidity, colour, COD and TSS respectively. As stated by Zhang et al<sup>42</sup>, activated carbon was typically used in water and wastewater treatment as a porous adsorption material with different sizes and a high specific surface area. However, FM 2 demonstrated lower percentage removal of 63.8%, 49.28% and 44.16% for colour, COD and TSS respectively. The percentage removal for turbidity was negative, in which the final value was higher than the initial value. It was postulated that during the chemical treatment, the floc formed dispersed and remained in the suspension. The final pH values for the secondary and tertiary POME samples were 8.71 and 8.77 respectively.

As discussed above, both filter media can remove the pollutants from POME samples. Activated carbon can adsorb organic materials as the forces between the surfaces of carbon and the contaminants are greater than the forces that hold the components submerged in water<sup>8,28</sup>. Activated carbon also has a high surface area and porosity that can improve water quality by adsorbing various compounds<sup>9,28</sup>. Hoslett et al<sup>12</sup> stated that activated carbon was a highly adaptable filtration material that can be used to remove a wide range of pollutants. Hence, FM1 was then selected for the subsequent parameters in the study.







Figure 5: The removal efficiencies of turbidity, colour, COD and TSS for the treatment of tertiary POME sample using different filter media

**Effect of retention time:** The effect of RT on the final pH value and the removal of turbidity, colour, COD and TSS was evaluated. The parameters were evaluated using the filter media with the best performance as discussed previously. The coagulant, dosage and mixing speed were maintained. The RT was varied from 0 to 2 h at the sedimentation tank prior to filtration to ensure the formation of flocs. The graphs for the percentage removal of turbidity, colour, COD and TSS of secondary and tertiary POME samples are shown in figures 6 and 7 respectively.

Figure 6 shows that different RTs produced different percentage removal efficiencies for the secondary POME sample. For turbidity, the highest percentage of turbidity removal was 62.16% at 2 h RT. Meanwhile, colour recorded its highest percentage removal at 0 h RT when the system flowed continuously with 95.16% removal without any addition of RT at sedimentation tank. For COD, an increase in RT resulted in decreased COD removal at 0, 1 and 2 h RT respectively. It postulated that increase in retention time will lead the suspended materials disperse back and remains as a suspension as it has reached its optimum retention time at 0 h.

A study by Simate<sup>37</sup>, on the treatment of brewery wastewater

showed good COD removal performance (i.e. 96.0%) by a semi-continuous treatment plant consisting of coagulation using ferric chloride, flocculation and filtration by filter beds and carbon nanotubes. For TSS, the highest percentage removal was recorded at 2 h RT with 90.40%.

The graph of the percentage removal of turbidity, colour, COD and TSS for the tertiary POME sample for the effect of RT is shown in figure 7. From the figure, it was observed that the semi-continuous treatment system achieved its optimum removal efficiency at 0 h RT with 54.81%, 81.08% and 78.05% for turbidity, colour and COD respectively. Even though 1 and 2 h RT resulted in reduced percentage removal, the percentage was higher than 50%, except for turbidity. For TSS, an increase in RT increased the removal efficiency. The highest removal was obtained at 2 h RT with 85.71%.

A study showed that with 24 h RT, the removal of turbidity, TSS, COD and anionic surfactant was 89.5%, 81.5%, 56.4% and 77.4% respectively from the treatment of laundry waste using a plant-based coagulant. Mohan<sup>23</sup> stated that a settling period of one day or more is required for a natural coagulant in removing pollutants. It is believed that the use of a chemical-based coagulant requires shorter sedimentation time in removing the contaminants from the POME sample.







Figure 7: The removal efficiencies of turbidity, colour, COD and TSS on the treatment of tertiary POME sample at different retention times

		Secondary POME sample			
Parameter	Initial value	After sedimentation	After filtration	Percentage removal	
				(%)	
рН	7.96	8.56	8.77	-	
Turbidity	143.33 NTU	66.73 NTU	61.13 NTU	57.34	
Colour	2075Pt-Co	226. 00 Pt-Co	100.33 Pt-Co	95.16	
COD	738.67 mg/L	253.33 mg/L	132.33 mg/L	82.08	
TSS	656 mg/L	120 mg/L	110 mg/L	83.23	
		Tertiary POME sample			
Parameter	Initial value	After sedimentation	After filtration	Percentage removal	
				(%)	
pН	8.15	8.19	8.71	-	
Turbidity	107.33 NTU	96.27 NTU	48.5 NTU	54.81	
Colour	444 Pt-Co	92.0 Pt-Co	84.0 Pt-Co	81.08	
COD	322 mg/L	151.00 mg/L	70.67 mg/L	78.05	
TSS	308 mg/L	115 mg/L	97 mg/L	68.51	

 Table 2

 Summary of the best performance of both samples in each stage of the semi-continuous treatment system

Both secondary and tertiary POME samples showed percentage removal, depending different on the characteristics of the water quality of the sample. A study conducted by Choi et al<sup>7</sup> on the treatment of tetracycline antibiotic by coagulation (polyaluminium chloride as a coagulant) and granular activated carbon filtration achieved a removal efficiency higher than 68% of the incoming tetracycline antibiotic. Another study on the post-treatment of tannery wastewater using bittern as a coagulant, followed by adsorption using activated carbon showed good removal efficiency. The study obtained 99% turbidity, 97% TSS, 71% COD, 99.7% chromium, 57% BOD<sub>5</sub> and 87% phosphorous removal efficiency<sup>3</sup>.

Table 2 presents the summary of the samples before and after subjected to the semi-continuous treatment system in terms of the selected wastewater quality for secondary and tertiary POME samples. The phases of coagulation/flocculation and sedimentation are significant in turbidity, colour, COD and TSS removal. Copper as combined with lime effectively removed the pollutants in both samples. Ismail et al<sup>14</sup> stated that the use of ferrous sulphate and lime increased the particle size of suspended materials, hence increasing the settling and coagulation of suspended materials.

Moreover, the presence of filter media enhanced the selected wastewater quality as the particles settled in the sedimentation tank, resulting in less pore clogging in the filter media<sup>37</sup>. The addition of activated carbon after sand filter helped more for the contaminants removal. As stated by Hoslett et al<sup>12</sup>, activated carbon was able to adsorb organic/taste/odour materials, synthetic organic chemicals and other contaminants. This is due to the large surface area of the activated carbon itself in adsorbing the organic matter<sup>28</sup>.

A study of variance (ANOVA) with a significance level of 95% ( $\alpha$ =0.05) was used, tabulated in table 3 and 4 for

turbidity, colour, COD and TSS removals of secondary and tertiary POME treatment respectively. The findings in both table showed that there was a significant difference (p-value<0.05) in all parameter studied, indicating that the null hypothesis ( $H_o$ ) should be rejected which assumes that the retention time (treatment) was equal for all the parameter removals. So, these data are statistically significant according to the ANOVA.

# Conclusion

A semi-continous laboratory scale wastewater treatment system comprising of chemical and physical treatment was analysed for the elimination of the pollutant from the wastewater. The combination of copper as a low-cost coagulant with calcium hydroxide was used during chemical treatment and followed by physical treatment of dual-filter media whch was a) FM1: sand+activated carbon and b) FM 2: sand+ceramic ring.

The treatment system can treat both secondary and tertiary POME sample in the range of 50-80% removal for turbidity, colour COD and TSS and in acceptable range of pH value to be discharged by using 80:20 copperas:lime as coagulant, followed by sand and activated carbon as the filter media.

# Acknowledgement

This work was supported by Venator Asia Sdn. Bhd. for the funding (Vot 53335). We are thankful to Faculty of Ocean Engineering, Technology and Informatics, Universiti Malaysia Terengganu for the contribution and support of this project.

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(Received 12<sup>th</sup> March 2023, accepted 19<sup>th</sup> May 2023)