

THE EFFECTS OF LIGHTWEIGHT MACROCOMPOSITE ON THE TREATMENT  
OF PALM OIL MILL EFFLUENT

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***To God be the Glory! Specially dedicated to my beloved one,  
Mum, Dad and family members, my never ending supportive  
supervisors Dr Nurliyana Ahmad Zawawi, Prof Zaharah Ibrahim &  
Assoc Prof Dr Zaiton Abdul Majid, my caring lecturers, HOPE JB  
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Kuching Girls Brigade Family...***

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## ABSTRACT

As agricultural industries are developing over the years, the discharged of untreated waste materials are also on the rise. Example includes the discharged of palm oil mill effluent (POME) from the palm oil mill industry. To mitigate this matter, final discharged of POME was treated using three types of macrocomposites coded MAC1, MAC2 and MAC3 developed in this study. MAC3 was developed consisting of 77% pumice rock as lightweight aggregates (5-10 mm size range), 10 % pumice powder, 20% cement, 2% zeolite (500-200  $\mu\text{m}$ ) and 1% activated carbon (AC). The efficiency of MAC3 for POME treatment was compared to other macrocomposites made at similar composition as MAC3 but without zeolite (MAC2) and without both zeolite and AC (MAC1). All macrocomposites were chemically tested for its durability, in acidic to alkaline conditions. Physical test conducted for its thermal property at 121 °C for 20 mins at 15 psi and deterioration test under running tap water for 14 days. All three macrocomposites were analyzed for their ability to reduce chemical oxygen demand (COD), color and ammoniacal nitrogen ( $\text{NH}_3\text{-N}$ ) in examine the applicability for POME bioremediation efficiency. The pH value following treatment were monitored too. Rate of COD removal was examined in another set of experiment, using adsorption kinetics and isotherm models. The biofilm developed on macrocomposites were calculated for its dry cell weight and examined its surface morphology using scanning electron microscopy (SEM). From the results, all macrocomposites demonstrated no change of the structures after exposed to both 1M of hydrochloric acid (HCl) and sodium hydroxide (NaOH). As for 5 M of HCl and NaOH, the macrocomposites were all disintegrated after a period of time, as the cement may lose its binding properties in high concentration of acidic and alkaline conditions. When treated with POME, MAC3 demonstrated highest POME removal compared to MAC2 and MAC 1 with color reduction data of  $65.17 \pm 1.67\%$  (3100 ADMI), COD removal ( $42.51 \pm 0.28\%$  (741 mg/L) and  $\text{NH}_3\text{-N}$  reduction ( $65.77 \pm 0.05\%$  (38 mg/L) after 10 days incubation. Treatment with macrocomposites, however, indicated high alkalinity of the POME that was highly attributed to the alkali content of cement that was used up to 20% of total macrocomposite content. the isotherm analysis, MAC3 gave the maximum adsorption ( $q_e$ ) at  $6.946 \times 10^{-4}$  mg/g. Pseudo-first-order gave the best model equation for COD removal of POME and that the rate of adsorption is increased with  $\text{MAC2} > \text{MAC3} > \text{MAC1}$ . The color, COD and ammoniacal nitrogen reduction by macrocomposites is also suggested to be enhanced by the biofilm coated on the macrocomposites, indicated by cluster cells that deposited on the porous structure of macrocomposites in SEM. MAC3 contained the highest biofilm dry cell weight ( $3.14 \pm 0.11$  mg/g) than MAC2 and MAC1, in which suggested that the highest surface roughness produced a higher biofilm formation. In conclusion, MAC3 is promising to treat POME, however, further improvement is recommended to improve their performance in terms of reducing the pH value towards an environmentally friendly absorbent system.

## ABSTRAK

Sektor perindustrian pertanian yang semakin membangun dari tahun ke tahun menyebabkan berlakunya peningkatan sisa buangan. Antara contoh termasuk sisa buangan efluen kepala sawit (POME) dari industri kelapa sawit. Bagi mengatasi masalah ini, discaj terakhir POME telah dirawat menggunakan tiga jenis makrokomposit yang dibangunkan dalam kajian ini iaitu MAC1, MAC2 dan MAC3. MAC3 telah dibentuk menggunakan 77% agregat ringan daripada batu pumis (julat saiz: 5-10 mm), 10% serbuk pumis, 20% simen, 2% zeolit (julat saiz: 500-200  $\mu\text{m}$ ) dan 1% karbon teraktif. Keberkesanan MAC3 dalam rawatan POME dibandingkan dengan macrocomposites MAC2 yang mempunyai kandungan sama seperti MAC 3 kecuali zeolit, dan MAC1 yang tidak terkandung kedua-dua zeolit and karbon teraktif. Kesemua makrokomposit diuji tahap ketahanan dalam keadaan berasid dan beralkali. Kajian fizikal juga dilakukan dengan menguji sifat haba pada 121 °C selama 20 mins pada 15psi, dan tahap ketahanan kikisan makrokomposit dibawah aliran air paip selama 14 hari berturut-turut. Ketiga-tiga makrokomposit tersebut diuji kandungan oksigen kimia (COD), warna, kandungan ammoniak-nitrogen ( $\text{NH}_3\text{-N}$ ) untuk mengenalpasti kecekapannya membio-pulih POME. Nilai pH berikutan dengan rawatan tersebut juga dipantau. Kadar penyingkiran COD diperiksa dalam set eksperimen lain menguunakan model penyerapan kinetik dan isoterma. Pembentukan biofilem pada setiap makrokomposit juga dihitung bagi menentukan berat kering sel dan diperiksa morfologi dengan mikroskopi electron pensakanan (SEM). Hasil keputusan kajian menunjukkan kesetiap makrokomposit tidak menunjukkan sebarang perubahan setelah terdedah dengan 1M HCl dan NaOH. Ketiga-tiganya didapati terserak apabila didedahkan dengan 5M HCl dan NaOH, berikutan kehilangan sifat pengikatan simen pada kepekatan asid dan alkali yang tinggi. MAC3 menunjukkan tahap penyingkiran POME yang tinggi dalam rawatan bio-pemulihan berbanding dengan MAC2 dan MAC1, ditunjukkan melalui data, penyahwarnaan POME sebanyak  $65.17 \pm 1.67\%$  (3100 ADMI), penyingkiran COD ( $42.51 \pm 0.28\%$  (741 mg/L) dan  $\text{NH}_3\text{-N}$  ( $65.77 \pm 0.05\%$  (38 mg/L) selepas 10 hari. Rawatan menggunakan makrokomposit walaubagaimanapun menunjukkan peningkatan nilai alkali POME yang tinggi berikutan penggunaan komponen simen sebanyak 20% dari jumlah keseluruhannya. Dalam analisis isoterma, MAC3 menunjukkan kadar jerapan paling tinggi, ( $q_e$ ) sebanyak  $6.946 \times 10^{-4}$  mg/g. Ketiga-tiga makrokomposit juga mematuhi model pseudo kinetic tertib pertama bagi kadar penyingkiran COD dalam POME, dan tahap jerapan didapati meningkat dari  $\text{MAC2} > \text{MAC3} > \text{MAC1}$ . Penyahwarnaan, penyingkiran COD dan  $\text{NH}_3\text{-N}$  juga dicadangkan dipertingkatkan oleh pembentukan biofilem diatas permukaan makrokomposit, yang ditunjukkan melalui pembentukan endapan gugusan sel pada liang permukaannya di dalam SEM. Pembentukan biofilem oleh MAC3 mengandungi berat sel kering paling yang tinggi pada  $3.14 \pm 0.11$  mg/g berbanding MAC2 dan MAC1 disebabkan kerana permukaannya yang lebih kasar. Sebagai kesimpulan, MAC3 didapati berpotensi tinggi dalam rawatan POME namun kajian lebih lanjut diperlukan bagi memperbaiki prestasinya khususnya dalam pengurangan tahap pH kearah penghasilan bahan jerap yang lebih mesra alam.

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**LIST OF ABBREVIATION**

<i>ADMI</i>	-	American Dye Manufacturers Institute
<i>Al</i>	-	aluminium
<i>APHA</i>	-	American Public Health Association
<i>BOD</i>	-	Biological Oxygen Demand
<i>COD</i>	-	Chemical Oxygen Demand
<i>cm</i>	-	centimetre
<i>DOE</i>	-	Department Of Environment
<i>EPS</i>	-	Extracellular polymeric substances
<i>g</i>	-	gram
<i>g/L</i>	-	gram per litre
<i>h</i>	-	hour
<i>HCL</i>	-	hydrochloric acid
<i>K</i>	-	potassium
<i>ml</i>	-	millilitre
<i>Mg</i>	-	magnesium
<i>NaOCL</i>	-	sodium hypochlorite
<i>NaOH</i>	-	sodium hydroxide
<i>NH<sub>3</sub>-N</i>	-	ammoniacal nitrogen
<i>POME</i>	-	Palm oil Mill Effluent
<i>rpm</i>	-	rotation per minute
<i>Si</i>	-	silica
<i>Ti</i>	-	titanium
<i>°C</i>	-	celcius
<i>w/v</i>	-	weight per volume

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## CHAPTER 1

### INTRODUCTION

#### 1.1 Background of Research

Malaysia is the second largest producer of palm oil in the world after Indonesia (Norwana *et al.*, 2012; Sharma *et al.*, 2015). According to Bessou *et al.* (2017), palm oil is the first vegetable oil consumed worldwide that leads to high demands of productions by consumers. The process of palm oil requires large quantities of water from the mills to extract the oil from palm fruits. Crude palm oil's extraction will resulted to the production of palm oil mill effluent, (POME) (Okwute and Isu, 2007).

POME, a thick brownish viscous liquid waste has a high colloidal suspension with unpleasant odor and contains high organic nutrient compounds (Habib *et al.*, 1997, Muhrizal *et al.*, 2006, Ahmad *et al.*, 2009). Even though POME is biodegradable, it cannot be discharged without being treated first due to its high acidic content and the presence of residual oil (Madaki and Seng, 2013). POME usually discharged into nearby rivers or land as this is the easiest and cheapest method for disposal. The discharged of POME has been reported to give a great impact to the environment by causing contaminations and loss of biodiversity.

Besides, POME also caused increase rate of biochemical oxygen demand (BOD), chemical oxygen demand (COD) and the concentrations of total solids present in the river nearby (Sing nee'Nigam and Pandey, 2009; Wu *et al.*, 2010; Soleimaninanadegani and Manshad, 2014). The presence of the unwanted compounds in the waste also led to harmful effects on both aquatic life and human health (Verma,

2008; Verla *et al.*, 2014). The raw POME has an average value around 25 000 mg/L which is a hundred times of more pollution than domestic sewage (Maheswaran and Singam, 1977). Thus, indicating that palm oil industry is one of the largest polluters in Malaysia (Kwon *et al.*, 1989).

According to Irenosen *et al.* (2014), several measures have been conducted to treat POME in Malaysia such as the use of biological, chemical and physical method. For instance in biological treatment process, large pond is required to hold the POME in place for the effectiveness of biodegradation by microorganisms like bacteria, algae and fungi (Soleimaninanadegani and Manshad, 2014). Other biological treatment involves vermicomposting technique, a method that uses earthworms to digest POME and produce vermicompost products that can be used as fertilizer (Rupani *et al.*, 2010). Even so, these processes often leads to some drawbacks such as the presence of toxic materials in the wastewater that needed to be treated, which would cause death to the bacteria before the treatment could occur (Irenosen *et al.*, 2014).

Meanwhile for the chemical treatment, coagulants such as aluminium sulphate, ferric sulphate and ammonium sulphate with polymer used in treating POME (Tan *et al.*, 2006). Karim and Lau (1987) also mentioned that the use of chemicals in treating wastewater provide a short period of time without requiring an immense space for coagulation and flocculation to occur. However, chemical treatment is very challenging and expensive to handle as it may cause environmental damage into the microhabitats of the environment (Nwuche *et al.*, 2014). In physical method, it is applied most in the pre-treatment of POME such as filtration or skimming of the effluent. Nevertheless, the availability of oil and grease in POME are not effectively being separated during the pre-treatment process (Yacob *et al.*, 2005). The hurdles in treating POME has led to the advancement of technology such as adsorption technology that is well-known in treating wastewater of industrial effluent wastes.

Adsorption technique often used as adsorbent can be reusable with low operating cost and at the same time, the absorbent can aimed towards specific metals (Kilic *et al.*, 2009; Xia *et al.*, 2014). Example of adsorbents that have been proven to be efficient in treating wastewater are activated carbon (Daifullah *et al.*, 2003), carbon



nanotubes (Li *et al.*, 2007) and waste calcite sludge (Merrikhpour and Jalali, 2012). Das and Barman (2013) mentioned that adsorption has been found to be having lots of advantages compared to the other treatment processes in terms of cost effective, easily to be design and less sensitive to toxic substances.

Based on previous studies, a cement-based material known as *Biostructure* was developed for the treatment of various wastewater such as textile (Harun, 2004; Zaidoddin, 2004), domestic (Seng, 2003), river (Azhar, 2006), palm oil mill effluent (Mohamad Lazim *et al.*, 2014) and petrochemical wastewater (Zaini *et al.*, 2015). This study aimed at developing a cement-based material known as macrocomposite for the treatment of POME. The macrocomposite consist of the same components in *Biostructure* except that the aggregates was replaced with a lightweight aggregates, pumice rock, which was obtained from a volcanic area in Java, Indonesia.

The pumice rock has a property of high porosity and surface area for attachment and development of biofilm. The usage of pumice rock in purifying water has been studied by Matsunaga and colleague (2006) and was successfully being produced by a Japanese Company, KOYOH Corporation. Their EcoBio-Block (EBB) use pumice rock as one of the main components in the building structure that will incorporated with microorganisms such as *Bacillus subtilis natto* that aid in the purification of water (Matsunaga *et al.*, 2007). The use of EBB enable natural purification processes to occur that can renew and revitalize the environment. In Malaysia, the river of Malacca Marashiarancawi Shimauchi uses EBB for the water treatment processes (KOYOH Corporation, n. d).

Moreover, activated carbon and zeolite that act as absorbents were also used in this development of macrocomposite. Activated carbon is made up of pure carbon that has a porous structure with high surface area and work on the principle of adsorption. This contributes to the wide application of activated carbon in purification, discoloration and removal of odor process involved in wastewater treatment processes (Cazetta *et al.*, 2011; Agrawal *et al.*, 2017). Zeolite on the other hand, has a high cation-exchange characteristics which allows dissolved cations to be removed from

the wastewater via ion exchange on zeolite's exchange sites which can remove organic impurities such as humic acids, proteins and lipids (Kallo, 2001; Eapen *et al.*, 2016).

## 1.2 Research Objectives

The aim of the study is to develop macrocomposites comprising of activated carbon, zeolite, cement and lightweight aggregates for bioremediation of POME from Mahamurni Plantations Sdn. Bhd., Sedenak Palm Oil Mil, Johor Darul Ehsan.

The specific objectives of this study are as follows:

- a) To develop macrocomposite MAC3 comprising of activated carbon, zeolite, cement and lightweight aggregates (pumice rock)
- b) To investigate bioremediation efficiency of POME by MAC3, in comparison to macrocomposite without activated carbon and zeolite (MAC1) macrocomposite with activated carbon and without zeolite (MAC2)
- c) To analyze the efficiency of COD removal in POME discharge by developed macrocomposite MAC3 through kinetic and isotherm studies, in comparison to MAC1 and MAC2.
- d) To characterize the biofilm-coated formed on the macrocomposites MAC.

## 1.3 Scope of Study

This research involved the development of macrocomposite and investigation of its potential for the treatment of POME. The macrocomposite in this study consist the mixture of lightweight aggregates (pumice rock), zeolite, activated carbon and Ordinary Portland Cement (OPC), which act as a binder. The mixture of these components produced an adsorbent that is promising for wastewater treatment. Durability of these macrocomposites were tested by physical and chemical test which

includes heat and pressure alongside with introduction to acid and alkaline conditions. The developed macrocomposites were then tested for its applicability as new treatment system of POME.

Their effectiveness was analyzed through observation of color and COD removal, including pH analysis and ammoniacal nitrogen content. The adsorption efficiency of all macrocomposites were compared too, verified by kinetics and isotherm adsorption model equations. Finally, the formation of biofilm coated on the macrocomposites observed morphologically by SEM. and its total mass of dry biofilm weight was calculated to understand the correlation between biofilm development and bioremediation efficiency.

#### **1.4 Significance of Study**

Adsorption technique is a widely used method in treating POME. A number of studies opted this method in industrial use as an adsorptive immobilization systems such as the biomass support particle (BSP). BSP can be defined as a support material for immobilization and attachment for microorganisms' growth (Albrehtsen *et al.*, 1996; Martins *et al.*, 2013). This method are commonly used in wastewater treatment (Sokol and Woldeyes, 2011) such as in treating starch wastewater (Rajasimman and Karthikeyan, 2007), petrochemical wastewater (Zaini *et al.*, 2015), azo dyes (Lim *et al.*, 2013) and also livestock waste products (Ciborowski, 2001).

There are few numbers of other methods that have been developed in order to improve wastewater treatment system. It is known that treatment of POME by biofilm treatment system is preferable as biofilm can be used for bioremediation processes in treatment of wastewater (Takriff *et al.*, 2014; Ramadhani *et al.*, 2018) Often, developed biofilm is highly beneficial as the microorganisms adhered within the biofilms aid in removal of heavy metals or organic matter, thus, reducing the contaminants present in the wastewater (Aziz *et al.*, 2016; Rene *et al.*, 2016).

Previous researches combined both physical and biological treatment using bio-film macrocomposites due to its low cost and ease of operations (Muhammad Mubarak, 2012; Mohammad Lazim *et al.*, 2016). By replacing the coarse aggregates with pumice rock, it resolves on the weight and structure of macrocomposite. According to Kumar and Krishnaveni (2016), using pumice rock as a construction material gives one-third lighter lightweight quality compared to the conventional sand and gravel based construction material.

As most treatment system nowadays are not efficient to be applied for long term usage because of the heaviness of the system itself. Thus, some clients opt for a lighter weight material in which pumice rock is one of them. Having the advantages of high porosity and is lighter in weight with incorporation of pumice and both adsorbents zeolite and activated carbon, this macrocomposite served as a dual-purpose that is as an adsorbent and a support material for formation of biofilm in bioremediation treatment of industrial wastewaters

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