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TREATMENT OF PALM OIL MILL EFFLUENT USING SADWICH ULTRAFILTRATION TECHNIQUE

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By

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Nowadays, the treatment of palm oil mill effluent (POME) has become a major critical issue in Malaysia that needs to be highlighted. This is due to the extreme organic matter in POME that needs to be treated properly to tackle the environmental issue. POME is a thick brownish viscous liquid waste with an unpleasant odour, and high in colloidal suspension and organic matter. With the needs to solve the problems in terms of environmental protection, economical viability, and sustainable development, a recent innovative ultrafiltration technique using multiple membranes known as "sandwiches membrane", has been extended for POME treatment. This work was carried out to evaluate the potential of pre-treatment methods (adsorption-settling-surface filtration) prior to ultrafiltration treatment and to investigate the potential of ultrafiltration membrane using single and sandwich configurations to reclaim clear water in the POME. This work is divided into two parts which are: (1) pre-treatment stage prior to ultrafultration membrane and (2) POME treatment using sandwich membranes. For the pre-treatment of POME prior to ultrafiltration there were two stages involved; adsorption with settling treatment and surface filtration. In the first stage, the combined effects for adsorption with settling experimental conditions such as adsorbent dosage, treatment time and stirring speed in one hour settling time were investigated. The performance of the adsorption and settling stage using palm kernel shell-based activated carbon (PKS-AC) was based on the analysis of the suspended solid. The supernatant was then

pipette out and subjected to simple surface filtration, under vacuum through Whatman® qualitative filter paper, Grade 4 (20-25 μm) and finally to Whatman® qualitative filter paper, Grade 3 (6 μm) before proceeding to the ultrafiltration process. For ultrafiltration membrane treatment, dead-end filtration mode was used. Two 5 kDa flat sheets regenerated cellulose (RC) ultrafiltration membranes were sandwiched together in various orientations without any spacer in between. The effect of operating pressure, stirring speed and pH on the permeate flux and qualities namely total solid, dissolved solid, suspended solid, biological oxygen demand (BOD_5), chemical oxygen demand (COD), and turbidity were also investigated. The pollutant concentration percentage reduction for the first stage of pre-treatment using 0.20g/L of PKS-AC at 39.82 rpm stirring speed in 35.94 min and 60 min of sedimentation time of POME showed a significant reduction in term of total solid (67.30%), dissolved solid (47.11%), suspended solid (71.26%), BOD_5 (63.23%), COD (42.38%) and turbidity (63.31%) respectively. In the second stage of pre-treatment, the pollutant concentration was further reduced to 65.36%, 48.75%, 87.57%, 84.5%, 48.9% and 78.9% for total solid, dissolved solid, suspended solid, BOD_5, COD and turbidity respectively. After the pre-treatment stage, the membrane technique was applied with purpose to further reduce the pollutant concentration and reclaiming the crystal clear water that complied with WHO water reuse standard. Result for single membrane was compared to those of sandwich with various configurations. Interestingly, SB-sandwich configuration with bottom membrane in the reverse orientation significantly reduced almost 99% of pollutant in raw POME. The best conditions using SB-sandwich was achieved at pH 8, 600 rpm of stirring speed and by applying 2 bar of operating pressure for 90 min of operating time with 47.24 L/m^2h of permeate flux. Applying these conditions successfully reclaimed clear water with low concentration of total solid (138 mg/L), dissolved solid (123 mg/L), suspended solid (15 mg/L), BOD_5 (90 mg/L), COD (113.3 mg/L) and turbidity (0.8 NTU) from POME. Moreover, by comparing between the single and SB-sandwich membrane, showed some differences in pollutant reduction: total solid (11.45%), dissolve solid (0.28%), suspended solid (53.85%), BOD_5 (60%), COD (14.12%) and turbidity (70.12%). Hence, by applying the combination technique of adsorption-settling-surface filtration (pre-treatment) with sandwich membrane ultrafiltration technique successfully treat POME and reclaim crystal clear water that complied with WHO water reuse standard.

Abstrak tesis yang dikemukakan kepadaSenatUniversiti Putra Malaysia
sebagai memenuhi keperluan untuk ijazah sarjana sains

RAWATAN EFLUEN KILANG MINYAK KELAPA SAWIT MENGGUNAKAN TEKNIK SANDWIC MEMBRAN ULTRATURASAN

Oleh

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Pada masa kini, rawatan efluen kilang minyak kelapa sawit (POME) telah menjadi isu utama dan kritikal di Malaysia. Isu ini telah diketengahkan kerana kandungan organik yang amat tinggi dalam POME yang perlu dirawat dengan betul untuk mengurangkan isu alam sekitar. POME adalah sisa cecair likat berwarna coklat dengan bau yang tidak menyenangkan, dan tinggi di dalam pepejal terampai dan bahan organik. Oleh kerana perlunya untuk menyelesaikan masalah ini dari segi perlindungan alam sekitar, daya maju ekonomi, dan pembangunan mampan, teknik ultraturasan yang inovatif bagi rawatan POME, dengan mengabungkan beberapa membran yang dikenali sebagai "membran sandwic", telah dikaji dengan lebih lanjut. Kerja penyelidikan ini dilakukan untuk menilai potensi teknik pra-rawatan (jerapan-enapan-turasan permukaan) terdahulu sebelum rawatan ultraturasan dan mengkaji potensi membran ultraturasan dengan menggunakan konfigurasi tunggal dan sandwic bagi pulih guna air jernih daripada POME. Kerja penyelidikan ini terbahagi kepada dua bahagian iaitu: (1) peringkat pra-rawatan terdahulu sebelum membrane ultraturasan dan (2) rawatan POME menggunakan sandwic membrane. Terdapat dua peringkat pra-rawatan terdahulu sebelum ultraturasan; rawatan jerapan dengan enapan dan turasan permukaan. Di dalam peringkat pertama, kajian telah dilakukan terhadap kesan gabungan keadaan uji kaji bagi jerapan dengan enapan seperti dos bahan penyerap, masa rawatan dan

kelajuan dengan satu jam pengenapan. Prestasi peringkat jerapan dan enapan menggunakan karbon teraktif berasaskan tempurung buah kelapa sawit (PKS-AC) adalah berdasarkan analisa peratusan pepejal terampai yang dapat dikurangkan. Selepas itu, supernatan di keluarkan menggunakan pipet dan menjalani turasan permukaan yang mudah melalui kertas turasan kualitatif Whatman®, Gred 4 (20-25 μm) dan akhir sekali kertas turasan kualitatif Whatman®, Gred 3 (6 μm) menggunakan vakum Untuk rawatan membran ultraturasan, mod penapisan buntu digunakan. Dua helai ultraturasan bersaiz 5 kDa selulosa berregenerasi selulosa (RC) membran ultraturasan telah diapit bersama-sama dalam pelbagai orientasi tanpa pemisah di antaranya. Kesan tekanan operasi, kelajuan aduk dan pH terhadap fluks dan kualiti air seperti jumlah pepejal, pepejal terlarut, pepejal terampai, keperluan oksigen biologi (BOD_5), keperluan oksigen kimia (COD), dan kekeruhan juga disiasat. Daripada analisis pengoptimuman dengan menggunakan 0.20g/L PKS-AC pada kelajuan 39.82 rpm selama 35.94 min dan 60 min masa pemendapan,peratus kepekatan bahan cemar bagi pra-rawatan POME peringkat pertama menunjukkan penurunan signifikan pada jumlah pepejal (67.30%), larut pepejal (47.11%), pepejal terampai (71.26%) BOD_5 (63,23%), COD (42.38%) dan kekeruhan (63.31%). Kepekatan bahan cemar dapat dikurangkan lagi di dalam pra-rawatan peringkat kedua sebanyak 65.36%, 48.75%, 87.57%, 84.5%, 48.9% dan 78.9% untuk jumlah pepejal, larut pepejal, pepejal terampai, BOD_5, COD dan kekeruhan. Selepas peringkat pra-rawatan, teknik membran telah digunakan dalam tujuan untuk mengurangkan lagi kepekatan bahan pencemar dan pulih air jernih. Menariknya, konfigurasi SB-sandwic dengan susunan konfigurasi membran yang berada di bahagian bawah yang sonsang menunjukkan pengurangan hampir 99% pengurangan bahan pencemar. Kondisi yang terbaik telah dicapai dalam ultraturasan POME dengan menggunakan konfigurasi membran SB-sandwic, pH 8, 600 rpm kelajuan pengadukan dan dengan tekanan 2 bar selama 90 minit bersama 47.24 L/m^2 h fluks telap. Dengan menggunakan kondisi ini, pulih guna air bersih berjaya diperolehi daripada POME dengan kepekatan jumlah pepejal (138 mg/L), larut pepejal (123 mg/L), pepejal terampai (15 mg/L) BOD_5 (90 mg/L), COD (113.3 mg/L) dan kekeruhan (0.8 NTU) yang rendah. Tambahan pula, perbandingan antara membran tunggal dan SB-sandwic menunjukkan perbezaan penurunan bahan pencemar: jumlah pepejal (11.45%), larut pepejal (0.28%), pepejal terampai (53.85%) BOD_5 (60%), COD (14.12%) dan kekeruhan (70.12%). Oleh yang demikian, dengan menggunakan teknik gabungan jerapan-enapan-turasan permukaan (pra-rawatan) dengan teknik ultraturasan membran sandwic boleh merawat POME dan memperoleh air guna semula yang jernih yang memenuhi syarat standard WHO bagi air guna semula berjaya diperolehi dari POME

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I certify that a Thesis Examination Committee has met on 29 May 2015 to conduct the final examination of (student's name)on her thesis entitled "Treatment of Palm Oil Mill Effluent using Sandwich Ultrafiltration Technique" in accordance with the Universities and University Colleges Act 1971 and the Constitution of the Universiti Putra Malaysia [P.U.(A) 106] 15 March 1998. The Committee recommends that the student be awarded the Master of Science.

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LIST OF ABBREVIATIONS

POME	Palm oil mill effluent
EFB	Empty fruit bunches
FFB	Fresh fruit bunches
PKS-AC	Palm kernel shell - based activated biocarbon
ANOVA	Analysis of variance
CAC	Commercial activated carbon
BOD_5	Biological oxygen demand
COD	Chemical oxygen demand
DOE	Department of the environment
PKS	Palm kernel shell
RC	Regenerated cellulose
SEM	Scanning electron microscope
BET	Brunaeur, Emmett, and Teller
BJH	Barret-Joyner-Halenda
FC-CCD	Face centred central composite design
DO	Dissolved oxygen
FAS	Ferrous ammonium sulphate
DOE	Design of experiment

LIST OF NOMECLATURE

CH_4	Methane
VS	Volatile solid
MOs	Microorganism
J	Permeate flux
A_m	Membrane area
dv	Amount of permeate collected
dt	Time taken for permeate flow to be taken
S_{BET}	Surface area
NTU	Nephelometric turbidity units

CHAPTER 1

INTRODUCTION

1.1 Background of study

1.1.1 Palm oil industry in Malaysia

Oil palm has become one of the most important and profitable crops in Malaysia. Malaysia was operated about 425 palm oil mill and became the largest producer and exporter around the world (MPOB, 2012). Furthermore, during the period between 2013 and 2014, the palm oil production is increasing 6,975,292 tonne to 7,494,682 tonne (MPOB, 2014). Hence, the palm oil production is expected to increase based on the current demand for palm oil and oleo chemical industries (Ahmad *et al*., 2011).

Unfortunately, along with the increase of this profitable industry, the productions of wastes resulted from the palm oil processing also increases. There are two types of wastes which are solid and liquid wastes. Solid wastes typically consist of palm kernel shell, mesocarp fruit fibres and empty fruit bunches. The liquid waste generated from the palm oil mill is called palm oil mill effluent (POME).

Typical palm oil processing flow chart can be seen on Figure 1.1. From the waste production view point, the amount of solid palm oil waste available from a mill can be substantial. This consists of empty fruit bunches (EFB), palm kernel shell, mesocarp fibres, and possibly solids from decanters. This biomass normally can be converted into useful product. Along with the palm oil production, a palm oil mill also produces an average of 0.65 tonne of raw POME from every ton of fresh fruit bunches (FFB) processed. Mainly, the most critical waste that needs to be treated seriously is POME due to the high contents of pollutant concentration such as natural organic matter, BOD_5, COD, oil and grease. This is because POME will give big impact to the river and environment with the emission of biogas if it is discharge without proper treatment.

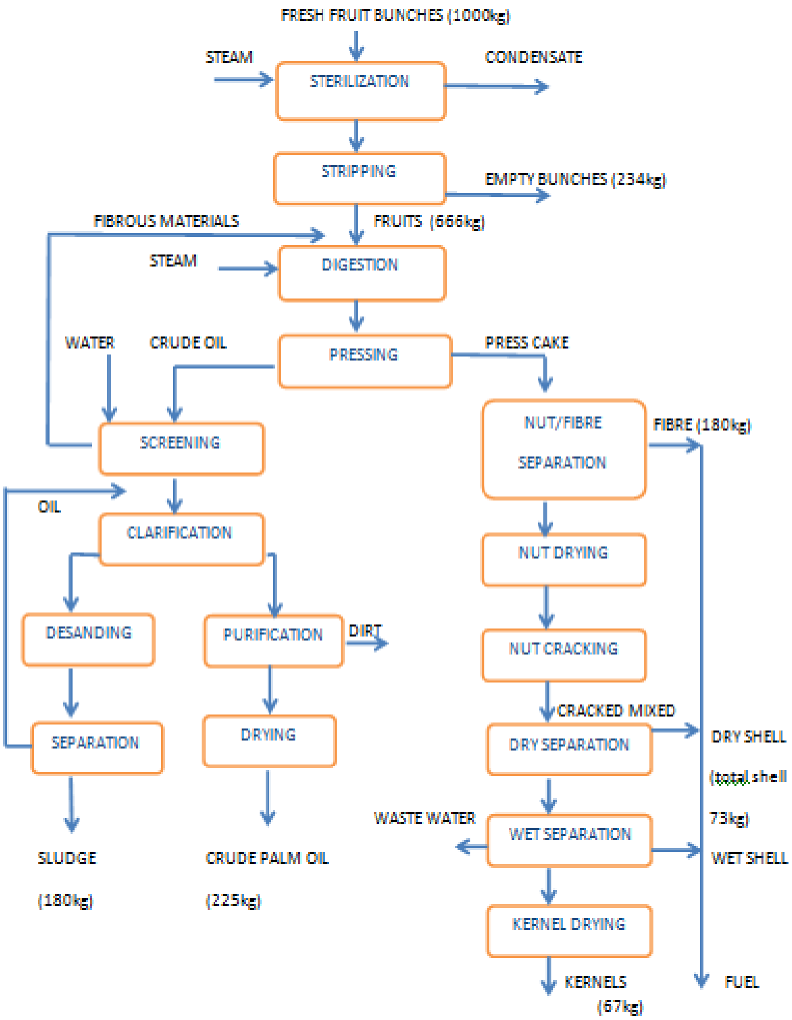

Figure 1.1: Palm oil processing flow chart (Sivasothy, 2000).

1.1.2 Palm oil mill effluent (POME)

As a general definition from the words itself, palm oil mill effluent (POME) is a wastewater that produce from the palm oil processing plant. From visual and sensory observation, POME is a thick brownish viscous liquid waste with an unpleasant odour that is high in colloidal suspended matter (Ahmad *et al*., 2009). POME is produced mainly from oil extraction, washing and cleaning processes in the mill and it includes cellulosic material, fat, oil and grease (Agamuthu, 1995; Rupani *et al*., 2010). POME can be classified as non-toxic waste because there were no chemical added during the process. It contains water (95-96%), suspended solids (2-4%) and oil (0.6-0.7%) with pH 4.0 – 4.5 (Foo and Hameed, 2010). POME also has a high organic content, namely oil and fatty acids (Baharuddin *et al.*, 2010). If POME is discharged without a proper treatment apply, waterways will be polluted due to its high BOD_5 (25,000 mg/L), COD (53,630 mg/L), oil and grease content (8,370 mg/L) and suspended solid content (19,020 mg/L) (Ma 1995; Wu *et al.*, 2007). In addition, as reported by Department of Environmental (DOE), Malaysia account about 10% of heavily polluted or dead river, 63% are polluted and only 27% are clean (Fuadi *et al.*, 2012). For this reason, the palm oil industry has a strong responsibility to face the problem in terms of environmental protection, economical viability, and sustainable development.

In order to implement the strict regulations of the Environmental Quality (Prescribed Premises) (Crude Palm Oil) Order, 1997 and the Environment Quality (Prescribed Premises) (Crude Palm Oil) Regulations, 1997, POME needs to be treated effectively and in a proper way. Moreover because of the huge capacity in this sector, it is critical to treat palm oil mill effluent (POME) to an acceptable level (Table 1.1) before discharging it. If POME did not being treat accordingly, it give a big impact and cause severe pollution of waterways due to oxygen depletion and other reflected effect. This is due to the presence of unrecovered oil which causes the raw or partially treated POME to have an extremely high content of degradable organic matter.

Table 1.1: Characteristics of POME and its respective standard discharge limit by the Malaysian Department of the Environment (Ahmad, 2003; Hanif, 1994).

Parameter	Standard limit, mg/L
pH	5-9
Oil and grease	50
BOD_5	100
COD	-
Total solids	-
Suspended solids	400
Total nitrogen	150

1.1.3 POME treatment

Nowadays, there is several innovative treatment technologies have been developed to treat POME wastewater. It is important to consider some of the factors before applying the technology such as available space for construction for treatment facilities, ability of process equipment, limitation of waste disposal, desired final water quality and cost of capital and operating (Fuadi *et al.*,2012). There are three treatments technologies that can be used to treat POME wastewater which are biological, physical and chemical treatment. Biological treatment is normally involved with biodegradation process that involves adsorption by microbial biomass and bioremediation systems (Ahmaruzzaman, 2008). This is because many microorganisms are able to accumulate and degrade different pollutant. Anaerobic digestion, ponding system, activated sludge process and aerated lagoon fall under biological treatment. Physical method of wastewater treatment is solid-liquid separations technique of which filtrations is the main role that can use various media as medium of filtration (Cheremisinoff, 2002).

Some of the examples of physical treatment are granular-medium filtration, sedimentation, membrane separation, and screening. Chemical method relies upon the chemical interactions of the components that need to be demolished from wastewater. The use of the chemical is as either aid in the contaminant separation from wastewater or assists in the destruction or neutralization of harmful effects that associated with contaminants. Some of the chemical method uses are chemical precipitation, adsorption, disinfection, coagulation and others.

1.2 Problem statement and research gap

Over the years, most of the palm oil mills apply biological treatment such as anaerobic or facultative digestion which is believed can overcome the waterways pollution problem (Quah *et al*., 1982; Ahmad *et al.*, 2003a). Other than that, there are also some mills that utilized zero waste technologies and the treated POME is applied to land (Shah and Kaur, 2004). Conversely, there are several problems and disadvantages arise from the current treatment. In order to ensure that a conductive environment is maintained for microorganisms in which to survive, the biological treatment system requires proper maintenance and monitoring as the bacteria is very sensitive to the changes in the environment. In addition, attentions from skilled operators as well as the commitment from the management are also required. Other than that, biological treatment also generates vast amount of biogas. This biogas consists of methane, carbon dioxide and trace amounts of hydrogen sulphide. These gases are corrosive and odorous (Ahmad *et al*., 2003a). Normally these issues are always ignored by the mill owners because the effluent treatment is often viewed as a burden and given the lowest priority. Due to the problems and disadvantages of biological treatment, it is suggested and believed that membrane separation technology will be able to treat POME in a more beneficial ways.

However, palm oil industries always find the membrane technology as the expensive treatment due to the high cost of installation set up and mantainence. A lot of study has been done on applying the membrane technology for POME treatment but still it cannot convince the palm oil management due to this high cost problem.

Currently, the ultrafiltration of POME showed a great potential in reclaim a better quality of treated POME. However, the industries face the problem in obtaining the clear crystal water from ultrafiltration of POME without using reverse osmosis membrane which is more expensive. This is mainly due to an uneven and rather broad pore size distribution of the membranes which often limit the rejection and the efficiency of the separation. Although some commercial membranes today have a tighter pore size distribution, it has been found by other reseacher that sandwich membrane can improve the rejection characteristics of ultrafiltration membranes. Based on the critical review of previous work, the multilayer or sandwiches of membrane was commonly studied for the protein separation and there was no study applying this technology for POME treatment yet. The potential of membrane layers or sandwich membrane for ultrafiltration of POME has not been explored yet. Therefore, an interest arises to investigate the potential of sandwich membranes of various orientations and the effect of these configurations on the filtration behavior of particles solid in POME. These particles solid in POME which will contribute in determine the other pollutant concentration in POME. Consequently, two flat membranes are sandwiched together in various configurations in a single stirred cell ultrafiltration device and the effects of various sandwiches on the ultrafiltration of POME are examined in this study. Moreover, the high cost of maintenance can be solved by applying the adsorption-settling-surface filtration treatment as a pre-treatment to reduce fouling effect during ultratfiltration process.

1.3 Motivation

The use of membrane separation technology is normally coupled with pre-treatment. There are several advantages to use membrane separation technology. It can be applied across a wide range of industries; the quality of the treated water is more consistent regardless of the influent variations; it can be used in a process to allow the recycling of selected waste streams within a plant; highly skilled operators would not be required when the plant can be fully automated (Cheryan and Rajagopalan, 1998); and the water reclaimed from this treatment could be reused in the mill. Thus, the primary advantages lie in the reduction of the cost for the water supply and its further treatment as well as in the effective elimination of the pollutant from the POME. Furthermore, if the methods of treatment are easy to operate, this method will then reduce the cost of operation (Wang *et al.*, 2012).

Generally, membrane cannot be used directly to treat oily wastewater like POME. This is because membrane is sensitive and costly component of the treatment process. In order to reduce the cost of maintenance later on, pre-treatment should be applied to POME and membrane life can be extended also long-term system performance can be improved (Water Environment Federation, 2006). Pre-treatment was applied to avoid membrane fouling which can reduce membrane performance. There are several pre-treatment can be applied such as flow equalization, fine screens, strainers, chlorination, chemical coagulation, adsorption and others. These pre-treatment is include based on influent water quality, filtrate quality requirements, and the membrane used (Water Environment Federation, 2006). It is suggested to apply palm kernel shell-based activated carbon (PKS-AC) adsorption-settling-surface filtration as pre-treatment.

1.4 Objectives

The objectives of the study are as follows:

1. To evaluate the potential of pre-treatment methods (adsorption-settling-surface filtration) prior to ultrafiltration treatment
2. To investigate the potential of ultrafiltration membrane using single and sandwich configurations to reclaim clear water in the POME.

1.5 Summary of thesis

This study describes the treatment process in treating the raw POME. The raw POME was treated by coupling together ultrafiltration membrane treatment with pre-treatment method involves adsorption-settling-surface filtration treatment. This study also describes sandwich ultrafiltration membranes and discusses the potential of sandwich membrane configuration to enhance the performance of ultrafiltration module to effectively treating the POME.

The first part of the study involved pre-treatment by using adsorption-settling-surface filtration which initially applied before ultrafiltration of POME, to reduce the sludge and particles in POME. This pre-treatment was performed prior to reduce and avoid fouling effect on membrane during ultrafiltration membrane process.

The second part of this study proposed the use of ultrafiltration membrane in POME treatment. After pre-treatment stage, pre-treated POME was subjected to ultrafiltration membrane treatment. The ultrafiltration experimental was conducted using single and sandwich membrane (two flat ultrafiltration membranes were stack together without any spacer). The performance and behavior of these configurations were compared. The performance was in term of permeate flux behaviour and reduction in dissolved solid and turbidity.

1.6 Significant and limitation of the study

Current treatments applied by the palm oil mill industries to treat POME have some disadvantages in term of economy viability, management facilities and maintenance, large treatment area required and time consuming. Moreover, almost all the method applied by the palm oil mill industries manage to reduce the pollutants in POME but failed to improve the color (clear water). Thus it is necessary to suggest and investigate a more practical method in treating POME.

This study will provide knowledge concerning the suggested method applied which is sandwich membrane configuration couple with PKS-AC adsorption-settling-surface filtration as pre-treatment. It is believed the findings from the suggested method will improve the colour of treated POME to clear water in line with significant reduction on the pollutant in the POME. The findings in this study can also provide a fundamental knowledge for the sandwich membrane configuration in term of improving the pore size distribution arrangement in the membrane.

As a result of this study, knowledge of the ultrafiltration of POME using sandwich membrane couple with PKS-AC adsorption-settling-surface filtration (pre-treatment) can be obtained. Suitable operating conditions for both pre-treatment and sandwich membrane treatment also can be obtained to achieve high output with the better quality of treated POME

Unfortunately, there were some limitations encounters in this study. The problems were based on time constraint, condition of the lab surrounding and experimental rig (stirred cell) used in this study. In addition, the temperature of the solution cannot be changed due to the ultrafiltration cell was not suitably set up for temperature control. Due to these limitations, there was some ranges of variables were not being included in this study.

1.7 Outline of thesis

Chapter 1 – Introduction:

This chapter contains a short description of the subject that initiated this research, background on POME characteristics and treatment, motivation, objectives, summary and outline of the thesis.

Chapter 2 – Literature review:

This chapter reviews the overall background of the POME treatment, including the concepts and technology that form a core of the thesis, and give a brief overview of the previous work. This overview provides a general discussion of related research.

Chapter 3 – Materials and methods

This chapter presents the material and methods relevant to adsorption-settling-surface filtration treatment and ultrafiltration membrane treatment. This includes the experimental details of this proposed technique and procedures use in the thesis.

Chapter 4 - Characteristics of POME and pre-treatment stage prior to ultrafiltration

This chapter presented the result and discussions on the pre-treatment method (adsorption-settling-surface filtration treatment).

Chapter 5 – POME treatment using sandwich membranes

The objective in this section is to evaluate and compare the effectiveness of single and sandwich membrane in ultrafiltration of POME.

Chapter 6 – Conclusion and recommendations

This chapter contains concluding remarks and recommendation for future work.

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