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EVALUATION OF PERFORMANCE AND FOULING RESISTANCE OF SANDWICH ULTRAFILTRATION FOR TREATMENT OF FINAL DISCHARGED PALM OIL MILL EFFLUENT

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By

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Chairman : Khairul Faezah Md Yunos, PhD
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Oil palm is the important agriculture industry in Malaysia which consumed hundreds tonnes of water for the proses and 50% of which ends up as effluent. However, water can be sustained and conserved by treating final discharged and recycle back to the plant. Membrane ultrafiltration has been proven a reliable tool in treating wastewater, therefore, it is a promising green technology to treat final discharged from palm oil mill. Thus, this work is carried out to evaluate the effectiveness of ultrafiltration in reclaiming water from final discharged palm oil mill effluent (POME) as well as to investigate the fouling of membrane. Two types of membrane used were polyethersulfone (PES) and regenerated cellulose (RC) of 5kDa and 10 kDa. The first part of this study is to evaluate the effect of parameters that are: pressure (0.5 bar, 1.0 bar, 1.5 bar, 2.0 bar), stirring speed (0 rpm, 400 rpm, 600 rpm, 800 rpm) and pH value (6, 7, 8, 10) on ultrafiltration treatment and fouling resistance. The appropriate filtration condition was recognized and the permeate was further analysed for COD, BOD_5 and suspended solid. Based on the parametric study, selected condition was fixed for the second part to evaluate the effect of sandwich configuration to further improve the final discharged quality and to analyse fouling resistance. Cake layer resistance was found to be dominant for all membranes tested. Two flat membranes were sandwiched together with both effective layer facing up (SS) or the effective layer of the bottom membrane facing down (SB) The results showed of the best permeate quality was achieved with 5 kDa PES membrane at pressure 1.0 bar, 600 rpm and pH 8: the reduction of COD, $BOD_{5,}$ turbidity, and total dissolved solid were 67.3%, 72.47%, 94.2% and 40% respectively. For sandwich membrane, interestingly, SS-sandwich showed the best permeate

quality with pollutant reduction up to 80-90% (PES 5kDa) compared to 60-70% for single membrane. The quality of permeate from SS-sandwich membrane of 5 kDa was beyond reuse standard and approaching drinking water standard for TSS, TDS and turbidity. Therefore it can be concluded that, with the appropriate arrangement of sandwich membrane and operating condition, water reuse which successfully complies with World Health Organization (WHO) standard can be reclaimed from POME using UF technique

Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia sebagai memenuhi keperluan untuk ijazah Master Sains

EVALUASI PRESTASI MEMBRANE DAN RINTANGAN KOTORAN ULTRAFILTRASI SANDWIC SEMASA RAWATAN AIR DARI KILANG MINYAK KELAPA SAWIT

Oleh

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Minyak kelapa sawit adalah industri terpenting di Malaysia yang menggunakan beratus tan air semasa pemprosesan dan 50% of daripada air yang digunakan menjadi efluen. Membran ultrafiltrasi telah dibuktikan sebagai alat yang dipercayai dalam rawatan air buangan, oleh itu, ia ada teknologi mesra alam yang berpotensi untuk merawat efluen akhir daripada kilang minyak kelapa sawit. Oleh itu, kajian ini dijalankan untuk menilai keberkesanan membran ultrafiltrasi untuk menebus guna air daripada efluen akhir di samping untuk menyiasat rintangan kotoran membran. Dua jenis membran yang digunakan iaitu polyethersulfone (PES) dan regenerated cellulose (RC) bersaiz 5kDa dan 10 kDa. Peringkat pertama kajian ini adalah untuk menilai kesan parameter iaitu: tekanan (0.5 bar, 1.0 bar, 1.5 bar, 2.0 bar), kelajuan aduk (0 rpm, 400 rpm, 600 rpm, 800 rpm) dan pH (6, 7, 8, 10) terhadap rawatan ultrafiltrasi dan rintangan kotoran. Kondisi filtrasi yang wajar di kenal pasti melalui kaedah kajian parameter dan permeat dianalisa lagi untuk COD, BOD_5, dan pepejal terampai. Berdasarkan kajian parametrik, kondisi wajar ditetapkan bagi bahagian kedua untuk mengevaluasi kesan konfigurasi sandwic untuk menambah baik kualiti efluen dan untuk menganalisa rintangan kotoran. Rintangan kotoran kek layer adalah dominan bagi kesemua membran yang diuji. Dua membrane disandwickan bersama dengan kedua-dua lapisan efektif mengadap keatas (SS) atau lapisan efektif membran bawah mengadap ke bawah (SB). Hasil kajian menunjukkan kualiti permeat terbaik berjaya diperolehi menggunakan membrane ultrafiltrasi bersaiz 5 kDa pada kondisi 1.0 bar, 600 rpm dan nilai pH 8 dengan pengurangan COD, BOD_5, kekeruhan dan pepejal larut sebanyak 67.3%, 72.47%, 94.2%, dan 40% masing-masing. Bagi membran sandwich, SS-sandwic menunjukkan kualiti permeat terbaik dengan pengurangan bahan pencemar sebanyak 80-90% (PES 5kDa) dibandingakn dengan membran

tunggal iaitu sebanyak 60-70% sahaja. Kualiti permeat yang berjaya mencapai melebihi piawai penggunaan semula air dan menghampiri kualiti piawai air minuman bagi pepejal terampai, pepejal larut dan kekeruhan. Oleh itu, sebagai konklusinya, dengan susunan sandwic dan kondisi operasi yang wajar dan bersesuaian, penggunaan semula air berjaya memenuhi syarat piawai yang ditetapkan oleh organisasi kesihatan dunia (WHO)

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This thesis was submitted to the Senate of Universiti Putra Malaysia and has been accepted as fulfilment of the requirement for the degree of Master of Science. The members of the Supervisory Committee were as follows:

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LIST OF ABBREVIATIONS

AFM	Atomic Force Microscopy
BOD	Biological Oxygen Demand
CA	Cellulose Acetate
CFV	Cross Flow Velocity
COD	Chemical Oxygen Demand
CP	Concentration Polarization
CSLM	Confocal Scanning Laser Microscopy
DO	Dissolved Oxygen
DOE	Department of Environment
FESEM	Field Emission Scanning Electron Microscopy
MF	Microfiltration
MPOB	Malaysian Palm Oil Board
MPOC	Malaysian Palm Oil Council
MWCO	Molecular weight cut off
NF	Nanofiltration
NOM	Natural Organic Matter
PAC	Palm Kernell Shell-Activated Carbon
PAN	Polyacrylonitrile
PES	Polyethersulfone
POME	Palm oil mill effluent
PS	Polysulfone
PTFE	Polytetrafluoroethylene
RC	Regenerated Cellulose

RO	Reverse Osmosis
RO	Reverse Osmosis
TDS	Total Dissolved Solid
TMP	Transmembrane Pressure
TOC	Total Organic Carbon

LIST OF NOMECLATURES

CH_4	Methane
J	Flux
R_m	Membrane Resistance
R_c	Cake Resistance
R_p	Pore Resistance
NTU	Turbidity

CHAPTER 1

INTRODUCTION

This chapter contains a brief description on ultrafiltration membrane, fouling development of membrane and an overview of application of ultrafiltration membrane in treating final discharged from palm oil mill effluent (POME). Besides, problem statement, objectives and scope of this research were included in this chapter.

1.1 Ultrafiltration Membrane

Membrane is a material which one type of substance can pass more readily than other, this permits the separation of components from water. The implementation of membrane technology in reclamation and reuse of wastewater from industrial plant has grown steadily with rapid growth of human population along with fast industries development caused water scarcity problem become more severe throughout the year (Choi et al., 2005). The main advantage of membrane technology are: compact process and plant, separation based on size exclusion, water quality independent of feed water quality, constant production, easy automation and absence of bacterial regrowth, (Xia et al., 2005). Ultrafiltration (UF) membrane processes appear to be the most suitable membrane for wastewater treatment because they produced high fluxes at relatively low pressure. UF is a low-pressure membrane with pore size diameter between 1 – 100 nm (Koros et al., 1996), where its separation were based on pore sizes. UF can retained bacteria and viruses, which allows its application for water disinfection, producing drinkable water as the permeate of the process (Arnal et al., 2004).

1.2 Membrane Fouling

In spite of all the advantages of UF, the major drawback of membrane application is membrane fouling due to the deposition and accumulation of particulate matter, microorganisms or colloids better known as foulant particle on a membrane surface and/or inside membrane pores (Kraume and Meng, 2012). Membrane fouling causes an increase in membrane cleaning cost, process down time and also shorten membrane lifespan due to frequency and harshness of membrane cleaning condition (Wu *et al*., 2007).

Previous researchers have carried out great efforts in understanding membrane filtration of bio-products. Tracey and Davis (1994) as well as Bowen, Mohammad and Hilal (1997) reported that membrane pore size and protein concentration play an important role in membrane fouling in microfiltration of pure bovine serum albumin (BSA). Membrane fouling types changed from internal blocking into cake formation after a period of filtration. Huisman, Prádanos and Hernández (2000) conducted BSA cross-flow ultrafiltration using membranes with different molecular weight cut-off. When a membrane with a small molecular weight cut off (MWCO < 30) is used, membrane fouling at the early stage was determined by the interactions between protein and membrane, whereas in the later period it was dependent on interactions between protein molecules. The membrane plays a trivial effect on the membrane fouling when a large MWCO was used. Blatt *et al*. (1970) claimed that the membrane fouling in protein ultrafiltration depends strongly on the transmembrane pressure. The concentration polarization layer presented an important role under low pressure. At high pressure, the solutes molecules would be deposited onto the membrane surface or adsorbed into the wall surface in a membrane pore.

1.3 Treatment of POME using membrane

Previous studies of UF were mostly focused on filtration to remove natural organic matter (NOM) (Zularisam et al., 2007), such as protein (bovine serum albumin) BSA, humic acid and sodium alginate which parts of NOMs that present in wastewater, river, and seawater (Lee et al., 2005), much less attention has been devoted to elucidate fouling during filtration of final discharged of palm oil mill effluent (POME). Along with the increasing number and scale for the applications of UF, studies of fouling development becomes an important issue as fouling caused increased of maintenance cost if this technology were implemented for pilot operation.

The palm oil industry in Malaysia grows rapidly as our country has become the world's top three producer and exporter of palm oil. Being one of the biggest producers and exporters of palm oil and palm oil products, Malaysia has an important role in fulfilling the growing global need for sustainable oils and fats (Malaysian Palm Oil Board [MPOB], 2014). Unfortunately, along with the increase of this profitable industry, the generation of palm oil mill effluent also increased. According to MPOB (2017), in the year 2014 to 2015 the production of palm oil increase from 19,669,206 tonnes to 19,961,581 tonnes, which means, more than millions of tonnes of palm oil mill effluent has been generated throughout the year. If the effluent is left untreated, it can cause a serious environmental problem. Therefore, it is urgent to find a compromising way to control this problem. The conventional treatment system, anaerobic and aerobic systems are not efficient enough to treat the effluent. The final discharge usually does not comply with the standard discharge limit of the Department of Environment (DOE).

Studies have been done to investigate the effectiveness of membranes for treatment of POME. Wu *et al*. (2007) who studied the effect of pressure on membrane fouling. It was reported that at 0.8 MPa fouling increased up to a maximum value of 85.8% but simultaneously enabled the recovery of protein and carbohydrate in POME up to 61.4% and 76.4%, respectively. Ahmad *et al*. (2003) used coagulation and flocculation as pre-treatment for POME, before passing through RO and UF membrane. Results showed that the treatment system has a huge potential for producing boiler feed water that can be recycled back to the plant. Hence, UF may work as a reliable tool for treatment of POME. Azmi and Md Yunos (2014) combined adsorption treatment using palm kernel shell-based activated carbon as pre-treatment with ultrafiltration membrane. These authors reported significant reduction of pollutant elements up to 90% at optimum conditions.

However, performance of membrane can be enhanced and fouling can be mitigated by modifying membrane configuration. One of the technique of membrane modification is by stacking two membranes together without any spacer with different configurations, also known as sandwich membrane (Md Yunos and Field, 2008). The purpose of sandwich membrane is to compensate the imperfection of the pore size distribution of available commercial membranes. Feins and Sirkar (2005) successfully improved the selectivity of membranes by stacking 2 to 3 flat UF membranes. Md Yunos and Field (2008) enhanced the efficiency of ultrafiltration membrane during fractionation of protein. Azmi and Md Yunos (2014) successfully improved the quality of permeate from POME using sandwich ultrafiltration technique. They reported that with reverse membrane orientation, the pollutant significantly reduced by almost 99%. However, very little appreciation for the potential of sandwich ultrafiltration either in protein separation or waste treatment has been shown. Therefore, this study focused on the effect of operating parameters on the performance and fouling formation of single membrane and sandwich membrane during treatment of final discharge from (POME).

1.4 Problem Statement

Water scarcity problems have been more severe around the world. The rapid growth of population and industries as well as intense regulations caused the demand for freshwater becomes tripled and continue to rise over the year. Billion tonnes of freshwater are being used by industries during the production process. One of the solutions for addressing this issue by reclamation and reuse of wastewater from municipalities and industrial plants.

The industry that contribute to the most of the effluent is palm oil industry as Malaysian palm oil industry is growing rapidly and become a very important agriculture-based country. However, this important economic activity unfortunately generates an enormous amount of liquid effluent or palm oil mill effluent (POME). It is estimated for 1 tonne of crude palm oil produced, almost 5-7 tonnes of water were used during wet process of palm oil mill and 50% of the water end up as POME (Ahmad et al., 2003). Nevertheless, water can be sustained and conserved by reclaiming water from final discharge of POME using membrane technology as tertiary treatment. However, palm oil industries always find the membrane technology as the expensive treatment due to high cost of maintenance due to fouling phenomenon. Therefore, it is important to understand the mechanism of fouling in order to control and reduced membrane fouling.

Currently, filtration of POME using UF membrane showed great potential in reclaim better quality of treated POME. However, the industries face the problem in obtaining crystal clear water without using reverse osmosis membrane which is more expensive. This is due to pore size distribution of the membranes often limit the rejection and efficiency of the separation. Although, some commercial membrane today have tighter distribution, it has been found by other researcher that sandwich membrane can improved the rejection characteristics of ultrafiltration membrane.

Sandwich membrane are commonly studied to enhance the separation of different types of proteins such as myglobin, lysozyme and BSA as different arrangement amplified the separation of different types of protein (Field et al., 2009). Currently, application of sandwich membrane in treatment of pre-treated POME successfully achieved 60% reduction of pollutant. However, in the study, fouling mechanism of membrane occur during treatment was not elucidated. Thus, this project was proposed to study effect of operating parameters on UF membrane performance and fouling resistance and effectiveness of sandwich membrane in reclaiming water from final discharged and how it effect fouling.

1.5 Objectives of the Research

The objectives of this study are as follows:

i) To study the effect of pressure (0.5 bar, 1.0 bar, 1.5 bar and 2.0 bar), stirred condition (0 rpm, 400 rpm, 600 rpm and 800 rpm) and pH (6,7,8 and 10) on permeate quality and fouling resistance of single membrane and sandwich membrane.
ii) To evaluate the effectiveness of sandwich membrane in improving the quality of permeate and fouling analysis.

1.6 Scope of Research

The effect of pressure (0.5 bar, 1.0 bar and 2.0 bar), stirred condition (0 rpm, 400 rpm, 600 rpm and 800 rpm) and pH (6, 7, 8 and 10) on permeate quality (total dissolved solid (TDS) and turbidity) and fouling resistance of membrane. Dissolved solid concentration in POME is related to the value of BOD_5 and COD. If dissolved solid in POME reducing, the concentration of BOD_5 and COD also reduced (Azmi and Md Yunos, 2015). Turbidity has the linear relationship with the suspended matter in the solution. These suspended mater create turbidity and impact color to the water (Boyd, 2000). As for fouling study, fouling intensity and fouling resistance were investigated in this research. Fouling intensity is the study of how intense the fouling of membrane after filtration (Mohammad et al., 2009) while fouling resistance exhibited the fouling occurred during filtration run.

The proper condition was selected for each parameter based on acceptable value of TDS and turbidity and fouling. At this condition, the permeate were further analyse with BOD_5, COD and suspended solid. These values were compared with standard of effluent discharged limit standard (B) stated in Malaysian Department of Environment (DOE),2010, and EPA Guidelines for water reused standard as well as Drinking Water Quality Standard from DOE (2010), which can be seen in Table 4.4.

For second stage of this experiment, the condition was fixed based on first experiment. Sandwich membrane were done by stacking two flat sheet membrane together without any spacer in between. The configuration of sandwich membrane was varied by adjusting the arrangement of the bottom membrane only. The configuration were called SS-Sandwich and SB-Sandwich. S term is referring to skin layer while B referring to the support layer of the membrane. The illustration of sandwich membrane can be seen in Figure 2.7 and Figure 3.6.

1.7 Thesis Outline

Chapter 1 – Introduction

This chapter gives an overview on the background of this research. A brief explanation on ultrafiltration membrane, fouling mechanism during treatment and a brief of palm oil industry in Malaysia. The objectives and research scope also included in this chapter.

Chapter 2 – Literature review

This chapter review previous research of palm oil treatment, application of membrane in palm oil industry and fouling mechanism of ultrafiltration. This overview provides a general discussion of related research.

Chapter 3 – Materials and Method

This chapter present the materials and method includes origin of the sample, types of membrane used and set-up of the experiment. . This includes the experimental details of the proposed technique and procedure used in the thesis.

Chapter 4 - Result and discussion

This chapter present result and discussion on the effect of operating parameters on membrane performance and fouling formation. Also discussion on the effect of sandwich configuration on membrane performance.

Chapter 5- Conclusion

This chapter contains concluding remarks and recommendation for future works.

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