UNIVERSITI PUTRA MALAYSIA

PERFORMANCE OF ULTRAFILTRATION MEMBRANES FOR TREATMENT OF PALM OIL MILL EFFLUENT

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By

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Chairman : Professor Luqman Chuah Abdullah, PhD
Faculty : Engineering

The production of palm oil is the core of Malaysia economy and known globally. However, palm oil liquid waste known as palm oil mill effluent (POME) has been identified as one of the major sources of pollution in water stream throughout Malaysia. This is because POME contains high biochemical oxygen demand (BOD), high chemical oxygen demand (COD), high salt content, high suspended solid and low pH. High demand of crude palm oil has contributed to the high generation of POME and led to serious environmental issue in rivers all over the country. Based on this figure, a proper treatment method is very important in order to treat POME efficiently before releasing to the water source. Membrane separation processes are today have been adopted in many industries as its ability to treat wastewater and this includes POME. Between microfiltration (MF), nanofiltration (NF) and reverse osmosis (RO), ultrafiltration membrane (UF) was selected for this research work. UF is a cross-flow membrane process which capable of removing all microbiological species, viruses, humic materials, better rejection than MF, more applicable than NF and RO in treating POME. In this work, POME was treated with four types of ultrafiltration (UF) membranes namely EM006 (6kDa), ES625 (25kDa), FP100 (100kDa) and FP200 (200kDa). The UF membranes were studied and compared in terms of permeate quality and fouling analysis. It was observed that all UF membranes were able to treat POME and shows significant reduction in COD, total suspended solids (TSS), total dissolved solids (TDS), color, pH, and turbidity. In the comparison of the EM006, ES625, FP100 and FP200 membranes, EM006 membranes yields the highest result in terms of the foulant removal compare to other membranes with removal value of COD (82.26%), TS (95.63%), TSS (95.23%), turbidity (96.65%) and color (68.87%). Study on fouling mechanism shows that cake filtration dominates the fouling activities on surface of UF membrane, compare to standard blocking, intermediate blocking and complete blocking. The evaluation of the four membranes found that ES625 membrane are the most stable membrane used for POME treatment with a highest coefficient value of R^2 (0.9981) at an applied pressure of 100kPa. This is supported by the direct observation on membrane surface through SEM result and particles analysis. The relationships between multifactor in the treatment of POME using UF

membranes were also evaluated by RSM. The results shows that with the application of RSM, it was able to show the correlation between measured and predicted of experimental results.

The post-treatment of UF membranes after the cleaning processes were done by the coagulation methods. The characteristic of effluent after membrane washing (EMW) still shows high pollutant contents and further treatment with coagulation and flocculation with alum and Organo-floc was able to remove the pollutant to meet with limit sets for standard regulatory of POME. The findings result shows both alum and organo-floc were able to reduce the COD, TSS, color and turbidity to the standards limit sets by DOE. The best removal achieved using coagulation and flocculation method were 93% of COD, 97% of TSS and 94% of color for alum, and 96% of COD, 94% of TSS and 97% of color for organo-floc. The optimizations of the post-treatment were later performed by response surface methodology (RSM).

Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia sebagai memenuhi keperluan untuk Ijazah Doktor Falsafah

KEUPAYAAN ULTRAFILTRASI MEMBRAN DI DALAM RAWATAN AIR KUMBAHAN KILANG KELAPA SAWIT

Oleh

MOHD AZWAN BIN AHMAD

Januari 2017

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Hasil tanaman kelapa sawit merupakan tunjang ekonomi di Malaysia dan terkenal di serata dunia. Sungguhpun begitu, hasilan air sisa kelapa sawit atau lebih dikenali sebagai air kumbahan kilang kelapa sawit (POME) telah dikenal pasti sebagai punca utama yang menyebabkan pencemaran air di sungai-sungai di Malaysia. Ini kerana kandungan POME yang tinggi dengan permintaan oksigen biokimia (BOD), permintaan oksigen kimia (COD), kandungan garam, sisa pepejal serta pH yang rendah. Permintaan yang tinggi terhadap kelapa sawit mentah telah menghasilkan jumlah POME yang sama banyak dan ini telah memudaratkan keadaan sungai di serata negeri. Hasil gambaran ini menunjukkan betapa pentingnya kaedah rawatan yang berkesan terhadap POME sebelum ianya dilepaskan ke punca-punca air. Proses rawatan air kumbahan menggunakan aplikasi membrane telah banyak dilaksanakan di dalam pelbagai industri dan ini termasuk juga POME. Di antara mikrofiltrasi, nanofiltrasi, dan osmosis berbalik, ultrafiltrasi telah dipilih untuk tujuan kerja projek ini. Ultrafiltrasi ada proses aliran silang di dalam membrane di mana ia mampu untuk mengeluarkan segala spesis mikrobiologi, virus dan juga bahan-bahan humik, keupayaan yang lebih baik berbanding mikrofiltrasi, nanofiltrasi dan juga osmosis berbalik. Di dalam kajian ini, POME telah dirawat dengan empat jenis ultrafiltrasi (UF) membrane iaitu EM006 (6kDa), ES625 (25kDa), FP100 (100kDa) and FP200 (200kDa). Kesemua membran ini telah dikaji dan dibandingkan berdasarkan keupayaan membran terhadap kualiti hasil resapan dan juga analisis terhadap kecacatan yang telah terjadi. Dari pemerhatian, didapati kesemua membran ini mampu untuk merawat POME dengan menunjukkan pengurangan yang ketara di dalam COD, TSS, TDS, warna, pH dan juga tahap kekeruhan. Perbandingan di antara EM006, ES625, FP100 dan FP200 membran, didapati penyingkiran hasil bendasing adalah yang tertinggi dengan nilai COD (82.26%), TS (95.63%), TSS (95.23%), kekeruhan (96.65%) dan warna (68.87%). Kajian terhadap mekanisma kerosakan didapati lapisan kek mendominasi pada permukaan membrane berbanding kerosakan yang lain. Berdasarkan penilaian terhadap keempat-empat membran, didapati ES625 adalah membran yang paling stabil dengan nilai R^2 0.9981 pada tekanan 100kPa.Ini dibuktikan dengan tinjauan terus terus terhadap membrane melalui keputusan SEM dan

juga PSD. Hubungan di antara pelbagai faktor di dalam rawatan POME menggunakan UF membran juga dimodelkan dengan RSM. Dengan aplikasi RSM, ianya didapati mampu menunjukan hubung kait di antara keputusan ramalan dengan keputusan sebenar hasil kajian eksperimen yang telah dilaksanakan.

Rawatan susulan ke atas UF membran selepas proses pembersihan adalah menggunakan kaedah penjerapan. Terdapat dua bahan penjerap yang digunakan iaitu alum dan organo-floc. Keputusan kajian menunjukkan kedua-dua bahan penjerap mampu untuk mengurangkan kandungan COD, TSS, warna dan kekeruhan selari dengan piawaian yang telah ditetapkan oleh Jabatan Alam Sekitar. Keputusan terbaik melalui kaedah penjerapan dengan bahan penjerap alum adalah COD (93%), TSS (97%) dan warna (94%), manakala keputusan terbaik menggunakan bahan penjerap organo-floc adalah COD (96%), TSS (94%) dan warna (97%). Keupayaan optimum untuk rawatan susulan kemudiannya dilakukan dan di model berpandukan RSM.

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I certify that a Thesis Examination Committee has met on 25 January 2017 to conduct the final examination of Mohd Azwan Ahmad on his thesis entitled "Performance of Ultrafiltration Membranes for Treatment of Palm Oil Mill Effluent" 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 Doctor of Philosophy.

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INTRODUCTION

1.1 Background of the study

Based on the report by Malaysian Palm Oil Council (MPOC), it's noted that Malaysia is one of the largest producers of palm oil and contributed around 62% of world exports (MPOC, 2009). It is also means that palm oil mill effluent (POME) has produced synchronously to the large amount of palm oil production. The raw effluent discharged by a single average-sized palm oil mill of 30 t/h FFB processing capacity generates the pollution equivalent of 300,000 persons (Yejian et. al., 2008). In the year 2008 alone, Malaysia has produced around 44 million tonnes of POME and this value will increase every year due to high demand of crude palm oil. By looking at this figure, POME is identified as one of the largest contributors of pollution in rivers all over the country in Malaysia (Hwang et. al., 1978).

Many treatment and removal techniques have been explored and proposed in order to remove the pollution of POME and one of them are membrane separation technology. Membrane separation was popular technology used in water and wastewater treatment and has been applied in many types of industry. The use of membrane separation technology is recognized as an efficient, economical and reliable technology that exhibits high potential to be applied in POME treatment (Ahmad and Chan, 2009). Separation of fluids by membrane separation processes is primarily dependent on the pore size and pore size distribution of the membrane. The pore size for ultrafiltration (UF) membrane are range of 1 ~ 100 nm. UF is the process of separating small particles and dissolved molecules from solution. The UF application are include the production of pure water, fractionation or concentration steps in the food, pharmaceutical and biotechnological industries, and also treatment of water and wastewater (Van Reis and Zydney, 2001).

However, the obstacles of using membrane treatment process include membrane fouling and decline in permeate flux. Due to that, this will cause to an increase in membrane cleaning and high replacement cost. The effectiveness of membrane performance can be analyzed by a couple of different parameters such as transmembrane pressure, temperature, flow rate or flux decline which acts as a good indicator to describe system performance.

1.2 Environmental Regulations of Effluent Discharge

As the rise of environmental concern due to effluent discharge from industrial wastewater which contains the possible pollution source to the environment such as palm oil industry, the environmental act was then applied in order to control and monitor the discharge properties. The environmental regulations of effluent discharge for the palm oil industry was first set of regulations

promulgated under the Environmental Quality Act (EQA), 1977, for environmental control of industrial pollution (Thani et al., 1999). The effluent discharge standards of palm oil mill were presented in Table 1.1:

Table 1.1: Effluent discharge standards for POME

Parameter	Regulatory discharge limit	Unit
Biochemical Oxygen Demand (BOD)	100	mg/L
Chemical Oxygen Demand (COD)	-	mg/L
Total Solids (TS)	-	mg/L
Total Suspended Solids (TSS)	400	mg/L
Oil And Grease (O&G)	50	mg/L
Ammonia-Nitrogen (NH3-N)	150	mg/L
Total Kjeldahl Nigrogen (TKN)	200	mg/L
pH	5-9	-
color	-	PtCo

(Saifuddin and Dinara, 2011)

1.3 Problem Statement

Despite recent research advances in the treatment of palm oil mill effluent, the application of using membrane separation as a treatment in treating POME is still a big concern. There are many studies on the parameters that can affect membranes performances, but it requires more effective indicators on the performance of membranes in order to monitor and to predict the efficiency of the treatment. The problem statement outline as below:

1. An effective treatment system is needed in order to treat and control the effluent discharge by palm oil mills to the water sources with regard to the standard parameters set the Malaysian Department of the Environment. This is because POME contains high biochemical oxygen demand (BOD), high chemical oxygen demand (COD), high salt content, high suspended solid and low pH (average between 3.5 – 4.2). This composition will be the major sources of the water pollution if not well control and treat. The compound in the POME is harmful to the environment and it becomes necessary these effluent water should be treat of purified before discharge to the environment. At the present time, ponding system is the most popular treatment for POME, but it is requiring a large area of application and some of the outputs are not well treated. Membrane separation technology is a promising treatment process for treating water and wastewater and has been applied in various sectors. The advantages of using membrane separation technology include stable effluent quality, low footprint required, high

level of automation, and additional chemical is not required. However, there are limited studies about treating POME in using ultrafiltration (UF) membranes as a separation process. In this study, the source of POME is from the last an anaerobic pond where this process will act as a pre-treatment for UF membrane.

2. The application of membranes filtration process in industrial wastewater treatment especially POME has not yet applied due to membrane fouling where this defect has led to high operating cost and maintenance. To date, membrane fouling is the major problem affecting system performance which related to POME treatment as it is contains high amount of suspended solids and a macrosolute-like protein. Most of the fouling studies have concentrated more on side stream membrane system, whereas limited investigation on cake layer characterization in particular for POME treatment. Therefore, one of the aims of this research is to investigate the fouling mechanisms in order to reduce membrane fouling and to determine controlling strategies in producing high quality output of membrane treatment.
3. Previous research mostly focused on the direct applications of UF membranes in the treatment of POME, and no studies was reported on the effects of cleaning procedures. Hence, this research is to focus on the secondary wastewater produced after the cleaning processes of the UF membranes in the treatment of POME which known as “effluent of membrane washing (EMW)”. The EMW may also harm to the environment if not well treat as it contains with the pollutant from the POME treatment earlier and chemicals used during cleaning procedures.

1.4 Objective

The specific objectives of the research are:

1. To investigate the potential of UF membranes for POME treatment and examine the factors that affects the removal efficiency. In order to accomplish this objective, the quality of parameters such as chemical oxygen demand (COD), color, turbidity, and suspended solids concentration were monitored and compared before and after treatment.
2. To characterize the possible fouling mechanism which affecting membrane performance. This includes the characterization of the cake layer and foulants analysis in order to understand and controlled fouling behavior on the membrane performance.
3. To investigate the “effluent of membrane washing (EMW)” properties and using coagulation method as a treatment process for foulant removal.
4. To analyze the optimization of UF membranes performance in the treatment of POME and coagulation treatment process of EMW.

1.5 Scope of the research

The application of membrane separation process for the treatment of palm oil mill effluent (POME) by ultrafiltration membranes is the main focus of the present study. A lab scale ultrafiltration membrane system was designed and runs to study its feasibility for POME treatment. At first, the POME source was tested for chemical oxygen demand (COD), turbidity, color, pH, total solids, and suspended solids in order to get the preliminary value. Further, the clean water was also applied to the ultrafiltration membrane reactor for initial reading. Later, POME was then applied through the ultrafiltration process at various applied pressure values ranging from 50 to 200 kPa. The permeate quality such as COD, turbidity, color, pH, total solids, and suspended solids were monitored and compared.

The fouling behavior of the membrane treatment was evaluated throughout the experiments. The results obtained were then calculated and compare for fouling control and analysis. Later, the foulants characteristics were examined through scanning electron microscopy (SEM) and particle size analyzer (PSA).

After the end of each experiment, the membrane cleaning process was performed in order to remove unwanted compound and returned the membranes characteristic to the initial value. Resistance removal and flux recovery were used to confirm the cleaning efficiency and the result should at least reach 90% compared to the initial value. Later, the “effluent of membrane washing (EMW)” was collected and test for chemical oxygen demand (COD), turbidity, color, pH, and total solids. Coagulation procedures will apply as a treatment of EMW and final results will compare with the standard sets by DOE for effluent discharge limit.

The optimization and process modeling of the POME treatment through ultrafiltration membrane system will be studied by using response surface methodology (RSM). In this part of the study, the runs of experiments as based on independent variables of surface model in order to evaluate their interactions and optimize the response. The experiment also runs in triplicate in order to confirm the prediction with the predicted value in the optimum region.

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