Jurnal Teknologi

Full paper

Ultrafiltration Membrane for POME Treatment: Comparison Physical & Chemical Cleaning Process

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Article history

Received :4 October 2013
Received in revised form :
17 April 2014
Accepted :8 May 2014

Abstract

Membrane technology has been improved and implied in wide range of application such as in food, chemical, medicine and many more. Nevertheless, the application of membrane in the separation process for treating palm oil mill effluent (POME) has not been applied on an industrial scale though researches have proven many advantageous of membrane system over conventional treatment such as high efficiency, low space requirement and low energy consumption. The main obstacle in using membrane in treating POME is fouling as it increase in cost and energy of the process, process down time and decrease in the efficiency. To overcome the challenge specific cleaning process for ultrafiltration membrane for palm oil mill effluent treatment is the current focus study. The experiments were conducted on polysulfone membrane with molecular weight cut off 30 KDa.The effect of physical cleaning of forward flushing and backwashing were compared. The effect of different chemical cleaning of; sodium hydroxide, hydrochloric acid and sodium dodecylbenzene sulfonate were studied. Comparison between physical of backwashing and chemical cleaning by immersed in NaOH solution were performed to eliminate the fouling with the aims to minimize the time consume and maximize the membrane efficiency. Combination of both physical and chemical cleaning gave an interesting trend of reducing and increasing of membrane performance. Membrane separation process has improved the quality of the final effluent. The results show that by introducing the cleaning process, the performance of the flux can be recovered to more than 80 percent.

Keywords: Palm oil mill effluent; ultrafiltration; wastewater treatment; fouling; polysulfone; physical cleaning; chemical cleaning

1.0 INTRODUCTION

Recently, research on the application of membrane in various industries were greatly being done since membrane technologies have brought great potentials to the separation process such as water and wastewater treatment to remove particles, inactivation of pathogens and improvements of aesthetic [1]. Moreover, these membranes have been vastly improved in the area of water flux, salt rejection, and especially in their ability to maintain high performance levels at substantially lower operating pressures [2]. Membrane technologies are progressively being improved and employed in a wide range of application such as in food, chemical, medicine, pharmaceutical, biotechnology and many other fields of industries [3, 4] over these last few decades. This is because membrane technology has been one of the promising technologies to help the industry effectively due to its high removal capacity and ability to meet multiple treatment objectives [4].

The components of the feed that are allowed passage by the membrane is called permeate, whereas others that are retained by it and accumulate is called retentate. There are mainly four types of membrane filtration processes involving reverse osmosis (RO), nanofiltration (NF), ultrafiltration (UF), and microfiltration (MF). Different membrane types provide different pore size [5], different ways of process and different pressure process. Ahmad and Chan (2009) reported that membrane separation process is well suited for the recycling and reuse of wastewater. The main advantages of membrane technology are the constant production of high quality water, nontoxic and fully automated process [6].

In order to attain sustainability, any development should be socially acceptable, technologically appropriate, economically viable and environmentally compatible. However, the barrier that encountered in using the membrane in separation process is membrane fouling. Membrane fouling is caused by the accumulation of certain constituents in the feed on the surface of the membrane or within the membrane pores. This membrane fouling problem causes the increasing of cost and energy of the process, process downtime and decreasing the efficiency of the membrane itself because of the hardness and frequency of cleaning condition [7]. Membrane fouling causes a great reduction of productivity where the decrease in flux with time of operation due to the increasing of hydraulic resistance which can be interpreted as a need for additional energy supply to the filtration system to keep the system performance constant [8]. Mostly, the fouling is influenced by the membrane type, module configuration and the process itself [9]. Fouling can occur in any membrane system, regardless of the membrane polymer, system manufacturer, and mode of operation [10]. To remove the membrane fouling, a good management of cleaning process must be done frequently to the membrane. The membrane should be cleaned when the permeate rate drops off. Various cleaning process can be used to remove the fouling materials from the membrane and to restore the membrane flux. The factors that affect the efficiency of cleaning process are mass transfer and chemical reaction such as concentration, temperature, length of cleaning period and the hydrodynamic conditions [1]. The cleaning process can be divided into two methods that are physical and chemical method. The examples of physical methods such as backwashing, hydrodynamic shear stress scouring and high cross flow velocity and intermittent suction operation. While, chemical methods such as immerse or circulate using chemicals cleaning agents. The examples of the chemicals are hydrochloric acids, sodium hydroxide, sodium hypochloride and nitric acids. Thus, this paper aims to overcome the challenge on specific cleaning process for ultrafiltration membrane for palm oil mill effluent (POME) treatment. POME was treat using polysulfone flat sheet membrane with 30 kDa molecular weight cut off (MWCO).

■2.0 EXPERIMENTAL

2.1 Materials

The membrane used in this research is flat sheet ultrafiltration membrane; polysulfone (PS) with 30 kDa MWCO. The effective membrane area is 33.6 cm^2. The chemical used for cleaning studies are sodium hydroxide (NaOH), hydrochloric acid (HCl) and sodium dodecylbenzenesulfonatem (SDBS) with concentration of 0.005 M. While aluminium sulfate, was used to pretreat the raw palm oil mill effluent collected from Oil Palm Mill located in Nibong Tebal, Penang.

2.2 Experimental Set-Up

2.2.1 Preparation of Pretreated POME

The raw POME was filtered to remove the coarse solids in the suspension. A jar test procedure was conducted by using six beakers of 200 ml of POME each. The coagulant agent (aluminium sulfate) was added to the POME and this mixture was stirred uniformly at 150 rpm for 2 min (rapid mixing) and at 50 rpm for 30 min (slow mixing) at pH 6.5. The pH was adjusted by adding NaOH or HCl accordingly. The mixture was left to settle for 2 hours. The supernatant was then taken as pretreated POME for membrane studies.

2.2.2 Pure Water Flux for UF Membranes

The membrane was soaked overnight in distilled water to remove impurities left over from the manufacturing process or additives used for stabilization, prior to any experiment conducted. After soaked, the membrane was wetted again by circulating the distilled water at 1 bar for 30–60 min. This procedure will prevent membrane compaction during separation experiments. Distilled water flux Jo was measured with a clean membrane at the TMP 1 bar at a temperature of 25°C.

2.2.3 Membrane Fouling Studies

A membrane testing rig for POME treatment was used to perform the UF experiment. A schematic diagram of the experimental setup as shown in Figure 1. The test rig was equipped with membrane-holder for flat sheet membrane and the permeate was measured automatically using a weighing balance connected to computer acquisition using the software RsWeight Version 1.0. 500 ml of pretreated POME was pumped to the UF membrane at TMP of 1 bar. When the TMP was set up to designated test value, the flux was monitored until reach stabilization and every new TMP were tested for 60 minutes and permeate volume was recorded.

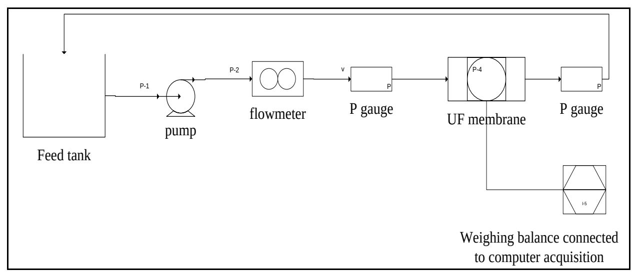


Figure 1 Schematic diagram of testing rig Ultra-filtration membrane for POME treatment

Permeate flux was calculated based on the changes in mass of permeate water with an assumption the density of POME solution is similar to water density (1 g/cm^3) as a function of time as shown in Equation 1

$$\text{Flux, } J_o = \frac{permeatevolume\ (L)}{membranesurfacearea(m^2)Xtime\ (\text{min})} \quad (1)$$

2.2.4 Membrane Cleaning Procedure

Physical cleaning: For physical cleaning the membrane was cleaned using distilled water with several techniques of forward flushing and backwashing. The cleaning time of forward flushing with distilled water were fixed to 30 seconds. The cleaning technique for physical cleaning also varies to only forward flushing and backwashing.

Chemical cleaning: For chemical cleaning the membrane was cleaned using several types of chemicals; sodium hydroxide, hydrochloric acid and sodium dodecylbenzenesulfonate. An immersed technique was used for chemical cleaning. The concentration of chemicals used was fixed to 0.005 M. The membrane surface was immersed for 1 minute. The immersion was carried by injecting the solution slowly into the membrane holder. The inlet and both outlet valves were off after the solution fully covers the membrane surface.

Combined cleaning: The membranes were continuously run with pretreated POME for 12 hours. For physical cleaning the membrane was cleaned by back washing for 30 seconds at every 2 hours interval using distilled water. For chemical cleaning the membrane was immersed with 0.005 M NaOH for 1minute at every 2 hours interval. The NaOH solution was flowed slowly into the UF membrane, after the solution fully cover the surface of the membrane both inlet and outlet valve were closed. The membrane surface was immersed for 1 minute. All cleaning procedures were conducted with fully open valve at the inlet and retentate flow and the feed pressure was kept at atmospheric pressure.

2.2.5 Scanning Electron Microscope (SEM)

The morphology and surface of membrane was scanned using a SEM (Quanta Feg 450, Holland). The surface of fouled membrane was also being inspected using various magnifications. The sample was placed on a single stub mount and mounts directly onto stage. The emission scanning electron microscope is a flexible ultrahigh performance with variable pressure and controlled by a computer system as operating system. The resolutions were carried out in High Vacuum (HV) and Variable Pressure (VP) mode. The samples were scanned with SEM at the magnification of 1,000x to 30,000x.

3.0 RESULTS AND DISCUSSION

3.1 Membrane Fouling

Introducing cleaning to the membrane process, the characteristics of flux and membrane fouling must be analysed. The reduction of flux over time for pretreated POME filtration on the 30 kDa flat sheet PS membrane at TMP of 1 bar is shown in Figure 2. Throughout the experiments, consistent and reproducible fouling were observed.

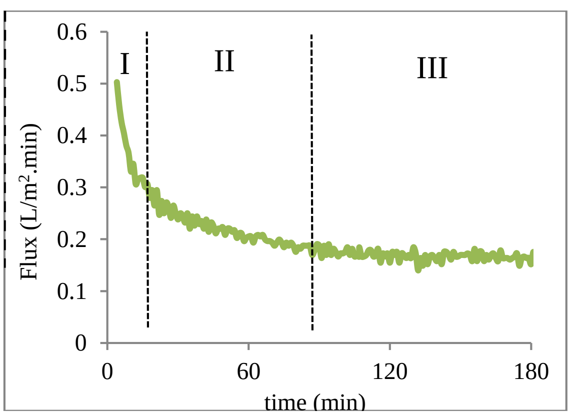


Figure 2 Flux of POME versus time

As shown in Figure 2, the permeate flux can be categorized under three stages. Stage I is rapid initial slump from flux of POME filtration, stage II; long steady flux decrease and it ended with state III; steady-state flux. According to Song (1997), stage I occurs in all membrane fouling processes regardless of their operating conditions meanwhile stage II and III sometimes is not observed in some experiments. Song (1997) also stated that, stage III can only be observed after a long time operation if the pressure is sufficiently high and the feed concentration is sufficiently low. Sometimes stage III will not be attained if the experiments stop before it achieved steady state [11].

The result also shows that permeate flux was drastically dropped for first 20 minutes. Ahmad *et al.* (2005) reported that this drastic reduction of flux for first 20 minutes indicated a phenomenon of gradual build-up of a cake layer on the membrane surface [12]. After 20 minutes onwards the flux decreased to about 50% of the initial flux. It is an evidence of cake layer completely covers the membrane surface that caused high resistance to the membrane [13]. According to Baker (2012) membrane fouling occurred through various mechanism such as internal pore constriction form adsorption of small solute onto pore walls, external (complete or partial) blocking of pores by initial deposition and cake or gel layer formation on top of the membrane [5].

3.2 Membrane Cleaning Performance

3.2.1 Effect of Physical Cleaning

Chen *et al*., (2003) reported that some findings found that forward flushing is insignificant and tempting to assume that forward flushing is totally ineffective in physical cleaning and should be removed from the physical cleaning procedure. However, his further research showed that the forward flushing serves a purpose in physical cleaning and is not fully dispensable [14]. To evaluate the effect of the physical cleaning between forward flushing and back washing method, the cleaning periods were fixed for 30 seconds. Cleaning processes using distilled water were introduced at every 60 minutes interval time. The dotted lines in Figures 3 indicate the cleaning process performed during the POME filtration.

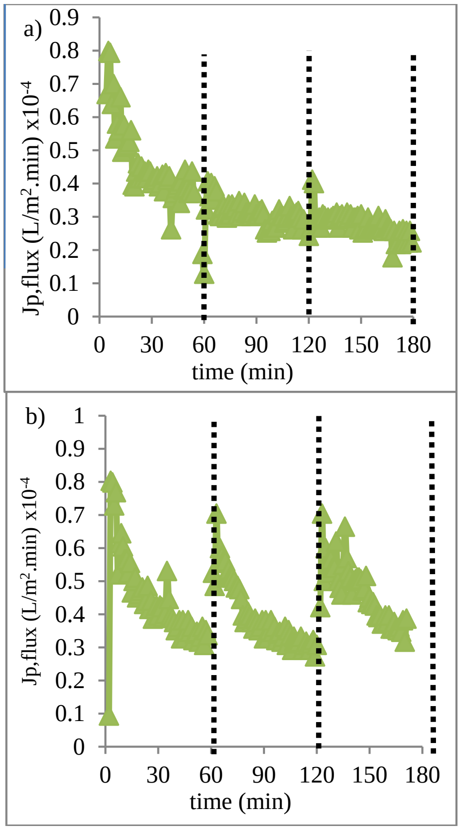
Comparing Figures 3(a) and (b) significant differences can be observed specifically in terms of cleaning efficiency. In the duration of 30 seconds, backwash method was more efficient than flushing method. The result showed that 30 seconds of backwashing is sufficient to remove the foulants but 30 seconds of forward flushing is not sufficient to remove the foulants from the membrane. It was found that fouling on the membrane surface and membrane pores could be remove effectively when the appropriate cleaning mode was applied to the operation. According to Psoch and Schiewer (2006) who compared two MBRs with and without backwashing applied at the same equivalent constant trans-membrane pressure (TMP) and they observed that the flux with backwashing was twice that without backwashing [15].

3.2.2 Effect of Different Cleaning Chemicals

Three types of cleaning chemicals were tested to distinguish the most effective chemicals solution for membrane cleaning after treating POME. Figure 4 shows the membrane performance after being cleaned with different types of chemical solutions. The concentration used was 0.005 M and being immersed for 5 minutes as it was the range practise in industrial application for membrane cleaning. The results showed that NaOH has the highest membrane performance with 93% and 92% after first and second wash respectively.

For membrane performances of HCl and SDBS, the results obtained were 84% and 78% after first wash and 74% and 76%

after second wash respectively. HCl solution showed the worse performance after being immersed for 5 minutes. The permeate flux recovery drop significantly from 84% to 74% after second time of cleaning. Based on the result, immersing membrane in 0.005 M of HCl for 5 minutes is not sufficient to remove all the foulants from membrane surface. Either higher concentration or longer time was needed to clean the membrane.



Figures 3 Flux versus time: (a) forward flushing (b) back wash for 30 seconds

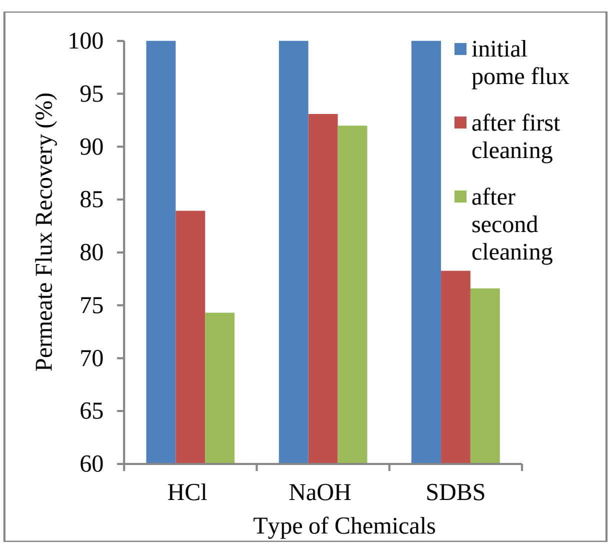


Figure 4 Results of permeate flux recovery with different chemical cleaning agents (Immersed for 5 minutes at the concentration of 0.005 M of cleaning chemicals)

For SDBS, the permeate flux recovery drop drastically to 78% after the first wash, indicates that SDBS was the least suitable detergents to remove the foulants from the membrane surface. The membrane performance after being treated or cleaned by these three chemical were significantly obvious where alkaline solution produced best results.

According to Zhang and Ma (2009) oxidant and alkaline detergent are suitable to remove organic foulants, meanwhile inorganic acid, organic acid and acid detergent are suitable to remove inorganic foulants [16]. Therefore, NaOH solution which is alkaline detergent is excellent in removing organic foulants of POME, while HCl and SDBS are suitable to remove inorganic fouling. According to Chen *et al*. (2003) the significant factor that affecting the chemical cleaning for UF and RO membranes are the high pH and the concentration of the cleaning solution [14]. NaOH was used to clean membranes fouled by organic and microbial foulants and foulants occurred on the membranes are caused by natural organic matter. Therefore, NaOH solution is proven as the most effective chemicals to be used to clean PS membrane especially in treating POME. Since POME is waste water contained high natural organic matter with high concentrations of protein, carbohydrate, nitrogenous compounds, lipids and minerals.

3.2.3 Effect of Combining Physical and Chemical Cleaning

Throughout this study, the main fouling mechanism identified from the fouling analysis is cake layer and the appropriate cleaning method is chemical cleaning. However, the factor of pore blocking mechanism cannot be neglected as being tested through physical cleaning, where backwashing for 30 seconds able to regain the permeate flux. Therefore, to compare the effectiveness of cleaning procedure both physical and chemical cleanings needs to be operated for long hours. The cleaning was introduced at every 2 hours time interval for 12 hours continuous operation.

Figure 5 illustrates the analysis results of permeate flux performance with five cycle of backwash cleaning. The result showed that after first backwash the membrane performance reduce to 97%, however after second time being backwash the membrane performance increase to 99%, 103%, 107% and 108% for third, fourth, and fifth backwash respectively. From Figure 5, backwash has proven to be efficient in recovering the membrane performance but it is also has a drawback. Frequent backwash on the membrane may damage the membrane pores. According to Wu *et al*. (2007) membrane damage may occurred due to the frequency and harshness of cleaning condition [17]. Bowen and Sabuni (1994) stated that flow reversal from the permeate side to feed side creates mechanical stress on the membrane in a direction in which it is not supported [18] because the membrane were supported by a thicker and more porous sub-layer, therefore it could be easily damage by repeated mechanical stress during flow reversal.

Figure 6 presents the analysis results of permeate flux performance with five cycles of chemical cleaning through immerse in 0.005M NaOH for 1 minute at every 2 hours interval. The result shows that after first immersed the membrane performance reduce to 96%, and keep reducing to 95%, 91%, 89% and 84% for second, third, fourth, and fifth cycles respectively. The chemical cleaning efficiency found to be gradually decreasing. Chen *et al*., (2003) postulated that cleaning chemicals cause deposits on the membrane surface to swell that for a compressed fouling layer [14]. This is due to operation intervals, when the interval is stretched, the resulting fouling layer may be more tightly bound and may have undergone some degree of compaction. For this fouling layer it requires more concentrated cleaning solution after each cleaning interval. Based on result obtained, it is not advisable to only apply chemical cleaning throughout the operation.

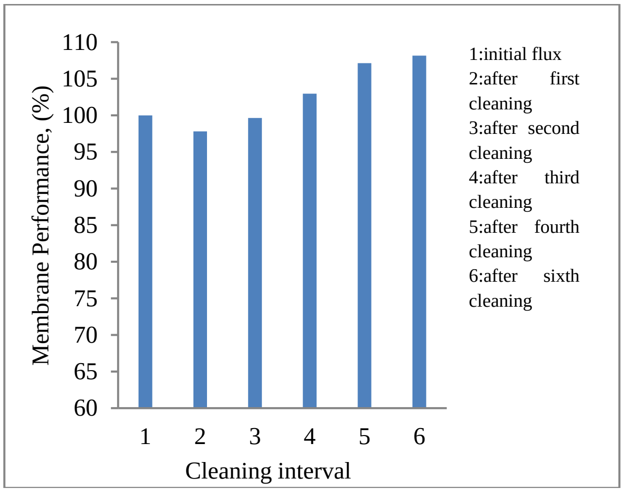


Figure 5 Membrane performance versus cleaning interval: backwash 30 seconds

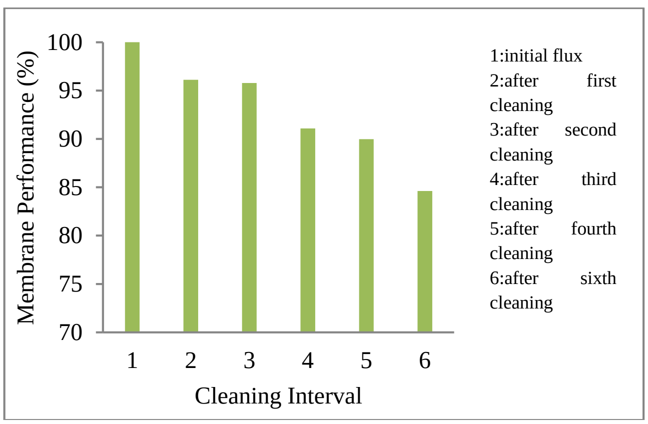


Figure 6 Membrane performance versus cleaning interval: immerse 0.005M NaOH for 1 minute

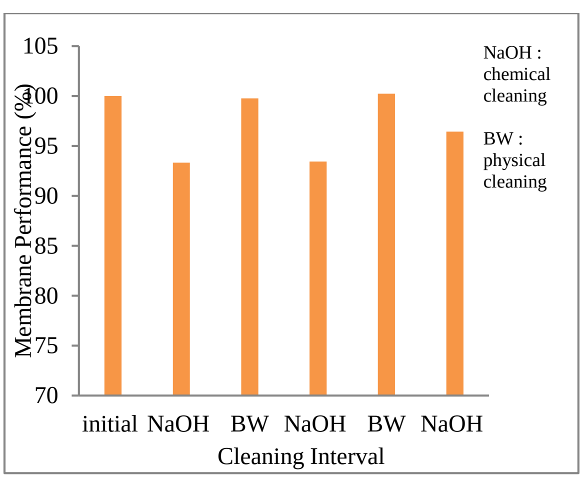


Figure 7 Membrane performance versus cleaning interval: immerse with 0.005M NaOH 1 minute and alternate with backwash 30 seconds

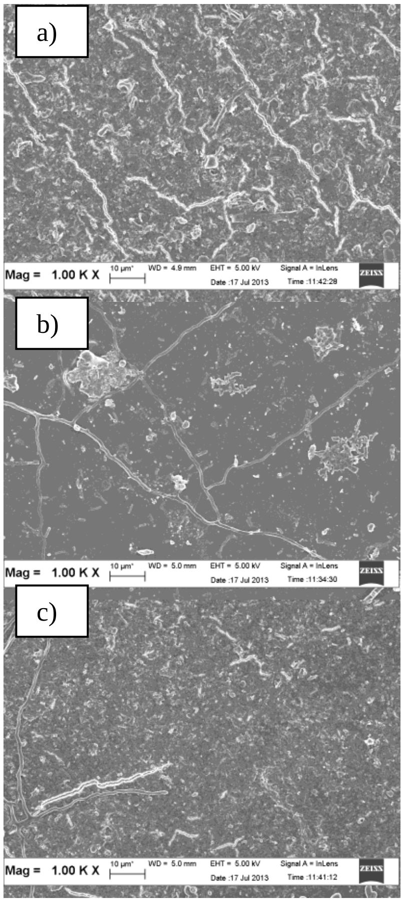
Comparing Figures 5 and 6, both physical and chemical cleaning need to be combined in order to maintain the membrane performance and the cleaning efficiency. Ang *et al* (2006) stated that efficient cleaning can be achieved through the coupling between the chemical reaction and mass transfer along with the optimization of cleaning conditions responsible for the favorable chemical reaction and mass transfer [19]. Figure 7 showed the result for combining physical and chemical cleaning of UF membrane in treating pre-treated POME. The membrane was being cleaned by immersing with NaOH and backwashing with distilled water alternately. For the first cleaning, the membrane was being immersed with NaOH for 1 minute, the membrane performance reduced to 93%, then after being backwash for 30 seconds the membrane performance increased to 99%. The result showed a unique trend of reducing and increasing of membrane performance after being alternately cleaned with different methods. The flux can be recovered to 100% after being cleaned by backwashing method. The overall performance can be maintained above 90% throughout 12 hours operation.

The effective cleaning process requires both of physical and chemical interactions to remove the foulants from membrane surface and pores. During chemical cleaning, chemical reaction will occur between the solution and the foulants in the fouling layer. When chemical solution and the foulants make contact, it will be able to weaken the structural integrity of the fouling layer [19]. The physical cleaning which responsible for the mass transfer of the reactions products then will help by removing the foulants from the membrane surface. Therefore by combining the physical and chemical cleaning is the suitable clean-in-place process to eliminate the fouling, in the same time reducing cost, time and energy consumption. With this result, combination of both chemical and physical cleaning methods with tested concentration and time exposure is proposed especially for POME treatment.

3.3 Visual Inspection of Membrane Surface

The visual inspection using scanning electron microscope (SEM) was done to observe any deterioration and abnormalities on the membrane surface after the cleaning process. All the membranes were treated for 12 hours excluding the cleaning time with different cleaning methods. Three conditions of the fouled membrane were taken; after chemical cleaning, physical cleaning and combine cleaning. Figures 8 and 9 showed the surface morphology for chemical cleaning, physical cleaning and combine cleaning respectively.

Figures 8 and 9 showed the surface of PS membrane after being treated for long time operation and being cleaned with chemical cleaning, physical cleaning and combine cleaning with 1000 and 30000 magnifications. Figures 8(a) and 9(a) showed the surface of PS membrane after being treated for long time operation and being cleaned with chemical cleaning by immersed into NaOH solution for 1 minute. From Figure 8(a) showed the surface of fouled membrane was massively covered by the foulants. From Figure 9(a) the particles morphology can be seen clearly. The particles are flaky, some were clumped together and appear like a branched of crystalline shape. This is in agreement with Chen *et al* (2003) mentioning that cleaning chemical cause deposits on the membrane surface to swell [14]. Figures 8(b) and 9(b) showed the surface of PS membrane after being treated for long time operation and being cleaned with physical cleaning by backwash using distilled water for 30 seconds. From Figure 8(b) the surface of fouled membrane was spotted with little foulants.

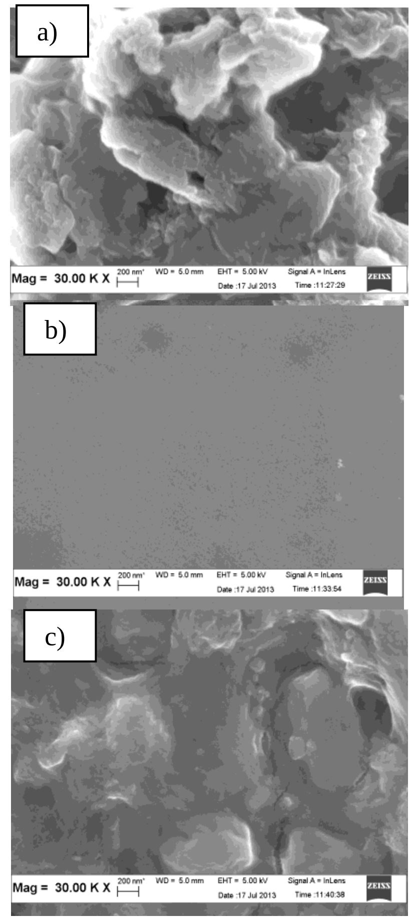


Figures 8 SEM image of fouled membrane after (a) chemical cleaning (b) physical cleaning and (c) after combine cleaning of physical and chemical with 1000x magnification

From Figure 9(b) the membrane surface morphology can be seen clearly. The membrane surface was cleaned from any foulant. This showed that backwash is efficient in removing foulants. However, the possibility of backwash method to damage the membrane was high if the membrane being exposed for backwash frequently. Figures 8(c) and 9(c) showed the surface of PS membrane after being treated for long time operation and being cleaned by combine cleaning of physical and chemical alternately. From Figure 8(c) the surface of fouled membrane was partly covered with foulants. From Figure 9(c) the membrane surface morphology was differ compared to Figure 8(a). The foulants on the membrane surface were in shape of fouling layer with some flake shape.

■4.0 CONCLUSION

The present study presents the investigation on membrane fouling and proposes appropriate cleaning solution for polysulfone membrane applied in treating POME. The reductions of flux versus time on the flat sheet membrane were studied and throughout the experiments consistent and reproducible fouling were observed. The performance of the membrane cleaning can be improved by introducing appropriate chemical cleaning for the membrane.



Figures 9 SEM image of fouled membrane after (a) chemical cleaning (b) physical cleaning and (c) after combine cleaning of physical and chemical with 30000x magnification

For physical cleaning back wash method was more efficient compared to forward flushing method because it can eliminates cake layer and clogging inside the membrane pores. For chemical cleaning NaOH is the most effective chemicals solution to clean the membrane after treating POME. By immersing the membrane in the 0.005 M NaOH for 1 minute is sufficient to remove the foulants. For long operation, physical cleaning using backwash the membrane performance reduce to 97%, however after second time being backwashed the membrane performance start increasing. Frequent backwash on the membrane may damage the membrane pores distribution. Therefore by combining the physical and chemical cleaning the membrane performance the result showed a unique trend of reducing and increasing of membrane performance after being alternately cleaned with different methods. The treatment of POME can improve by introducing a proper pre-treatment process before the separation process. From this study, it is obvious that UF membrane shows high potential in treating POME.

Acknowledgement

We thank our sponsors through Fundamental Research Grant (203/PJKIMIA/6071253) and Membrane Cluster Research Grant (1001/PSF/81610011) for this research.

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