

Effect of Regenerated Cellulose of Ultrafiltration Membranes on POME Treatment

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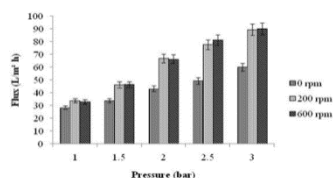
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Graphical abstract



Abstract

Palm oil mill effluent (POME) in palm oil industry has become a big issue of environmental pollution to be solved urgently and critically. This wastewater consists of water, oil, and natural sediments. Hence, in this study, the work was carried out to investigate ultrafiltration process feasibility for treating palm oil mill effluent. Palm kernel shell bioactivated carbon (PKS-AC) adsorbent was used in adsorption treatment (pre-treatment) was used to reduced solid particles in POME. For adsorption treatment, POME was stirred with 0.20 g/L of PKS-AC at 39.94 minute and sediment for one hour. Membrane separation was subsequently applied to further treat the pre-treated POME. In this study, the permeate flux was found to be dependent to pressure applied, solution pH and stirring speed. An optimum conditions was achieved at pressure 2 bar, with solution pH 8 using stirring speed 600 rpm. Considerable amount of POME pollutant is also reduced by used membrane for TS, DS, SS, BOD₅, COD and turbidity were 625.32 mg/L, 445.32 mg/L, 180 mg/L, 1296 mg/L, 541.76 mg/L, and 16.20 NTU respectively. Thus, this result show that the pollutant in POME was reduced significantly using this technique.

Keywords: Palm oil mill effluent; palm kernel shell-based activated carbon; ultrafiltration; treated POME

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1.0 INTRODUCTION

Oil palm is an important crop in Malaysia, accounting for 39% of the world's palm oil production and 44% of the world's exports [1]. About 4.49 million hectares of land in Malaysia are under oil palm cultivation and are producing 17.73 million tons of palm oil [1]. Currently, due to the demand for palm oil and oleo chemical industries, its production is expected to increase [2]. Given the huge capacity of this sector, it is critical to treat palm oil mill effluent (POME) to an acceptable level before discharging it.

Raw POME is a thick brownish viscous liquid waste with an unpleasant odor and high in colloidal suspension [3]. The wastewater generated from palm oil processing has 95-96% water, 0.6-0.7% oil and 4-5% total solids [4, 5]. Due to its high biological oxygen demand (25,000 mg/L), chemical oxygen demand (53, 630 mg/L), oil and grease (8, 370 mg/L) and suspended solid (19, 020 mg/L), its disposal without proper treatment in water bodies has become undesirable [6, 7]. Hence, the palm oil industry has a big responsibility to face it in term of environmental protection, economical viability, and sustainable development.

Over past few decades, there are several treatment that have been developed and employed by palm oil mills for the treatment of POME. Currently, the most popular applied the mill is conventional biological treatments involving anaerobic or facultative digestion [4, 8]. However, the biological treatment

system has some impact and disadvantage regarding its operation. This system requires proper maintenance and monitoring, as the bacteria are very sensitive to the changes in the environment. Thus, environmental conditions must be maintained that are conducive to the survival of the microorganisms. Requirement of the skilled operator for monitoring purpose will be costing. In the same time, commitment from the management is required also. Moreover, biological treatment also generates vast amounts of biogas, which is corrosive and odorous [4]. Due to the reason that effluent treatment is often viewed as the most significant burden, these issues have normally been ignored and given the lowest priority by mill owners. As to overcome the problems with the biological treatment method, it is believed that membrane separation technology will be able to treat POME in a more beneficial way. The membrane separation technology is normally coupled with pre-treatment. The pre-treatment is required to avoid high fouling affect toward the membrane. Commonly, fouling occurs due to the high content of sludge, colloidal matter, and suspended solids in POME [7, 9]. Most of the difficulties in membrane technology were to overcome the fouling membrane due to the POME characteristics. POME is rich in suspended solids, sludge, and colloidal particles which need to be degraded first. In this study, the membrane technology was coupled with pre-treatment to overcome the problem by using bio-char and surface filtration. The combination of this adsorption treatment and surface filtration

gave a huge good impact in water reuse recovery, making it possible to avoid the usage of acid or base to adjust the pH later in order to comply with the standard.

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 and the water reclaimed from this treatment could be reused in the mill [10]. 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 [11].

There were two stages involves in pre-treatment. The first stage was adsorption treatment using palm kernel shell bioactivated carbon (PKS-AC). One of these types of treatment is by using activated carbon. Adsorption-based innovative technology developed with low cost carbonaceous materials showed good potential [12, 13]. Generally, activated carbon is functionally prepared to exhibit an extended inter-particulate surface area and a high degree of porosity [13, 14]. Moreover, activated carbon is considered excellent adsorbent characteristics in reduction of color, adsorbable organic halides (AOX) and non-biodegradable of such wastewater [12, 16]. The activated carbon is very useful for many purposes including filtration, purification, deodorization, decolorization, purification, and separation [15]. Activated carbons have been produced from carbonaceous raw materials such as coal, lignite, wood, coconut shell, and some agricultural waste products [17]. All of these raw materials are abundant and renewable resources. The effectiveness of activated carbon as an adsorbent depend to its unique properties, large surface area, a high degree of surface reactivity, universal adsorption effect, and favorable pore size [14]. The second stage of pre-treatment is filtration treatment. As a definition, filtration is a fundamental unit operation that separates suspended particles matter from wastewater; its operate by passing the solution through a porous membrane or medium when the solid particles are retained on the medium surfaces or within the pores of the medium while the fluid known as filtrate or permeate passes through it [18]. There are two type of filtration that introduced as environmentally friendly which are depth filtration and surface filtration [7, 19]. Surface filtration is use in this study to be couple with adsorption process to treat POME. Surface filtration involves the removal of particulate particles by passing through the solution through a thin spectrum (medium).⁷ The medium suggested to be used is UF membrane.

The ultrafiltration (UF) membrane techniques were used to recover crystal clear water from the POME. The UF membrane has a finely porous surface layer or skin corroborated on a much more open microporous substrate. That finely porous surface layer is what actually performs the separation for which the microporous substrate provides mechanical strength. Meanwhile, the UF retains the bacteria and viruses, which allows for its use as a water disinfectant [9, 20].

The objective of the present study is to investigate the performance and feasibility of ultrafiltration membrane treatment in the reduction of pollutant in POME. The different parameters affecting the performances of ultrafiltration membrane such as operating pressure, stirring speed and solution pH were studied to obtain the optimum operating conditions.

2.0 EXPERIMENTAL

2.1 Material

Samples of wastewater were taken from a local palm oil mill in Labu, Negeri Sembilan. Raw palm oil mill effluent (POME) was taken from a sludge pit that had a temperature of around 80°C to 90°C. The sample was stored at 4°C to avoid biodegradation due to microbial action. For the analysis and experimental purpose, the temperature of the sample was allowed to reach room temperature.

Palm kernel shell-based activated biocarbon (PKS-AC) from K.D technology was used in granular form with size range less than 2000 µm and more than 500 µm All activated carbon were in laboratory grade and used directly as received from the supplier. All the adsorbent were analyzed using BET for the characterization.

The membrane use in this study was flat sheet regenerated cellulose (RC) membrane with 28.7 cm² effective membrane area and membrane diameter of 63.5 mm. The MWCO of the RC membrane used was 5 kDa. This membrane is hydrophilic and negatively charged surface. The membrane was supply from Milipore.

2.2 Analysis

The Biological Oxygen Demand (BOD₅) was measured using the Azide modification of the iodometric method. Meanwhile, the Chemical Oxygen Demand (COD) analysis was conducted using a dichromate reactor digestion method. The suspended solid analysis was carried out by evaporating the filtrate of the sample to dryness in a weight crucible and then drying it to a constant weight at 105°C. The addition in the weight indicated the presence of suspended solids. Turbidity was measured using a turbidity meter HACH 2100AN. The pH was measured by a pH meter (Eutech instrument). The details of all the analytical methods conducted were based on procedures given in the APHA, Standard Method for the Examination of Water and Wastewater [21]. Each analysis was done twice. Membrane fouling can be observed by the reduction of flux through a membrane [22]. It is caused by the buildup of contaminants at the surface or inside (pore) the membrane

2.3 Ultrafiltration Experiment

A stirred UF test cell of 200 ml was supplied by Milipore that can accommodate the flat membrane with diameter of 28.7 cm. The suspended bar impeller of 2 cm inside the test cell was magnetically driven by a stirrer. This device is pressurized to force fluid through the membrane while retaining and concentrating the macromolecules. The type of UF membrane unit used was dead-end filtration.

2.4 Experimental Procedures

First an amount of PKS-AC was mixed up with raw POME in a certain operating conditions (time and stirring speed) using Flocculator SW1 (Stuart Scientific). After that, the POME was left for one hour as for sedimentation process. The supernatant was then pipette out for the next treatment.

180 ml of pre-treated POME was prepared for each run. The pre-treated POME was put into the unit. The schematic diagram of stirred cell used can be seen in Figure 2. The experiment was done in batch mode. There were two parameters measured to see the effectiveness of membrane towards the rejection of DS and turbidity in the permeate (treated POME).

Two parameters control in this batch experiments were stirring speed, pH of the feed solution (pre-treated POME). The membrane unit was pressurized using five different compressed air pressure (1, 1.5, 2, 2.5 and 3 bar). For each cycle, the experiment was run in 90 minutes. The permeate flux was being observed by collecting the permeate volume for every five minutes and the volume was recorded.

3.0 RESULTS AND DISCUSSION

3.1 Quality of Pretreated POME

POME was first treated using PKS-AC with adsorption technique. There were three factors that were investigated to see the effect on SS of POME after adsorption treatment. The three factors were PKS-AC dosage, treatment time and string speed. The optimum conditions obtain from adsorption batch study was as follow, 0.20 PKS-AC dosage, 35.94 minutes and 39.82 rpm stirring speed with 71.26% SS reduction. As an overall, PKS-AC can be used as adsorbent in adsorption treatment for pre-treatment of POME. This pre-treatment successfully reduced TS, DS, SS, BOD₅, COD and turbidity up to 67.30%, 47.11%, 71.26%, 63.23%, 42.38%, and 63.31% respectively. Using this pre-treated POME it is believed can reduce the fouling effect during ultrafiltration process later

3.2 RG UF Flat Membrane and Quality of Permeate Analysis

As shown in Figure 1, the permeate flux was significantly different between with and without stirrer. For example at operating pressure 1.5 bar, the permeate flux at 0 rpm, 200 rpm and 600 rpm were 33.87 L/m²h, 46.20 L/m²h, and 46.41 L/m²h respectively. The trend shows that the permeate flux increased as the stirring speed increased. The same trend was also observed in investigations carried out at different operating pressure.

The higher flux was noticeable with the presence of stirring effect which will reduce concentration polarization within the solute and membrane surface. Moreover, as stirring speed increased the shear stress and hydrodynamic effect on membrane surface will increase too. It was due to the the accumulated compounds on membrane surface return in to the bulk of the fluid and concentration polarization effect diminishes [23]. Thus, it causes the osmotic pressure to decrease and permeation flux to increase [24, 25]. However, the permeate flux at 200 and 600 rpm at 1.5 bar were 46.20 L/m²h, and 46.41 L/m²h shown that it was not significantly increase. This phenomenon was also observed by other researchers which in finding that at a definite threshold, increasing speed does not affect permeation flux [25, 26]. The negligible flux change at elevated stirring speed was due to the portion of the cake layer resistance is relatively small compared to the other resistances caused by the membrane itself and fouling layer. Hence, the dislodging of the cake layer at higher shear rates may not show any obvious in flux changes. Moreover, the cake layer also too sticky to be removed by the shear applied [27]. Thus, permeate flux does not significantly affect by varies stirrer speed but still significantly affect the flux performance with the presence of stirrer. Hence, with stirring, its lower the fouling resistance and subsequently permeation flux is higher.

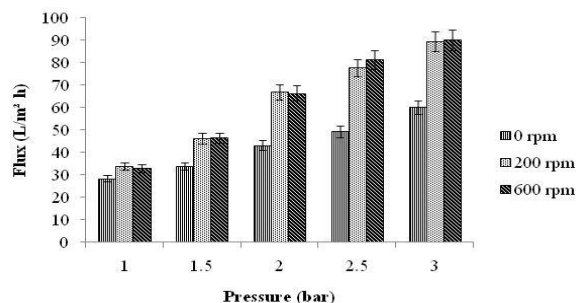


Figure 1 Permeate flux analysis on different pressure and speed at 90 minutes operation time with pH 5.85

By referring to Figure 2(a), as the pressure increased until 2 bar, DS rejection was increased with increasing stirring speed. In the other hand, the trend was suddenly changed at pressure 2.5 bar and 3 bar, DS rejection increased from 0 rpm to 200 rpm but decreased back at 600 rpm.

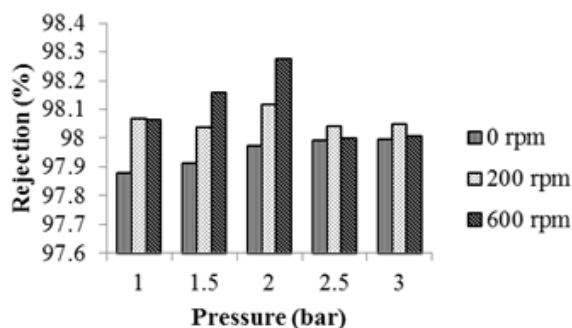
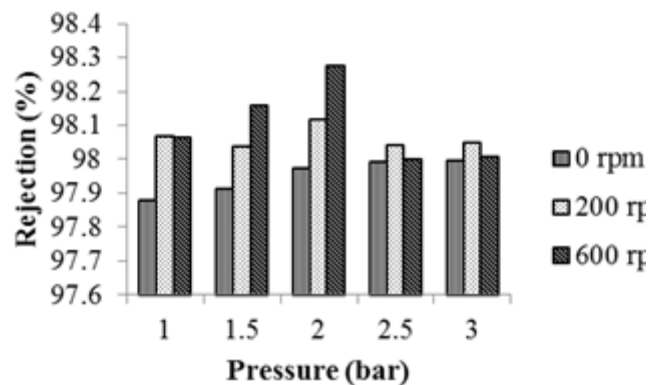
DS rejection is lower when the solution was unstirred because there was no hydrodynamic effect which can lead in increasing the deposition of cake layer on the surface of the membrane. This deposition of cake layer will increased the possibility of DS to pass through the membrane with forces from pressure applied. In the other hand, unstirred conditions showed increasing in DS rejection when the operating pressure is increasing. At higher pressure, it is believed that the gel polarization layer formed at the higher pressure is stable to disruption by pressure fluctuation [28]. This layer will act as filter on the top of membrane that reduced the permeability of DS through the membrane.

When the solution was stirred at 200 rpm and 600 rpm, the DS rejection was decreased from 1 to 2 bar but increased from 2.5 to 3 bar. This is because at low pressure, the pressure will not enough to pressurized some of the deposition of cake layer on the surface to the permeate side but at high pressure, it is possible with stirred condition. Moreover, when there the stirring speed is too high with high pressure, it will sweep away the deposition of the cake layer on the membrane surface and the dissolved particle will easily pass through the membrane [29]. From the above explanation, it can be state that the DS rejection has an impact on unstirred and stirred condition.

From Figure 2(b), as the stirrer speed, the turbidity rejection is also increase. At pressure 2 bar, the turbidity value at 0 rpm, 200 rpm and 600 rpm were 12.6 NTU, 7.32 NTU and 6.66 NTU. This decreasing order of turbidity indicates increasing in rejection percentage. The same trend was obtained at five different operating pressures. This is because by increasing speed, the hydrodynamic effect and shear stress is increase too which then returning back the accumulated compounds on the membrane surface back to the bulk of fluid [23]. Thus, the concentration polarization effect diminishes. As reported by Benito (2001), it is believe by increasing in hydrodynamic effect the possibility of gel layer formation is lower as a result of higher shear rates at the membrane surfaces. Thus, it will lower the possibility of the colloidal to pass through the membrane.

Meanwhile, in unstirred condition with increasing in operating pressure, the turbidity rejection was reduced. This might be due to the fouling phenomenon. This fouling could be due to the concentration polarization and deposition of cake layer on the surface of the membrane which will then lead to the pore blockage. As the pressure is increase, there will be more

forces to reject the particles that accumulate on the surface or particles that blocking in internal pores into the permeate side.



(a)

(b)

Figure 2 The rejection percentage of (a) DS and (b) turbidity after ultrafiltration of POME

From tabulated data in Figure 3, at pressure 1 bar, permeate flux decreased from pH 5.85 (32.51 L/m²h) to 7 (29.01 L/m²h) but increased back at pH 8 (35.86 L/m²h) and 9 (36.74 L/m²h). The same trend can be seen at pressure 1.5 bar. At pressure 2 bar, permeate flux at pH 5.85, 7, 8 and 9 were 66.38 L/m²h, 65.85 L/m²h, 65.59 L/m²h and 66.59 L/m²h respectively; permeate flux at this pressure were slightly similar although pH was varied. This phenomenon was observed from pressure 2 to 3 bar. It showed that permeate flux became insensitive to pH. The changes in permeate flux in regards to solution pH shows that the feed chemistry was changed at acidic and basic solutions. Advantageously, this will causes the fouling resistance on the membrane surface to reduce and the permeate flux of the membrane to enhance [31].

The higher permeate flux can be observed at pH 8 and 9 rather than pH 5.85. When pH is adjusted to alkaline, the

carboxylic group on the surface fully dissociates and in the same time membrane surface gains its strongest negative charge [32]. The negative charge of the polymer chains in three-dimensional network of the surface start to repel to each other and later will open up the pore of the membrane skin layers. Hence, the water permeability to permeate side will be more efficiently. It was also found that, at alkaline state, the particles form a stable suspensions and the fatty acid molecules (POME is an oily wastewater) are converted into ions and their accumulation on the surfaces reduces [23]. As a result, the flux will be increased. Meanwhile at neutr pH (pH 7), the permeate flux shows lowest value compared to other pH. At this pH, the polarized layer in the membrane surface was constituted by the small molecules which form the structure less open and resulting in a comparatively lower permeate flux [33].

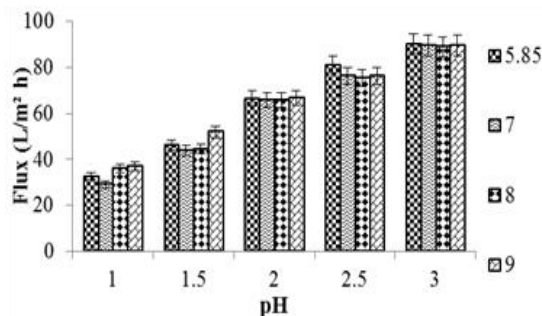


Figure 3 Permeate flux analysis on different pressure and feed pH at 90 minutes operation time using stirrer speed 600 rpm

Data in Figure 4(a) represents the DS rejection after ultrafiltration of POME at various pH and pressure. DS rejection seem to be decreased as pH increased from 5.85 to 7 and increased back as the pH increased to 8 and 9. For example at pressure 1.5 bar, the DS rejection for pH 5.85, 7, 8 and 9 were 98.15 (418.68 mg/L), 97.54% (558 mg/L), 97.94% (468 mg/L) and 98.05% (442.8 mg/L) respectively. This similar trend was also observed at other operating pressure.

DS in the solution (pre-treated POME) became unstable and degrade into more fractions of simple molecules. Moreover, the concentration of H⁺ ions and density for the negative ions on the surface particles became low as in acidic conditions [34]. In this situation, the DS particles carry (positive) opposite charge from the membrane (negative charge) which will increase the attraction forces. The ‘dynamic membrane’ will be formed due to the adsorption of the DS particles [35]. This dynamic membrane has the same charge as the other DS particles in the solution. As a result, the DS particles in the solution that approached the membrane surface will repel (self rejection) and consequently reduced the transmission of DS. Ds rejection at solution pH 8 and 9 were higher than pH 5.85 and 9. When POME is adjusted to alkaline, the particles in POME were in stable conditions [34]. Moreover, unlike in acidic conditions, the density of negative ions on the particles surface was higher in alkaline condition. Alkaline solution also carries more OH⁻ ions. The repulsion forces will repel the DS particles away from the membrane surface due to the same ion charge carry by the membrane and DS particles. This phenomenon called ‘intrinsic electrostatic rejection’ [35]. As expected, this repulsion force increased the rejection of DS.

From the result display in the Figure 4(b) it can be seen that, the turbidity rejection does affect by pH. Averagely, the turbidity rejection was higher at pH 8 with 99.72% and lowest at pH 5.85 and 7 with 99.63%. Meanwhile at pH 9, the turbidity

rejection averagely was 99.66 %. At acidic pH, the positive ions on the surface of the particles are much higher which will give more attraction forces toward the membrane surface. When the particles is near the membrane surface, the tendency for the particles to transmitted into permeate side is high. At pH 7, the particles have a balance ions charge. At pH 8 and 9, the particles with concentration of OH⁻ ion is high, which then will repulse away from the membrane surface since regenerated cellulose membrane is slightly negative charge. Hence, it increase the rejection of turbidity. However, when the pH is higher, while flux is increase, the permeability of the particles in feed solution will be increase too. At high pH the membrane matrix would be in a more expanded state due to the greater intramembrane electrostatic repulsion [32].

As an overall, with higher permeate flux (65.59 L/m²h), rejection of DS (98.04%) and turbidity (99.74%) and lower operating pressure (for low cost purpose), the suitable operating conditions to treat POME in this study was at pressure 2 bar with pH 8 and 600 rpm stirring speed. The characteristics of raw POME, pre-treated POME and treated POME were shown in Table 1.

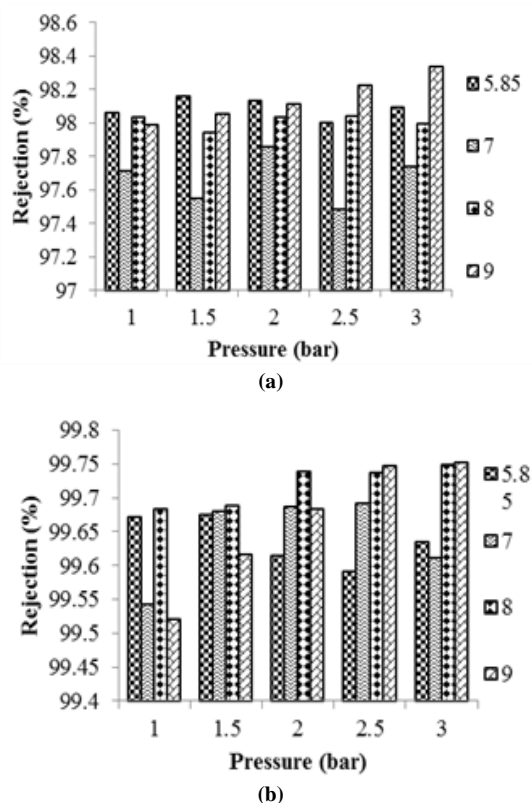


Figure 4 The rejection percentage of (a) DS and (b) turbidity after ultrafiltration of POME

Table 1 Quality of raw POME, pre-treated POME and treated POME

Parameter	Raw POME	Pre-treated POME	Treated POME
pH	3.9	5.85	8
TS, mg/L	75,200	32,000	625.32
DS, mg/L	43,015	22,750	446.40
SS, mg/L	32,185	9,250	180
BOD ₅ , mg/L	31,000	11,400	1296
COD, mg/L	73,306	42,240	541.76
Turbidity, NTU	17,000	6236.65	16.20

4.0 CONCLUSION

The membrane separation has been proved that it can be a promising process for the treatment of POME and water recovery due to the high fluxes obtained alongside significant rejection of pollutant in raw POME. It can be considered as a proper alternatives instead of the old methods. This treated POME can be used as water reuse by further applying any water treatment method for the better quality of water reuse.

Acknowledgement

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