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Effect of Chitosan Dosage in Polyethersulfone (PES) Membrane Performance in Treating Palm Oil Mill Secondary Effluent (POMSE) Wastewater

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Abstract: Micro and ultra-filtration membranes are eff2022.03.02.002ective in removing a wide variety of dissolved pollutants. In this study, Palm Oil Mill Secondary Effluent (POMSE) been used to be treat. Palm Oil Mill Secondary Effluent (POMSE) is the wastewater that generated from Palm Oil Mill Effluent (POME). POMSE that treated using conventional method require about 30 to 60 days to fully be treated. Thus, Polyethersulfone-Chitosan (PES/CS) been synthesized in this study to reduce the time to treat POMSE wastewater. Therefore, the purpose of this study is to investigate the performance of Polyethersulfone (PES) membrane with the presence of chitosan with different dosage in treating Palm Oil Mill Secondary Effluent (POMSE) wastewater. The PES membrane will be synthesized with the chitosan with different dosage which are 0.00 %, 0.25 %, 0.75 % and 1.00 % produce Polyethersulfone-Chitosan (PES/CS) membrane to improve the performance of PES membrane. The characterization of the membrane been observed through Scanning Electron Microscopy (SEM) and Fourier Transform Infrared (FTIR). The porosity of the membrane been observed by using SEM while the chemical composition of the membrane been observed using FTIR. The membrane performance of each different dosage of chitosan then been tested by using filtration method to treat the POMSE wastewater. According to the POMSE wastewater treatment results obtain in this study it is shown that PES/CS membrane with 0.75 % of chitosan dosage is the most efficient to treat POMSE wastewater. In this research, it also shown that membrane technology only takes one day to treat POMSE wastewater while conventional method takes about 30 to 60 days to treat POMSE wastewater.

Keywords: Polyethersulfone Membrane, PES, Chitosan, POMSE

1. Introduction

Nowadays, many industries developing a lot of technologies to treat wastewater starting from conventional method to membrane technology. Current technologies that well known in their effectiveness in treating wastewater are membrane filtration, nanotechnology, automatic variable filtration (AVF) technology and the microbial fuel cells. However, the involvement of membrane technology in treating wastewater have been brought

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to the attention. Membrane filtration is critical in the development of sophisticated water reclamation systems, and new and improved systems are likely to be developed in the future [1]. Micro and ultra-filtration membranes are effective in removing a wide variety of dissolved pollutants. Membrane bioreactor filtration technique is widely utilized for advanced treatment to generate water that can be reused by industry [1].

1.1 Polyethersulfone (PES) Membrane Chitosan

PES membrane had been used in treating wastewater since then because of its strong mechanical property despites of its low antifouling characteristics which made the PES membrane cannot be use without addition of the other material such as nanomaterials. As well as PES membrane, chitosan also had been used widely to treat wastewater. Chitosan is the organic material that can be easily be found from the seashells, prawn skin and also crab shell. Chitosan was known to be used as a flocculant in wastewater treatment.

1.2 Problem Statement

POMSE is conventionally treated with biological treatment involving two processes, aerobic and anaerobic which require a long period of hydraulic retention time and produce methane and carbon dioxide (CO_2) which can cause other environmental issues such as air pollution. POMSE that treated using conventional method require about 30 to 60 days to fully be treated. Therefore, the involvement of membrane technologies may decrease the time to treat the wastewater. PES is one of the most common membrane used in the industries to treat water. However, PES membrane cannot be use alone without any addition of addictive because of its low antifouling properties. Therefore, chitosan been chosen in this study to increase the performance of the membrane while increase the antifouling properties of the membrane.

1.3 Research Objectives

The objective of the research includes to prepare Polyethersulfone (PES) membrane with chitosan as an additive, to characterize the fabricated PES-Chitosan membranes by their physical and chemical properties and to investigate the PES-Chitosan membranes performances in treating POMSE wastewater.

1.4 Scope of Research

The scopes of the research study are the PES/CS membrane have been prepared by dissolving the PES membrane with chitosan solution, PES membrane with different dosage of chitosan (0.00 %, 0.25 %, 0.75 % and 1.00 %) been characterized by their absorption by using Fourier Transform Infrared spectroscopy (FTIR) and Scanning Electron Microscope (SEM) and the performance PES membrane with various dosage of chitosan in treating POME wastewater been tested by determine initial and final biological oxygen demand (BOD), chemical oxygen demand (COD) and pH value.

1.5 Significance Study

This study is essential to confirm that the membrane technology provides much easier and faster than conventional method in POMSE wastewater treatment high concentration of chitosan will increase the performance of the membrane in treating POMSE wastewater.

2. Materials and Methods

2.1 Materials

The chemical substances used in this study are 1% acetic acid (R&M Chemicals, United Kingdom), N-methyl-2-pyrrolidone (NMP) (R&M Chemicals, United Kingdom), deionized water, sulfuric acid (H2SO4) (R&M Chemicals, United Kingdom), and sodium hydroxide (NaOH) (R&M Chemicals, United Kingdom). Besides, the wastewater sample used which is Palm Oil Mill Secondary Effluent (POMSE) wastewater were collected from Sime Darby, Pagoh Palm Oil Mill in Johor, Malaysia.

2.2 Synthesis of PES/CS Membrane

The synthesis of the membrane started by synthesizing the chitosan solutions. Firstly, 1 gram of chitosan dissolved in 100 mL of 1% acetic acid and placed in the 500 mL beaker and stir by using magnetic stirrer for 24 hours until the solution become homogeneous. Then, the PES been dissolved in the N-methyl-2-pyrrolidone (NMP) solvent. PES concentration fixed at 18.00 % while the chitosan solution added will be at 0.00 %, 0.25 %, 0.75 % and 1.00 % as shown in the Table 1 below. After that, the PES with addition of chitosan solution stirred at the temperature of 70 °C until homogenous to create dope solutions. Each of the dope solutions then undergoes casting process. The casting process will be done by pouring each of the solutions onto a petri dish and being flatten on the entire surface of the petri dish at 300 µm thickness. Then, the petri dish being dip into the coagulation bath that contained deionized water for 24 hours to allow solidification of membrane occur. At last, all of the membranes were heated at 60 °C in the drying oven to remove any trapped water or solvent from the membrane. The schematic diagram of the membrane preparation is shown in the Figure 1 below.

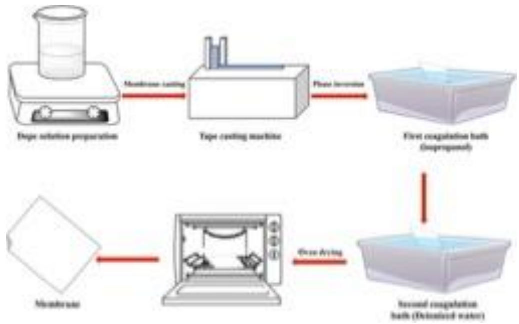

Figure 1: Membrane preparation flow [3]

2.3 Characterization of PES/CS Membrane

2.3.1 Scanning Electron Microscopy (SEM)

Scanning Electron Microscopy (SEM) been used to visualize the structure of the membrane surface with more details. Therefore, the porosity of the membrane can be observed by using SEM. The characterization of membrane by using SEM started with preparing the sample. Then, the SEM being set into the atmospheric pressure by press the and hold the button VENT. After that, the door been open and the sample holder carefully being grabbed and slide to the right. The blank sample from the holder being removed by loosen the screw and place the sample into the holder. The sample holder been slide back into its grooves on the sample stage and the door being closed. The button EVAC were pressed until it begins to blink to put the chamber back under vacuum. The electron beam being turned on and the picture of the membrane surface being taken using microscope software and computer.

2.3.2 Fourier Transform Infrared (FTIR)

Fourier transform infrared (FTIR) been used to characterize the chemical composition on the modification of the surface of PES/CS membrane. The characterization of membrane by using FTIR been done by using LUMOS II FTIR microscopy. The process been started by put the sample directly on the sample stage and then click the position on the computer and click next. After that, a single image of the viewed area being captured. The stage being moved with the controller, and “Add new border point” been clicked. After that, the background preferences been continued. After the background is measured, decide where to measure. Lastly, measurement preferences being selected to once more optimize acquisition time and measurement sensitivity and then measure sample being clicked. All collected visual and spectral data been presented in a new window which is the "chemical imaging view".

2.4 Membrane Performance Evaluation

The membrane performance evaluation been conducted by using filtration process using filtration unit (Figure 2). POMSE wastewater been fed into the filtration unit. The filtrates then being collected to undergoes quality water test.

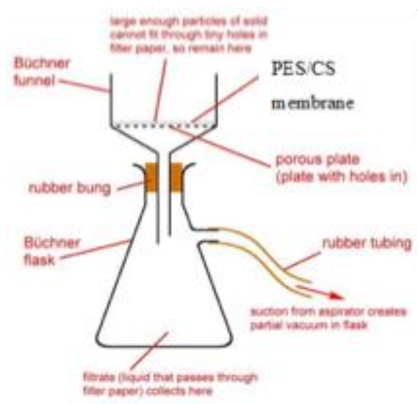

Figure 2: Filtration Unit [4]

2.5 Quality of the Wastewater Test

2.5.1 Biochemical Oxygen Demand (BOD)

The process of BOD determination being done by neutralizing the sample. In this step 1 N of sulfuric acid (H_2SO_4) or sodium hydroxide (NaOH) being added to the samples based on the sample pH value until the pH value reaches 7.0. Then, equal amount of sample being added in the BOD bottles. The initial dissolve oxygen concentration in each sample been determined by using dissolve oxygen meter and the results will be recorded. Then, all of the samples incubated in the incubator for five days at 20 ^oC. After five days, the samples been taken out from the incubator and the dissolve oxygen in the samples been determined. Based on the initial and the final reading of the dissolve oxygen, the BOD value of the samples being calculated by using Eq.1

$$BOD = \underline{DO_i - DO_f \;/\; Vs/Vb. \quad Eq.1}$$

Where,

BOD= Biochemical oxygen demand (mg/L)
DOi= Initial dissolve oxygen of the sample (mg/L)
DOf= Final dissolve oxygen of the sample (mg/L)
Vs= Volume of the sample (mL)
Vb= Volume of the bottle (mL)

2.5.2 Chemical Oxygen Demand (COD)

The chemical oxygen demand in each of the samples been determined by using the Hach DR6000 spectrometer. The sample first being blended while the reactor DRB 200 will be preheat at 150 ^oC. Then, the sample been pipetted into the vial. The sample then being placed in the DRB 200 for 2 hours. After 2 hours, the reactor needed to be turned off and the vial were let to be cool to 120^oC for 20 minutes. Then, the vial being put at the rack and let the vial cool in the room temperature. Next, by using colorimetric method, the stored program number for chemical oxygen demand being entered, high range (Press: PRGM). Then, press: 17 and ENTER until the display will show mg/L, COD and the ZERO icon. Then, COD/TNT Adapter being inserted into the cell holder by rotating the adapter until it drops into place. Then it being push down to fully insert it. The sample vial being placed in the adapter and being pushed straight down on the top of the vial until it seats solidly into the adapter. The vial ten being covered with the instrument cap. Lastly, READ button being pressed and the reading value being recorded.

2.5.3 pH

The pH of each sample been measured at the initial and after treated. pH meter being used to measure the pH of the samples. The pH of the sample before and after then being compared to identify the membrane effect towards the pH of the wastewater.

2.6 Time Analysis

The time taken to treat the wastewater by using the membrane is recorded and being compared with the time taken to treat wastewater using conventional method currently use to treat POMSE wastewater which is the trapping pond system.

3. Results and Discussion

3.1 Synthesis of PES/CS Membrane

PES/CS membrane with different concentration of chitosan which are 0%, 0.25%, 0.75% and 1% of chitosan dosage has successfully synthesized. Chitosan been chosen as the addictive in the membrane as chitosan is easy to be find and organic matter. Figure 3 below shown the membrane that have been synthesized which (a) is 0%, (b) is 0.25%, (c) is 0.75% and (d) is 1% of chitosan dosage in the synthesized membrane.

(a) (b) (c) (d)

Figure 3: Synthesized PES/CS membrane ((a) 0%, (b) 0.25%, (c) 0.75% and (d) 1% of chitosan dosage)

Based on the Figure 3 above, it is shown that all of the membrane shows same colour and texture. All of synthesized PES/CS membrane shows same white colours with paper-like texture. Therefore, further characteristics of the membrane need to be done by using SEM and FTIR.

3.2 Characterization of PES/CS Membrane

Figure 4 below shown the image of PES/CS membrane that been obtained from Scanning Electron Microscopy (SEM) with 1000 times magnification which (a) is 0.25 %, (b) is 0.75 %, and (d) is 1.00 % of chitosan dosage in the synthesized membrane.

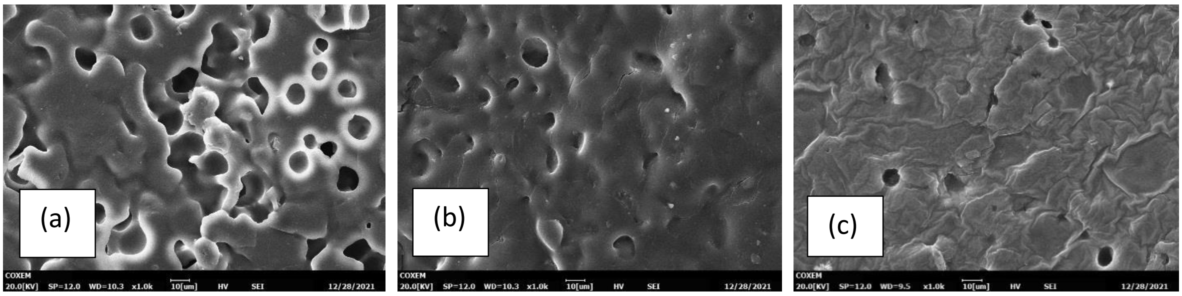

Figure 4: Image of PES/CS membrane surface ((a) 0.25%, (b) 0.75% and (c) 1% of chitosan dosage) by 1000 times magnification

Based on Figure 4 above it is shown that the porosity of the membrane decreases with the increase of the chitosan dosage. 0.25 % of chitosan dosage create more porous structure rather than 0.75 % and 1.00 % of chitosan dosage. Based on the studies made by [2], it is shown that in the comparison to the lower percentage of chitosan dosage, the image of PES/CS membrane with high chitosan dosage indicate a lower number of pores. This is because hydrophilic chitosan particles have been added. Increasing the chitosan particle concentration to 0.75 % and 1.00 % resulted in a viscous casting gel with a lower rate of phase inversion and a denser, more compact membrane [2]. The addition of 1.00 % of chitosan dosage increase the wall thickness of the membrane therefore decrease the porosity of the membrane. However, even though increase the percentage of chitosan dosage produced more compact structure of the membrane, the permeability of the membrane decreased when the addition of chitosan dosage reached 1.00 % [2].

Figure 5 below shown the FTIR spectra of PES/CS membrane that obtained. FTIR spectra been using to determine the chemical composition on the modified membranes.

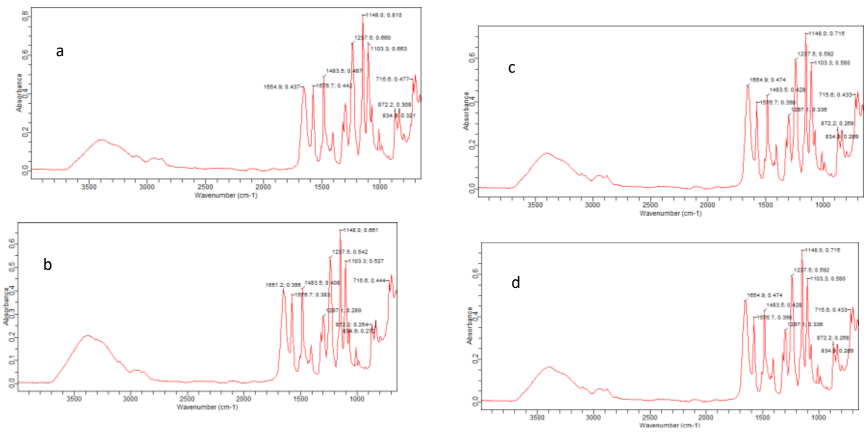

Figure 5: FTIR spectra of PES/CS membrane ((a) 0%, (b) 0.25%, (c) 0.75% and (d) 1% of chitosan dosage)

The PES sample was validated and is depicted in Figure 5. The C-stretching peak at 621 cm^{-1} is attributable to the aromatic ring structure, whereas the C=C stretching peak at 872 cm^{-1} is due to the aromatic ring structure. The sulfonyl (O=S=O) group is represented by the peaks at 1237.5 cm^{-1}, and 1483.5 cm^{-1}, whereas the aromatic ether (C-O-C) group is represented by the peak at 1297.1 cm-^{1}. The C-S stretching is shown by the strong peak at 715.6 cm^{-1}. All membranes had the same infrared pattern. On the surface of the PES membrane, chitosan which can be seen as O-H and NH2 stretches was discovered. It is shown between peak at 3100 cm^{-1} and 3700 cm^{-1}. Based on the figure above, in comparison to all of the membranes, PES/CS membrane with 0.25 %, 0.75 % and 1.00 % dosage of chitosan provided wide high intensity O-H and NH2 stretching rather than PES/CS with 0.00 % of chitosan dosage.

Besides, the O-H stretching structure that can be observed in PES/CS membrane with 0% of chitosan dosage can be observed above as there are a tiny quantity of water seems to have infiltrated and stayed within the porous structure. During the synthesis of membrane process, in order to allow full desorption, the membrane was placed in a coagulation bath containing deionized water and heated in drying oven at 60 °C to evaporate any trapped water or solvent. However, there might some amount of water trap in the membrane porous structure while in previous studies, PES membrane without chitosan does not show O-H stretching.

3.3 Quality of The Wastewater Test

3.3.1 Biochemical Oxygen Demand (BOD)

The biochemical oxygen demand values for each sample were presented in the Table 1 below while the percentage of BOD rejection were tabulated in Table 2 and presented in Figure 6.

Table 1: BOD Value for Untreated and Treated POMSE Wastewater Using PES/CS Membrane

Samples	BOD (mg/l)
Untreated POMSE	759.53
Treated POMSE with PES with 0% chitosan dosage	395.62
Treated POMSE with PES with 0.25% chitosan dosage	387.27
Treated POMSE with PES with 0.75% chitosan dosage	363.92
Treated POMSE with PES with 1% chitosan dosage	428.09

Table 2: Percentage of BOD Rejection of the Treated POMSE Wastewater using PES/CS Membrane

Treated POMSE with PES/CS Membrane	Percentage of BOD Rejection (%)
0% chitosan dosage	47.91
0.25% chitosan dosage	49.01
0.75% chitosan dosage	52.09
1% chitosan dosage	43.64

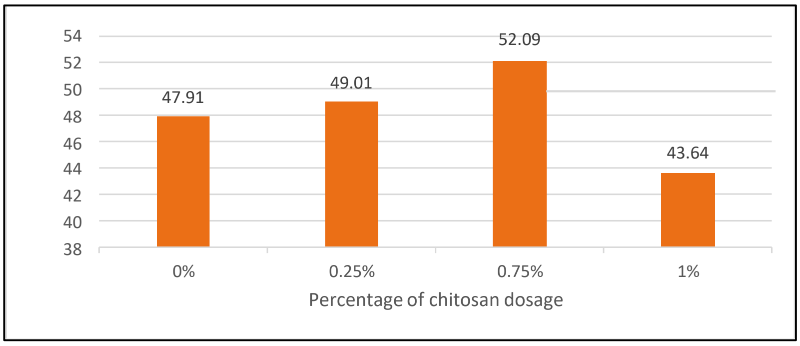

Figure 6: Comparison between The Percentage of BOD Rejection of Treated POMSE with PES/CS Membrane with different Percentage of Chitosan Dosage

Based on Figure 6, the percentage of BOD rejection for the treated POMSE wastewater increase with the increase of chitosan dosage. However, the percentage lowered when the chitosan dosage being added up to 1.00 %. The highest rejection value of BOD recorded at 52.09 % when the percentage of chitosan dosage added at 0.75 %. Meanwhile, the lowest percentage of BOD rejection value recorded at 43.64 when the chitosan dosage added to the membrane is at 1%. It might be because adding amount of chitosan up to 1.00 % resulted in the membrane effective pore sizes being blocked [2]. Besides, the degree of hydrophilicity of the membrane decreased as the chitosan dosage was raised up to 1.00 %. Increasing the concentration of chitosan particles to 1 wt% lowered membrane flow and rejection [2]. This might be due to molecular entanglement and aggregation, which results in the formation of a thick layer of chitosan and weaker pore walls with larger pore diameters [2]. This pore size increase allows pollutants to flow freely, resulting in lesser rejection [2]. However, even though the membrane can reduce the value of BOD, it is not reached DOE standard of discharge which at 100 mg/L. It might be due to the method choose in filtering the wastewater which is only by using basic filtration unit while based on research made by [2], the filtration method that been used to treat acid mine drainage wastewater is by using dead end filtration that is more accurate.

3.3.2 Chemical Oxygen Demand (COD)

The chemical oxygen demand values for each sample were presented in the Table 3 below while the percentage of COD rejection were tabulated in Table 4 and presented in Figure 7.

Table 3: COD Value for Untreated and Treated POMSE Wastewater Using PES/CS Membrane

Samples	COD (mg/l)
Untreated POMSE	1368.85
Treated POMSE with PES with 0% chitosan dosage	892.36
Treated POMSE with PES with 0.25% chitosan dosage	862.45
Treated POMSE with PES with 0.75% chitosan dosage	826.04
Treated POMSE with PES with 1% chitosan dosage	904.82

Table 4: Percentage of COD Rejection of the Treated POMSE Wastewater using PES/CS Membrane

Treated POMSE with PES/CS membrane	Percentage of COD rejection (%)
0% chitosan dosage	34.81
0.25% chitosan dosage	37.00
0.75% chitosan dosage	39.65
1% chitosan dosage	33.90

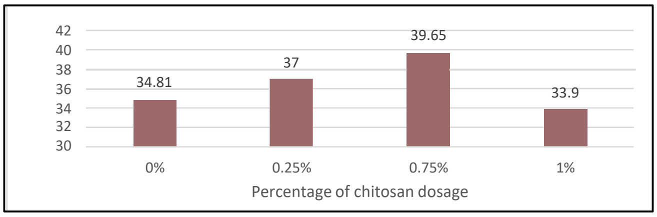

Figure 7: Comparison between The Percentage of COD Rejection of Treated POMSE with PES/CS Membrane with different Percentage of Chitosan Dosage

Based on Figure 7, the percentage of COD rejection for the treated POMSE wastewater increase with the increase of chitosan dosage. However, the percentage lowered when the chitosan dosage being added up to 1.00 %. The highest rejection value of COD recorded at 39.65 % when the percentage of chitosan dosage added at 0.75 %. Meanwhile, the lowest percentage of COD rejection value recorded at 33.90 % when the chitosan dosage added to the membrane is at 1.00 %. It might be because adding amount of chitosan up to 1.00 % resulted in the membrane effective pore sizes being blocked [2]. Besides, the degree of hydrophilicity of the membrane decreased as the chitosan dosage was raised up to 1.00 %. Increasing the concentration of chitosan particles to 1 wt% lowered membrane flow and rejection [2]. This might be due to molecular entanglement and aggregation, which results in the formation of a thick layer of chitosan and weaker pore walls with larger pore diameters [2]. This pore size increase allows pollutants to flow freely, resulting in lesser rejection [2]. However, even though the membrane can reduce the value of COD, it is not reached DOE standard of discharge which at 50 mg/L. It might be due to the method choose in filtering the wastewater which is only by using basic filtration unit while based on research made by [2], the filtration method that been used to treat acid mine drainage wastewater is by using dead end filtration that is more accurate.

4.4.3 pH

The pH values for each sample were presented in the Table 5 below and presented in Figure 8.

Table 5: pH Value of POMSE Samples Treated and Untreated

Samples	pH
Untreated POMSE	7.47
Treated POMSE with PES with 0% chitosan dosage	7.42
Treated POMSE with PES with 0.25% chitosan dosage	7.40
Treated POMSE with PES with 0.75% chitosan dosage	7.39
Treated POMSE with PES with 1% chitosan dosage	7.46

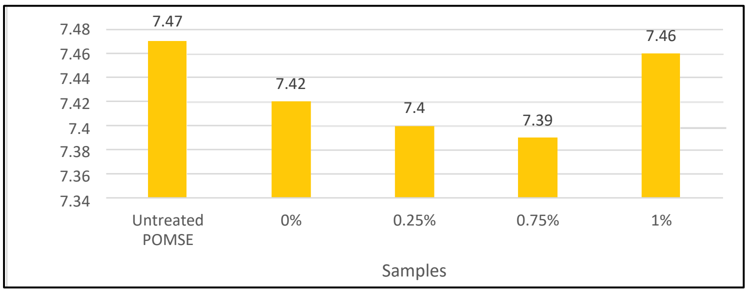

Figure 8: Comparison of pH Value between Samples

Based on Figure 8, the trend shows that the pH value for most of the samples are almost the same which lays between 7.3 and 7.5. The pH value of treated and untreated POMSE wastewater already reached DOE standard pH for wastewater discharge. The trend also shown that the value of the pH decreased until the value of 7.39 as the POMSE wastewater treated with PES/CS membrane with chitosan dosage 0.75 % however the pH value of POMSE wastewater treated by using PES/CS membrane with 1.00 % chitosan dosage shown that the pH value reduced only in small value. Therefore, shown that the efficiency of PES/CS membrane reached the limit when the percentage of chitosan dosage added up to 1%.

3.4 Time Analysis

Figure 9 below shown the time taken in days to treat POMSE wastewater using membrane technology and conventional method.

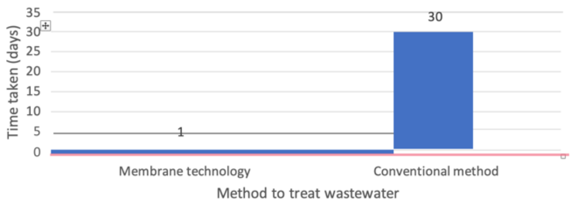

Figure 9: Comparison of Time Taken to Treat POMSE Wastewater Using Different Method

The time taken to treat the wastewater samples is faster than the conventional method based on Figure 7 above. The time taken to treat wastewater samples using membrane take a day while the conventional method which is oil trapping pond that takes about 115 days to treat POME wastewater and about 30 to 60 days to treat POMSE wastewater. Besides, oil trapping pond method require large area to be done and cannot be done in small quantity.

4. Conclusion

As the conclusion, the objective of the research study to synthesis the Polyethersulfone-Chitosan membrane with different dosage of the chitosan successfully achieved. The porosity of the membrane decreased with the increase of chitosan dosage. It is because increasing the chitosan particle concentration to 0.75 % and 1.00 % resulted in a viscous casting gel with a lower rate of phase inversion thus producing a denser and more compact membrane structure [2]. The addition of 1.00 % of chitosan dosage increase the wall thickness of the membrane therefore decrease the porosity of the membrane. Even though increase the percentage of chitosan dosage produced more compact structure of the membrane, the permeability of the membrane decreased when the addition of chitosan dosage reached 1.00 % [2]. The performance of the membrane with high concentration of chitosan increased until the chitosan dosage reached 1%. It is shown by the percentage of COD, BOD and pH rejection that increase when the chitosan dosage increases but however decrease when the chitosan dosage reached 1.00 %. It is because the degree of hydrophilicity of the membrane decreased as the chitosan dosage was raised up to 1.00 %. Increasing the concentration of chitosan dosage to 1.00 % lowered membrane flow and rejection [2]. This might be due to molecular entanglement and aggregation, which results in the formation of a thick layer of chitosan and weaker pore walls with larger pore diameters [2]. This pore size increase allows pollutants to flow freely, resulting in lesser rejection. Lastly, the time taken to treat the wastewater by using PES/CS membrane was only about one day which is lower than the time taken need to treat wastewater by using conventional method which is about 30 to 60 days to treat POMSE wastewater. However, even though the percentage of rejection shows decreasing trend when being treated with the PES/CS membrane, the value did not achieve the standard value of discharge by DOE. Therefore, in order to increase the efficiency of the experiment and achieve the standard discharge vale by DOE in the future studies, the method chosen for the wastewater filtration need to be change into better filtration method. As in this study, the method use for wastewater treatment is the basic filtration, the efficiency of membrane filtration can be increase by using dead end filtration unit or by combining the membrane with reactor to treat wastewater and eventually will reached the standard discharge value of wastewater by DOE. Besides, the characterization of the membrane can also be done by determine the contact value of the membrane, tensile strength of the membrane and also the image of the cross section of the membrane. Lastly, for better determination of the membrane performance in future studies, the experiment related to determination of heavy metal rejection in the wastewater should be done.

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