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Chemical cleaning of fouled polyethersulphone membranes during ultrafiltration of palm oil mill effluent

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Abstract. Fouling is one of the critical factors associated with the application of membrane technology in treating palm oil mill effluent (POME), due to the presence of high concentration of solid organic matter, oil, and grease. In order to overcome this, chemical cleaning is needed to enhance the effectiveness of membranes for filtration. The potential use of sodium hydroxide (NaOH), sodium chloride (NaCl), hydrochloric acid (HCl), ethylenediaminetetraacetic acid (EDTA), and ultrapure water (UPW) as cleaning agents have been investigated in this study. It was found that sodium hydroxide is the most powerful cleaning agent, the optimum conditions that apply are as follows: 3% for the concentration of NaOH, 45°C for temperature solution, 5 bar operating pressure, and solution pH 11.64. Overall, flux recovery reached 99.5%. SEM images demonstrated that the membrane surface after cleaning demonstrated similar performance to fresh membranes. This is indicative of the fact that NaOH solution is capable of removing almost all of the foulants from PES membranes.

Keywords: cleaning agent; flux recovery; pressure; temperature; concentration; pH

1. Introduction

For the past few years, membrane technology has been the focus of recent scientific advances in industrial processes due to its capacity to operate at room temperatures and its relatively low energy consumption (Rai *et al*. 2006). Despite these benefits, the fouling of the membranes remains the most prominent problem that is associated with its application, especially in wastewater treatment (Martín-Pascual *et al*. 2014, Masmoudi *et al*. 2014). IUPAC defines fouling as “is a process where membrane lost its performance due to the deposition of suspended or dissolved substances on its external surfaces, at its pore openings or within pores”. Accumulation of foulants on the membranes’ surface highly influences the lifetime of membrane and reduces the permeate flux, which increases the required pressure and energy for the membrane to operate (Lim

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and Bai 2003, Zhang and Liu 2003).

Palm oil mill effluent (POME) is a waste product generated from the palm oil mill industry. The extraction of crude palm oil from the fruit requires a huge amount of water. It is estimated that 5-7.5 tonnes of water is required to produce 1 tonne of crude palm oil. More than 50% of the water ends up as POME. The conventional treatment of POME generally consists of a combination of physical and biological treatments. For examples, natural coagulation (Shak and Wu 2014, Teh *et al*. 2014), vermitechnology (Lim *et al*. 2014), bio-adsorption (Mohammad and Chong 2014) were recently proposed for enhancing POME treatment. The detail information about POME treatment is explained by Wu *et al*. (2010).

In recent years, membrane technology has been applied for POME tertiary treatment (Wah *et al*. 2002, Ahmad *et al*. 2003, Wu *et al*. 2007, Yejian *et al*. 2008, Wu *et al*. 2009, Said *et al*. 2014) in order to improve the effluents quality. The membrane system has been shown to be able to significantly reduce the BOD and COD to acceptable levels set by regulatory agency. However, the problem of fouling remains, as fouling reduces the performance of the membranes, and causes it to require frequent maintenance, as fouling clogs the surfaces pore. This makes it vital that an accurate and detailed study be conducted to determine the efficiency and performance of the membranes' cleaning process.

Membrane foulants can be broadly classified into two categories i.e.,: reversible and irreversible foulants. Reversible foulants can be eliminated by physical and mechanical cleaning, such as filter back flushing, ultrasonic, rotary or vibratory shear enhanced, while irreversible foulants cannot be easily removed via conventional methods. This necessitates the advent of chemical method for the purpose of removing irreversible foulants (Feng *et al*. 2006, Al-Amoudi 2007, Mostefa *et al*. 2007, Salladini *et al*. 2007, Cai *et al*. 2010, Madaeni and Samierad 2010, Puspitasari *et al*. 2010, Shi and Benjamin 2011).

Generally, suitable chemical agents can be divided into four types i.e. bases, oxidants, acids and chelating agent (Porcelli and Judd 2010). Caustic solution is usually used as base to remove organic and microbial foulants. During the cleaning process, the caustic solution increases the pH of the solution, and as a result, also increased the negative charge and solubility of organic foulants via hydrolysis and solubilisation. Chlorine and hydrogen peroxide are the most common oxidants used in the membrane cleaning process. The addition of oxidants increases the hydrophilicity of the membrane and thereby, reduces the adhesion of foulants onto the membrane. Acid and chelating agents are mainly used to remove the scales and metal dioxides. For example, citric acid is effective in removing iron dioxide by complexing with iron and finally precipitating them. EDTA is a common chelating agent used to improve the membrane cleaning process.

Various studies have been reported on the effect of various parameters on the cleaning efficiency. Generally, these can be divided into three factors: the properties of the chemical agent, the membrane characteristic and operational conditions of the cleaning process. The successful removal of foulants from the membrane surface was commonly measured in terms of the recovery of the permeate flux. Results were supported using Scanning Electron Microscopy (SEM) and Infrared Spectroscopy (IR).

Ang compared the effectiveness of Sodium hydroxide (NaOH), Sodium chloride (NaCl), Sodium Dodecyl Sulphate (SDS), and Disodium ethylenediaminete-tetraacetate (Na_2-EDTA) to clean fouled RO membranes. The results showed that EDTA has a higher efficiency (95%) compared to other chemical agents (79-84%). In addition, adjusting the pH of chemical agent could also increase the cleaning efficiency. NaOH efficiency was increased from 47 to 76%, while that of EDTA, from 35 to 91% (Ang *et al*. 2011a).

Puspitasari used sodium hypochlorite (NaOCl) to remove foulants on a Polyvinylidene fluoride (PVDF) membrane. The results showed that increasing concentration of NaOCl increases the cleaning efficiency. The highest efficiency, i.e., 95%, was realized by using a 1% NaOCl solution. Further increase in the concentration of NaOCl (5%) had no pronounced effect on the cleaning efficiency. The cyclical cleaning time was investigated as well (Puspitasari *et al*. 2010).

Vaisanen used two types of ultrafiltration membranes: i.e., hydrophilic (C 30F) and hydrophobic (PA 50H and PES 50H). They varied the chemical cleaning agents (NaOH, HNO_3, *Ultrasil 11* and *Libranone 960*) to remove the whey protein solution (WPS) and ground wood mill circulation water (GWCW) from a pulp and paper mill effluent. The results were divided into two sections. First, for the membranes fouled with WPS, the 0.075 wt% of NaOH solution demonstrated the best cleaning efficiency, at 88%. Second, after the membrane that was fouled by GWCW had been cleaned with *Liberanone 960*, the permeability increase when rinsing began (Vaisanen *et al*. 2002).

Thus, the main aim of this study is to determine the best chemical agent for cleaning the membrane after its treatment with the Palm oil mill effluent (POME). Four cleaning agents were tested in this study, i.e., Sodium hydroxide (NaOH), Hydrochloric acid (HCl), Ethylenediaminete-tetraacetate (EDTA) and Ultra-Pure Water (UPW). The effectiveness of cleaning process was represented by the percentage of flux recovery (FR) and Resistance removal (RR). The study will also try to optimize the various experimental conditions i.e., pressure, temperature, concentration of cleaning agent, and pH to reach the highest flux recovery.

2. Material and methods

2.1 Material

A hydrophobic membrane made from Polyethersulfone membrane (SelRo MPF-U20-P) with a molecular weight cut-off (MWCO) of 25,000 Da was used for the experiments. The membrane was purchased from Sterlitech Corporation. The stirred cell (Amicon 8200, Millipore.co, USA) was used with a single blade stirrer equipped with an acrylic solution reservoir of 1000ml. Scanning Electron Microscopy (SEM, Gemini model SUPRA 55VP-ZEISS) was used to analyse the surface morphology of the membrane. All the chemicals used were of Analytical grade. NaOH, EDTA, and HCl were purchased from Friendemann Schmidt Chemical, while NaCl was procured from J.Kollin Chemicals, utilized without further purification. Partially treated POME (after the biological treatment) was collected from West Palm Mill Oil in Carey Island, Malaysia. The characteristics of the partially treated POME are listed in Table 1.

Table 1 Characteristics of partially treated POME

Parameter	Feed sample
COD (mg/L)	12,040
TSS (mg/L)	3103
Colour (PtCo)	54,200
Turbidity (NTU)	23,750
pH	7.43

2.2 Methods

2.2.1 Analysis

The POME samples were characterized to determine the content of Chemical Oxygen Demand (COD), Total Suspended Solid (TSS), colour, and turbidity levels by using a Hach DR/2010 spectrophotometer. The average value was taken from three repeated experiments. Detailed composition and features of the waste water are summarised in Table 2.

The rejection in the solutions' parameters after membrane experiments was calculated using the following equation

$$R(\%) = \left(1 - \frac{C_p}{C_s}\right) \times 100 \quad (1)$$

where C_p is the concentration in the permeate solution, while C_s is the concentration in the feed sample.

2.2.2 Fouling and cleaning operation

All of the experiments were carried out using the Amicon 8200, Millipore co. USA. First, the fresh membrane was placed at the bottom of a stirred cell. The stirred cell was first filled with Ultra-pure water (UPW). The permeate flux was measured and labelled as J_{wi}. After the permeation experiment with the flux decline stage, the fouled membrane was rinsed with ultra-pure water to remove the particles. The permeate flux for fouled membrane was measured using UPW, and labelled as J_{wf}. For the cleaning stage, the fouled membrane was reassembled upside down at the bottom of the stirred cell. Then stirred cell was locked and filled with the designated cleaning agents. The cleaning process took 30 minutes. At the first stage, four type of chemical cleaning agents were applied i.e., UPW, HCl, EDTA and NaOH. The chemical cleaning agent that gains the highest flux recovery was selected for use in subsequent experiments. The experiment was continued with varied concentration (1-4% m/v), pressure (2-5 bars), temperature (35-50°C), and pH (3.15-11.64). Once the cleaning process is completed, the membrane was washed again with water, and permeability tests were carried out with UPW, with the flux labelled as J_{wc}. The Trans membrane pressure (Pa) is designated as ΔP, and the viscosity of the solution is μ ($Nm^{-2}s^{-1}$).

Membrane resistance (R_m) can be calculated from the equation

$$R_m = \frac{\Delta P}{\mu \sum J_{wi}} \quad (2)$$

The resistance, which appear after fouling (R_f), can be measured from the equation

$$R_f = \frac{\Delta P}{\mu \sum J_{wf}} - R_m \quad (3)$$

The resistance, which remained after cleaning (R_c), can be estimated from the equation

$$R_c = \frac{\Delta P}{\mu \sum J_{wc}} - R_m \quad (4)$$

The overall resistance removal (RR) can be determined using the formula

$$RR(\%) = \left[\frac{\left(R_f - R_c\right)}{R_f} \right] \times 100 \tag{5}$$

The Flux recovery (FR) was calculated using the formula

$$FR(\%) = \left[\frac{\left(J_{wc} - J_{wf}\right)}{\left(J_{wi} - J_{wf}\right)} \right] \times 100 \tag{6}$$

The resistance removal and flux recovery have been used to determine the effectiveness of the chemical agent in cleaning the membranes (Moghadam and Mohammadi 2007, Mohammadi *et al.* 2003).

3. Results and discussion

3.1 Membrane fouling

The flux decline and fouling analysis is shown in Fig. 1. The two different concentrations (i.e., 0.05 M and 0.1 M) of NaCl were added in the POME solution. As shown in Fig. 1, the permeate flux is higher for pure POME solution compared to the solution that was added with NaCl.

For the first hour, a very sharp decline in flux was observed prior to a slight decrease before finally reaching a constant value. This phenomenon can be explained as follows: when NaCl is added to the solution, there is a reduction of repulsion force between the particles in the POME solution and the surface of the membrane. The attraction between particles and membranes lead to the accelerated accumulation of foulants on the membrane's surface, which finally causes the formation of a thick cake layer.

It was also observed that the high ionic strength increases the rate of rejection compared to its lower counterpart. Increased concentration of NaCl influenced the hydrophobic character of the membrane. When the concentration of NaCl was increased, the hydrophobic character in the membrane increased as well. The hydrophobic character meant that the membrane was more attracted to bind with the particles.

NaCl in the solution will be split into Na^+ and Cl^- ions. Natural organic matter (NOM), which is represented by the particles in POME, is generally negatively charged. When Na^+ ions come into contact with NOM, it will initiate ionizing reactions. The Na^+ ions will attach itself to NOM, neutralizing its charges. This will eventually reduce the repulsion force among NOM. Na^+ ions also serves as an ionic bonding bridge between the NOM molecules. NOM molecules combine to form a larger molecule, called macromolecules. In addition, Na^+ ions are also attached to the membrane's surface due to PES's negative charge. The attachment of Na^+ ions onto the surface of the membrane causes this charge to be converted to positive charge. This change reduces the repulsion between NOM and the membrane. This will cause the NOM macromolecules to be attracted and attach itself to the membrane's surface. The deposit of macromolecules takes place continuously, layer by layer, and will form a compact, dense, and thick layer of cake. The cake layers cannot be cleaned with only water, and requires the addition of chemicals (Al-Amoudi 2010).

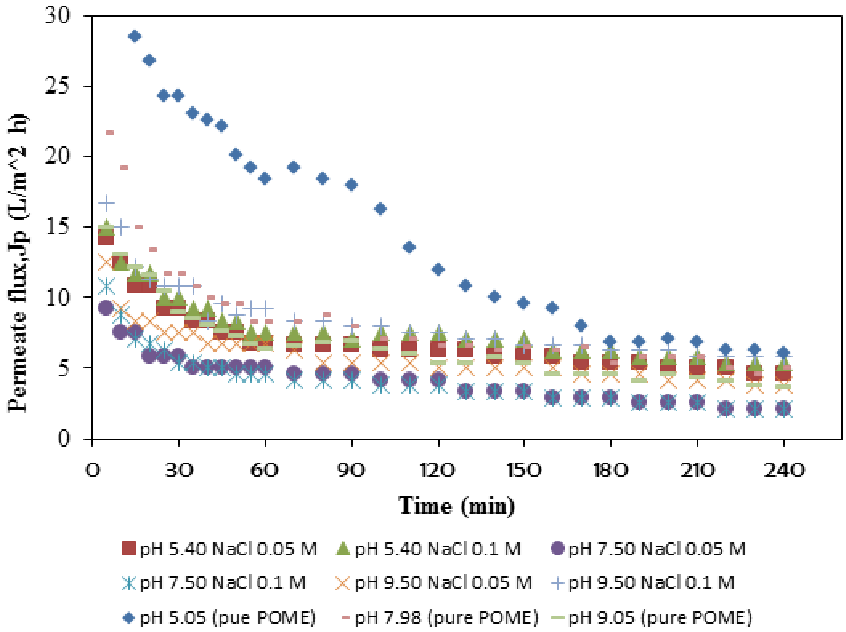


Fig. 1 Flux decline on the variations of pH and addition of Ionic strength

3.2 Type of cleaning agent

Fig. 2 shows the flux recovery of the different cleaning agents. From the figure, NaOH shows highest cleaning efficiency of 99.9% compared to other reagents, such as 24% for UP Water, 38% for NaCl, 63% for HCl, and 72% for EDTA. Cleaning with UP water only resulted in 24% of flux recovery, which indicated that irreversible fouling occurred. It can be concluded that the foulants in POME solution have strong chain bonding with the surface of the membrane. Due to this fact, it requires the addition of chemical agent to restore the permeate flux of the membrane.

In this experiment, four type of cleaning agents were used for cleaning process of fouled membrane. Acid cleaning is usually used to remove the hardness salts and metal hydroxides (Porcelli and Judd 2010). Since POME did not contain any metallic ions, it is expected that cleaning membranes using HCl will not provide suitable results. EDTA is a chelating agent with six molecule positions, which could contribute to the chemical reaction of hydrogen bonding and complex with the material on the surface of membrane (Mohammadi *et al*. 2003). EDTA reduces the concentration of Na^+ via complexion. Furthermore, the ligand exchange reactions between EDTA and NOM-Na^+ would eliminate the NOM-Na complex, which will increase the electrostatic repulsion between the NOM and the membrane. This will ultimately lead to the removal of NOM from the membrane's surface (Hong and Elimelech 1997).

An alkaline solution is regarded as a superior cleaning agent compared to Acid. This is due to the presence of OH^- ions in the caustic solution, which enhances disorder in the foulants' layer. NaOH increases the ionic strength and solubility of foulants as the pH is increased. Increase of pH implies increasing the negative charge of organic matters (Madaeni and Samieirad 2010). NaOH is alkaline, having high pH value (pH 11). The negative charge of the solution increases with the addition of NaOH. The negative charge of the solution meets the negative charge of the PES

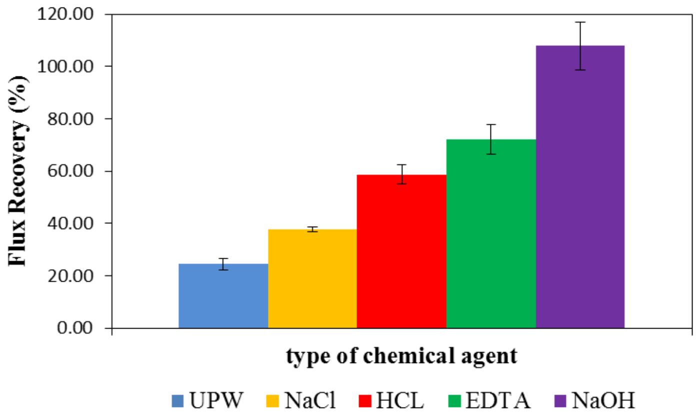
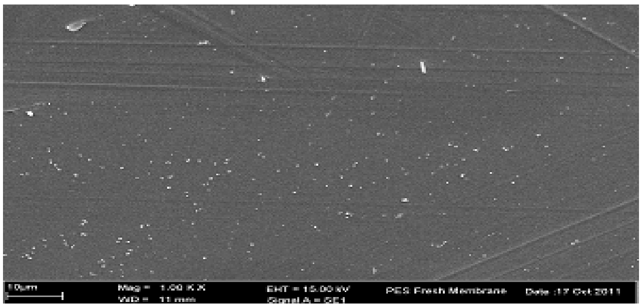
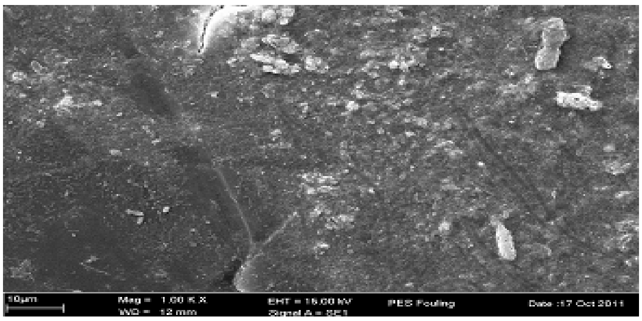


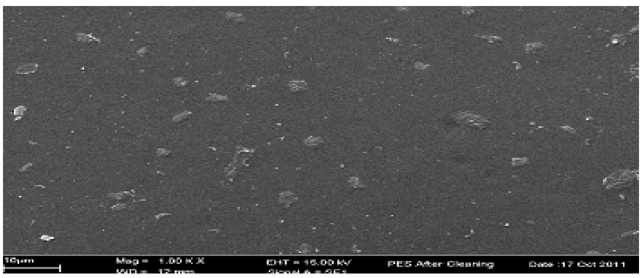
Fig. 2 The flux recovery at various cleaning agent



(a)



(b)



(c)

Fig. 3 SEM images of the membrane surface: (a) fresh membrane; (b) fouled membrane; (c) cleaned membrane membranes. As a result of this, a repulsive force is generated between the solution and PES membrane, which releases the foulants from the membrane's surface.

SEM images were taken to observe potential changes on cleaned membrane surface using NaOH, as shown in Fig. 3. From Fig. 3(a), fresh membrane is seen clean and free from particles/foulants, but in Fig 3b, the large number of flocs and roughness on membrane surface was observed, which proves the inefficiency of water cleaning. We found lesser foulants on the membrane surface when it was cleaned with the NaOH solution, as shown in Fig. 3(c). Similar results were observed by Wu *et al*. (2007) whereby all membranes after ultrafiltration of POME showed denser structure of pores, which might indicate the occurrence of pore plugging if comparing to the image of fresh membrane.

3.3 Effect of concentration of NaOH solution

The effect of varied concentration of NaOH on the flux recovery of membrane was studied in the range of 1-4%. The results are shown in Fig. 4. The maximum flux recovery of 99.9% was obtained using 3% of NaOH. But the flux recovery slightly decreased to 88% as the concentration increased to 4%. This phenomenon was assumed to be due to the higher rate of particles depositing on the surface of the membrane, which caused increased levels of cake thickness. This renders the cleaning process to be not as effective. In a general sense, it can be concluded that higher concentrations of NaOH resulted in higher cleaning efficiencies. This result is similar to findings of other researchers (Madaeni and Mansourpanah 2004, Madaeni and Samierad 2010, Puspitasari *et al*. 2010).

3.4 Effect of different pressure

Pressure plays an important role in the cleaning process. The effect of pressure on the cleaning process of the membrane is shown in Fig. 5. From the figure, a higher flux recovery was obtained with the increase in pressure. Increasing the pressure means increasing the hydrodynamics of mass transfer from the fouling layer to the bulk solution. This has allowed the collision of particles on

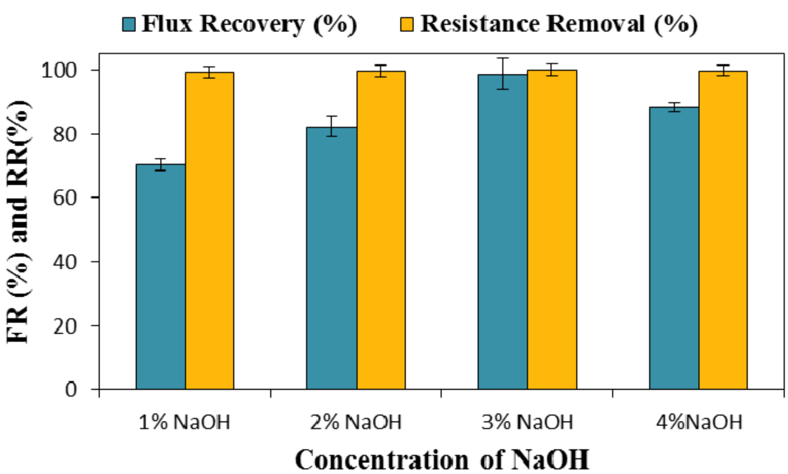


Fig. 4 Average of flux recovery of various NaOH concentrations

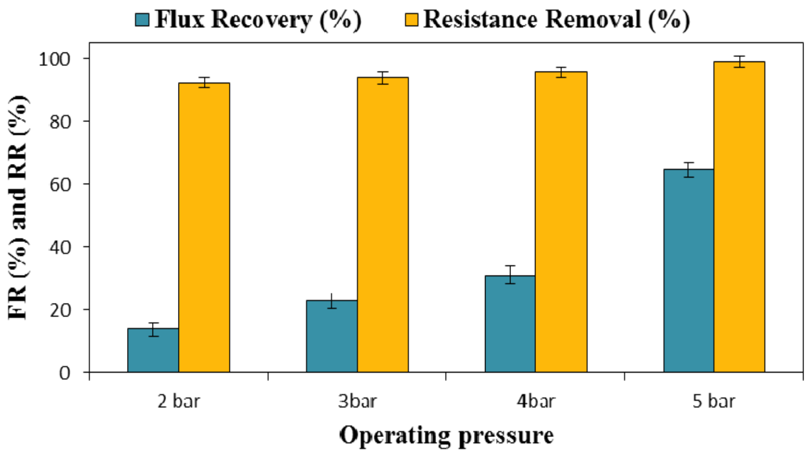


Fig. 5 The flux recovery at different pressure

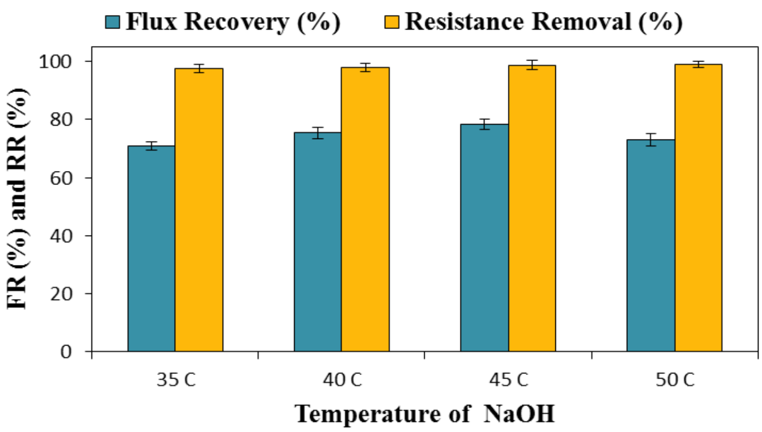


Fig. 6 The flux recovery at different temperature

the membrane surface to take place, which leads to the breakdown of particles into smaller sizes, leading to their easy removal.

Thus, the fouling that occurred was expected to be only a weak accumulation of particles on the membrane surface, and did not enter the pores of the membrane. However, for POME treatment by PES membrane, POME is complex waste water that contains many suspended solid and other pollutants, which makes complete blockage a possibility. It is characterized by a strong attachment of fouling layer onto the surface and inside the pores of the membrane. Fouling by stacking layer upon layer of particles onto the membrane surface resulted in the formation of a dense layer, which makes it more difficult to clean. To overcome this problem, it required a strong pressure from the flow of cleaning chemicals. As seen in Fig. 5, the optimum pressure is 5 bars.

3.5 Effect of different temperature

As shown in Fig. 6, the effect of temperature on the cleaning process was almost negligible.

The flux recovery ranged from 71-76%. This phenomenon was related to the properties of the membrane used. PES membrane have a contact angle value of between 58.50-59.75, and can be assumed to be moderately hydrophobic (Amin *et al*. 2010). It means that PES membranes have greater likeability in absorbing foulants compared to water. In addition, the sulphonyl group with a ring structure renders the PES membrane with a rigid structure and decreased flexibility. The results showed that increasing the temperature influences the flux recovery. Increasing the temperature increases the chemical reaction rates between NaOH and foulants.

The increasing temperature may reduce the viscosity of the solution, which simplifies the transfer of particles from the membrane to the bulk solution (Bird and Barlett 2012). However, the membrane possesses certain heat resistance properties. Therefore, it is recommended that the washing of membrane using a cleaning agent be carried out at temperatures below 45°C (Al-Amoudi and Lovitt 2007).

3.6 Effect of different pH

The effect of pH on flux recovery was tested at pH of 3.15-11.64. It is evident from Fig. 7 that as the pH increased, the flux recovery increased from 60.5% to 99.5%. The results showed that the optimum pH was about 11.64 (99.5%).

Increasing the pH values lead to the increase in the number of OH^- ions. The mechanism of membrane cleaning by NaOH is strongly influenced by the presence of OH^- ions. Hydroxyl ion in the solution will attract Na^+ ions from the membrane's surface, increasing the negatively charged nature of the membrane. The consequence of this is that the repulsive interaction among the foulants and PES membrane increased, leading the foulants to leave the membrane surface (Ang *et al*. 2011b).

Even though the flux recovery reached almost 100% (99.5%), the damage to the membranes was minimal. This was evident from the performance of the membranes after the cleaning was carried out. Table 2 shows that the rejections of major parameters remained similar to those prior

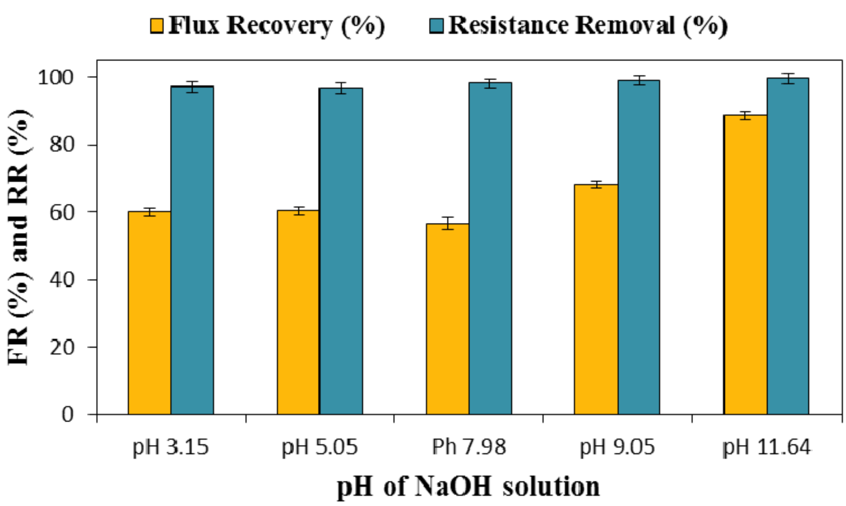


Fig. 7 The flux recovery at different pH of NaOH solution

Table 2 Parameter of POME after treatment with PES membrane

Parameter	Feed sample	Permeate	Rejection (%)
COD (mg/L)	12,040	1262.5	89.5
TSS (mg/L)	3103	140	95.5
Colour (PtCo)	54,200	5387.5	90
Turbidity (NTU)	23,750	612.5	97.5
pH	7.43	8.2	----

to cleaning. The rejections of COD, TSS, Color, and turbidity achieved were 89.5; 95.5; 90; 97.5%, respectively.

4. Conclusions

In this study, the fouling of ultrafiltration membrane (PES) was investigated using various cleaning agent after the treatment of POME. The results showed that sodium hydroxide is the most powerful cleaning agent due to the similarity in charges between NaOH and the surface of PES membrane. This condition increases the repulsive force between foulants and membrane surface, which ultimately increase cleaning efficiency. Overall, recovery of flux reached 99.5%. Various experimental conditions were optimized i.e., pressure, temperature, concentration of cleaning agent, and pH, which plays a vital role in flux recovery. Higher pressure, pH, and concentration of NaOH resulted in higher cleaning efficiencies. However, within the range investigated, temperature had an almost negligible influence in the cleaning process. The optimum conditions are found as follows: 3% for the concentration of NaOH, 45°C for temperature solution, 5 bar operating pressure, and solution pH 11.64. SEM images of the cleaned membrane were compared to the fresh and fouled membrane, and it was indicated that most of the foulants were successfully removed via chemical cleaning.

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