Study on the separation of phospholipids from crude palm oil using a polyethersulfone ultrafiltration membrane

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Abstract. Crude Palm Oil (CPO) extracted from the palm fresh fruit bunches (FFB) should be refined to meet the specified quality for edible oils. One of the components that are removed in the CPO refining process is a phospholipid. Phospholipids are undesirable because they form mucus (gum) in CPO and enhance the emulsion formation during oil processing. As one of the emerging separation processes, membrane technology could be proposed on phospholipid removal in CPO. The research objectives are to synthesize polyethersulfone (PES) ultrafiltration (UF) membrane and evaluate its performance on phospholipid removal. Besides, the effects of temperature and pressure on UF-PES membrane performance in phospholipid removal were also studied. The UF-PES membrane was prepared by phase inversion method with the composition of PES polymer of 17.5%; Polyethylene Glycol (PEG) polymer as much as 2.5% as pore-forming agent; and 80% (w/w) of N-Methyl Pyrrolidone (NMP) as a solvent. Two types of the UF-PES membrane have been synthesized, namely M1 and M2 membranes. Furthermore, the M1 membrane was selected for the filtration process due to its high resistance to pressures up to 3 bar. The result indicated that the pressure (T) and temperature (S) significantly affected phospholipid removal. The lowest phospholipid removal was obtained in the S2T2 treatment, about 78%. Moreover, the S3T1 and S3T2 treatments gave a stable permeation flux in comparison to the others treatment.

1. Introduction

Palm oil is a semi-solid fat that has a fixed composition [1]. However, according to Mangoensoekarjo and Semangun [2], the crude palm oil (CPO) and palm kernel oil (PKO) obtained from the milling process did not meet the specified water and impurities content specifications. Therefore, one of the components removed in the CPO purification process is phospholipids or phosphatides components. The phosphatide component will form mucus (gum) on CPO or hydrated triglycerides, which promote emulsion formation during the oil processing [3].

The refining by physical and chemical processes has been widely used for various vegetable oil productions [4]. Moreover, the conventional oil refining method leaves several drawbacks, including high-energy consumption and loss of essential nutrients from refined oil. Conventionally, degumming is the process of forming flocks from colloidal substances in crude oil. For instance, the chemical processes are adding an acid such as H₃PO₄, H₂SO₄, or HCl. However, the acid will precipitate the
components such as proteins, phosphatides, and resins in crude oil, needing further separation (i.e., adsorption). During the degumming process with alkaline caustic, the soap particles formed will absorb mucus substances or pigments, which form an emulsion of soap and cause oil losses.

Purification and separation processes using membranes are currently the most efficient option. This technique has been widely used in the food industry to purify juices and the concentration of milk proteins. Recent developments have reported an increase in the use of membranes for various vegetable oil processing [5]. For example, palm oil refining by a membrane process has low emissions and energy consumption, can reduce oil loss, does not require bleaching media, and does not generate wastewater [6].

The researcher has extensively studied phospholipid degumming by ultrafiltration (UF) membranes. Among them are de Moura et al. [7], who stated that using UF membranes made of polyethersulfone (PES) material in soybean oil degumming can remove phospholipids up to 89% at a temperature of 50°C and a pressure of 0.5-5 bar. Koris and Marki [8] removed the phospholipid in crude soybean oil using ceramic membranes TI-70-100 and TI-01070-20, which operated at a temperature of 45°C and a pressure of 1-5 bar. Phospholipid retention obtained reached 97% on membrane type TI-01070-20 and 47% on membrane type TI-70-100 at the same pressure of 2 bar. Aryanti et al. [9] used flat PES membranes in CPO purification with phospholipids rejection in the range of 73.94%, 86.60%, and 99.21% at temperatures of 30°C, 35°C, and 40°C, respectively.

The separation of phospholipids from CPO is an attempt to produce better quality CPO. The CPO purification process has been carried out physically and chemically, but it still has various shortcomings. The total phospholipids present in CPO have not been removed in the above studies, so further research is necessary. In this study, the membrane used was a 17% polyethersulfone (PES) ultrafiltration (UF) membrane made using NMP as a solvent. PES polymers are widely used and easy to synthesize for ultrafiltration and microfiltration membranes. Besides, PES is stable at high temperatures up to 210°C. In addition, PES has a wide pH tolerance both during manufacture and during the application, has good resistance to chlorine, and is easy to manufacture membranes with various configurations and modules [10]. This study aims to determine the characteristics of the UF-PES membrane made by phase inversion and evaluate its performance of the UF-PES membrane on the separation of phospholipids in CPO-isopropanol mixtures feed solution. In addition, the effects of temperature and pressure on the phospholipid removal using the filtration process on the CPO-isopropanol mixtures feed solution were studied in detail. We expect the synthesized PES ultrafiltration membrane can effectively separate phospholipids from CPO with high retention, improving the CPO quality as edible oil.

2. Materials and methods

2.1. Materials

Crude palm oil (CPO) obtained from Cot Girek palm oil mill was used as feed solution mixed with Isopropanol with the weight ratio of 50:50 (%w/w). PES polymer purchased from BASF (Germany) used a base-membrane polymer. N-Methyl pyrrolidone (NMP) purchased from Merck (Germany) was used as a solvent for PES polymers. The series of polyethylene glycol (PEG) with various molecular weights (MW) of 20.000; 40.000, and 100.000 Dalton (Da) purchased from Sigma Aldrich (Germany) were used for membrane pore size determination. Distilled water was used for the membrane coagulation bath and pure water permeation test. Ammonium molybdate (H8MoN2O4), sodium ammonium vanadate (H4NNaO6V2), and potassium dihydrogen phosphate (KH2PO4) were used for phospholipid analysis.

2.2. Preparation of flat sheet ultrafiltration (UF) membrane

PES UF membrane was prepared by phase-inversion method with the composition of polymer solution consisting of 17.5% PES polymer, 2.5% PEG 400, and 80% NMP solvent (% by weight). The PES UF
membrane synthesized steps were as follows: Firstly, the PES polymer was dissolved into NMP solvent and then added with PEG 400. Next, the solution was stirred vigorously until the homogeneous solution was obtained. The solution was kept at room temperature for 4 hours to remove air bubbles. Then it was poured onto a glass plate and cast with a casting knife. The casted polymer solution was kept at room temperature for 30 seconds to allow the solvent to evaporate before immersion into a coagulation bath. There are two types of coagulation baths used; those are (1) deionized water (DI) water and (2) an equal mixture of DI waters-isopropanol (%v/v). Furthermore, the film membrane was detached from the glass plate after immersion into the coagulation bath was then removed and kept stirred into DI water for ± 12 hours to remove the unreacted polymer and solvent. Finally, the film membrane layer was kept in DI water before membrane characterization.

2.3. Membrane Characterization

Membrane characterization was carried out to determine the best membrane for the degumming process to filtrate CPO-isopropanol mixtures as the feed solution. Membrane characteristics tested in this study, such as the pure water permeation and Isopropanol permeation, determination of functional groups using Fourier Transform Infra-Red (FTIR), water uptake, determination of membrane pore size using the MWCO (Molecular weight cut off) method.

2.3.1. The membrane pure water permeation and Isopropanol permeation. The water permeation was conducted by passing deionized (DI) water through the flat sheet PES membrane, which is mounted, in the dead-end module at the series of trans membrane pressure (TMP) of 1.0; 1.5; 2.0; 2.5 and 3.0 bar. Then the permeate (filtrate) solution was collected and weighed every 5 minutes until the steady-state condition was reached. Membrane flux (Jv) was calculated according to equation (1), and the pure water permeability (Lp) was determined by equation (2). The Lp data obtained from equation (2) was then compared to Lp data from literature [11] to categorize the prepared membrane.

\[ J_v = \frac{V}{A t} \]  

(1)

Where \( J_v \) is the membrane flux (l/m².h), \( V \) is the volume of permeate solution (l), \( A \) is the effective membrane area (m²), and \( t \) is the permeation time (h).

\[ L_p = \frac{J_v}{\Delta P} \]  

(2)

While the pure membrane permeability \( L_p \) is in l/m².h.bar, and \( \Delta P \) is the operating pressure (bar). Therefore, the isopropanol filtration was similar to pure water permeation procedures, except the feed solution was changed to Isopropanol.

2.3.2. The membrane functional group analysis and membrane water uptake. The IR spectra of the UF-PES membrane (M1 and M2) were analyzed by using ATR-FTIR-8100A (Shimadzu Co. Ltd., Japan). The IR spectra were recorded in the range of 800-4000 cm⁻¹. Moreover, the change in the membrane hydrophilicity was measured by water uptake described elsewhere [12]. The membrane was cut 3x3 cm in size, and then it was immersed in deionized (DI) water for 24 hours. After immersion, the membrane was taken out from DI water, then wiped gently using tissue paper to absorb the excess water off its surfaces. Afterward, the membrane was weighed, and the weight of the membrane was considered as wet weight \( W_w \) (g). Furthermore, the membrane was dried by placing it in an oven for 48 hours at 60°C. Then the membrane was re-weighted as dry weight \( W_d \) (g). The percentage of the UF-PES membrane's water uptake was then calculated according to equation (3):
Water uptake (%) = \frac{(W_w - W_d)}{W_w} \times 100 \tag{3}

2.3.3. The membrane pore size. The membrane pore size was determined based on the MWCO (Molecular Weight Cut Off) test bypassing standard solution of the PEG solution through the test membrane with the series molecular weight (MW) of PEG 20.000; PEG 40.000, and PEG 100.000 as described elsewhere [13]. The Membrane MWCO test was briefly described as the following: 1. Preparation of iodine reagent solution; The iodine about 1.27 gram was added into 100 ml of 2\% (%w) KI solution. Then the solution was diluted up to 10 times with DI water until it was ready to use. 2. Preparation of PEG standard solution; About 150 mg of the PEG with different molecular weights was dissolved into 1000 ml of DI water to prepare 150 ppm of PEG solution called a stock solution. Then they have diluted again to 25; 50; 75; and 100 ppm. 3. The filtration of 150 ppm PEG solution through the membrane for one hour and the permeate solution was then collected. The feed and the permeate of PEG solution were taken about 0.3 ml. Each of the series PEG samples were then mixed with 3 ml of iodine standard solution, and it was kept for 30 minutes to allow the complete reaction to occur. Then the absorbance of each sample was analyzed with UV-Vis Spectrophotometer at a wavelength of 535 nm. Finally, the rejection value (%R) of the PEG 20.000, PEG 40.000, and PEG 100.000 was calculated according to equation (4).

\[ R (\%) = (1 - \frac{C_p}{C_f}) \times 100 \tag{4} \]

Where R is rejection value (%), while C\(_f\) and C\(_p\) are the concentration of feed and permeate solution (mg/l), the membrane's MWCO was then determined by the rejection value of 90\% of PEG solution.

2.4. The membrane performance on phospholipid removal of CPO-Isopropanol mixtures solution. The membrane UF-PES (M1) with a higher-pressure resistance up to 3 bar was then selected to filter the CPO-Isopropanol mixtures solution. Before the filtration process, the M1 membrane was conditioned by immersing it in the mixture of 25\% isopropanol: 75\% (v/v) of DI water for 30 minutes. About 500 ml of the feed solution was prepared by mixing CPO and Isopropanol with a 50\%: 50\% (v/v) ratio. The filtration procedures were as follows. The feed solution of CPO-Isopropanol mixtures (50\%: 50\%) (v/v) was filled to a dead-end membrane module with a volume of 300 ml. The membrane module was mounted with the M1 flat-sheet membrane with an effective area of 38.46 cm\(^2\). Transmembrane pressure (TMP) varies from 1.0 to 2.0 bar and was regulated by a valve from the N\(_2\) gas cylinder to provide the membrane driving force. The feed solution was kept stirred at 50 rev/min during the filtration process. The permeate solution was collected every 5 minutes and then weighed using an analytical balance until the steady-state condition was obtained. Finally, the phospholipid content in the feed and the permeate solution was analysed with UV-Spectrophotometer at a wavelength of 450 nm, according to Chen et al. [14]. The flux permeation and percentage of phospholipid from the solution mixtures were calculated according to equation (1) and equation (4). The combination effect of operating pressure and temperature during the filtration process was also studied and shown in Table 1.
**Table 1.** The combination treatment of the filtration process of CPO-Isopropanol mixtures.

| Temperature (°C) | Pressure (bar) | Replication number (U) |
|-----------------|----------------|------------------------|
|                 | T1 = 1 bar     | S1T1U1                 |
| S1 = 27°C       | T2 = 2 bar     | S1T1U2                 |
| S2 = 30°C       | T1 = 1 bar     | S2T1U1                 |
|                 | T2 = 2 bar     | S2T1U2                 |
| S3 = 35°C       | T1 = 1 bar     | S3T1U1                 |
|                 | T2 = 2 bar     | S3T1U2                 |

3. Results and Discussion

3.1. The UF-PES flat sheet membrane

A preliminary study was conducted, which consisted of preparing the UF-PES flat sheet membrane. It was followed by characterization of the prepared UF-PES membrane to select the best membrane to be used in the main study (i.e., filtration of CPO-Isopropanol mixtures). Two types of UF-PES membrane were synthesized, namely, M1 membrane that was immersed into the mixtures of DI water-Isopropanol as a coagulant bath while the M2 membrane was only immersed into a DI water coagulant bath. The appearance of the M1 and M2 membranes can be seen in Figure 1. Both membranes are white with a flat surface and are not transparent. In appearance, there is no difference in the two membranes' shape, texture, or colour. So it is necessary to characterize the membrane suited for the filtration process of the CPO mixtures solution.

![Figure 1. The UF-PES flat sheet membrane. A. M1 Membrane (immersion into the mixtures of DI Water-Isopropanol coagulation bath). B. M2 Membrane (immersion into DI water coagulation bath).](image)

3.2. Characteristic analysis of the UF-PES flat sheet membrane

In order to find the best membrane suited for filtration of CPO-Isopropanol mixtures solution, a series of membrane analyses was conducted to characterize the M1 and M2 membrane properties.

3.2.1. The membrane pure water permeation and isopropanol permeation measurement. The pure water permeation and the isopropanol permeation of M1 membrane and M2 membrane against the operation pressure are shown in Figure 2 and Figure 3.

![Figure A and B](image)
Figure 2. Profile of permeation flux of M1 membrane at various operating pressures. (A) Pure water permeation. (B) Isopropanol permeation.

Figure 2(A) shows the profile of pure water permeation flux of the M1 membrane against operating pressure. The steady flux of the pressure of 1 bar was reached at 45-50 minutes with a final flux of 1.25 l/m².h. Meanwhile, for the pressure of 2 bar, steady flux reached faster at 25-30 minutes with a final flux of 3.74 l/m².h. Further, at the operating pressure of 3 bar, the steady-state flux was slowed down to 40-45 minutes with a final flux of 4.06 l/m².h. Thus, an increase in the operating pressure brought about a higher water flux. The same tendency was also observed on the isopropanol permeation, as shown in Figure 2(B). The steady state of Isopropanol flux at a pressure of 1 bar was reached at 45-50 minutes with a final flux of 1.87 l/m².h whereas at the operating pressure of 2 bar, the steady flux was faster at 25-30 minutes with a final flux of 11.23 l/m².h. However, at the operating pressure of 3 bar, the steady flux time was similar to the operating pressure of 2 bar about 25-30 minutes with a final flux of 24.65 l/m².h. In comparison, the permeation flux of Isopropanol was higher than the permeation flux of pure water. This phenomenon occurred because the M1 membrane hydrophilicity property reduced when the membrane was treated with Isopropanol before the filtration process.

The profile of permeation flux of M2 at various pressures against the filtration time was presented in Figure 3. Figure 3(A) shows that the higher the pressure, the higher the water permeation flux. For example, it can be seen that at an operating pressure of 1 bar, the steady flux reached at 35-40 minutes with a final flux of 7.8 l/m².h. In comparison, the steady flux at a pressure of 2 bar was attained at 45-50 minutes with a final flux of 19.65 l/m².h while at a pressure of 3 bar, the steady flux was achieved at 40-45 minutes with a final flux of 42.43 l/m².h.
Figure 3. Profile of permeation flux of M2 membrane at various operating pressures. (A) Pure water permeation. (B) Isopropanol permeation.

Figure 3(B) shows the steady flux of Isopropanol of the M2 membrane at a 1 and 2 bar operating pressure. It was observed that for a pressure of 1 bar, steady flux was attained at 40-50 minutes with a final flux of 4.68 l/m².h whereas at a pressure of 2, the steady flux was achieved at 40-45 minutes with a final flux of 32.13 l/m².h.

3.2.2. The analysis of membrane functional group and membrane water uptake. The IR spectra of the functional groups on the M1 and the M2 membrane were given in Figure 4. It can be seen that the IR spectrum on both membranes was almost identical to each others. However, the M1 membrane has higher absorption intensity due to the difference in the coagulant bath between the two membranes. The IR spectrum shows the S=O=S functional group at the wavenumbers of 1247 cm⁻¹. Furthermore, double C=C stretch has appeared at the wavenumbers of 1483 cm⁻¹. At the same time, the C=O stretching was attributed to the carbonyl group at wavenumbers of 1680 cm⁻¹. In addition, the C-O-C functional group was observed at wavenumbers of 1153 cm⁻¹. These results were in good agreement with the works of Lusiana et al. [15] and Aryanti et al. [9].

Moreover, a similar result was observed from the works of Razi et al. [16]. Furthermore, Lusiana et al. [17] observed that the strong absorption at the wavenumbers of 1245 belonged to the –C-O-C-group of PEG. In addition, the absorption at wavenumbers of 2874 cm⁻¹ was attributed to the stretch of the –CH methylene group. Moreover, the absorption at wavenumbers of 3200-3600 cm⁻¹ indicated the absorption of the –OH group.
The IR spectra of the UF-PES membrane. (A). Membrane M1. (B). Membrane M2.

Figure 4. The IR spectra of the UF-PES membrane. (A). Membrane M1. (B). Membrane M2.

The percentages of water uptake (WU) on the M1 and M2 membranes are shown in Figure 5. It was observed that the M1 membrane has a water uptake capacity of about 68.75% in comparison to the M2 membrane, about 73.33%. According to Lusiana et al. [17], adding a PEG additive brought about the change in the membrane hydrophilicity. In contrast, slowing down the diffusion process between solvent and non-solvent (water), resulting in the formation of more pores on the surface of the membrane and could increase the membrane water uptake. Thus, it can describe the hydrophilicity of the membrane, which is related to the porosity of the prepared membrane.

Figure 5. Membrane water uptake.

3.2.3. The membrane pore size analysed. The membrane pore size was determined by the MWCO test. It was employed for the M1 membrane only because the M1 membrane has a higher resistance against elevated pressure from pure water and the Isopropanol experiment. The series of PEG with different molecular weights (20 kDa; 40 kDa; 100 kDa) was utilized as a solute to test the membrane's pore size or MWCO. The rejection percentage was calculated based on equation (4) as described in the experimental section of 3.3.2. The M1 membrane pore was determined by the rejection (R) value of 90% of the solute rejected by the membrane during filtration. Figure 6 shows that the rejection (R) values were 17.2% of PEG 10 kDa, 43.67% of PEG 40 kDa, and 80.54% of PEG 100 kDa. From these
results can be estimated that the M1 membrane pore size was bigger than 100 kDa. This result differed from Aryanti et al. [9], which gave the rejection value of 88.52% for PEG 20 kDa and 96.72% for PEG 40 kDa. This difference may occur due to the composition of the membrane's dope solution and the type of coagulation bath used.

3.3. The effect of operating pressure and temperature on membrane flux performance
The effects of operating pressure of (1-2 bar) and operating temperature of (27, 30, and 35°C) on the membrane permeation flux during filtration of CPO-Isopropanol mixtures solution were studied in detail. Figure 7 shows the membrane permeation flux profile at the temperature of 27°C against operation pressure of 1 and 2 bar, respectively. It was observed that the permeation flux of 2 bar (S1T2) was higher than that of 1 bar (S1T1). It is because the higher operating pressure causes the membrane pores to deform and become enlarged.
The flux of the M1 membrane at the S1T1 combination experiment was constant at 40 minutes with an initial flux of 3.68 l/m$^2$.h then it sharply decreased to 0.92 l/m$^2$.h. Whereas the final flux at S1T2 was only slightly dropped in comparison to S1T1.

![Permeation flux profile at a temperature of 30°C given in Figure 8(A). The initial flux at S2T1 was 3.66 l/m$^2$.h and then dropped drastically after 5 minutes and became constant at 25 minutes; with the final flux about 1.1 l/m$^2$.h. A similar condition was taken in the flux at S2T2 that decreased severely after 5 minutes and then became plateau after 25 minutes filtration with the final flux of about 0.55 l/m$^2$.h. Thus, the Final flux at S2T1 and S2T2 filtration was lower than that of S1T1 and S1T2. It is presumably because, at S2T1 and S2T2, the more compacted membrane has occurred than S1T1 and S1T2. On the other hand, quite different results were shown for the temperature of 35°C (Figure 8(B)); the flux of S3T1 and S3T2 was initially dropped and gradually increased against filtration time. As a result, the final flux of S3T1 and S3T2 was about 3.66 l/m$^2$.h and 5.16 l/m$^2$.h, respectively. In addition, the permeation flux of S3T2 was higher than that of S3T1. The results were agreed to by Pagliero et al. [18] that stated that the increased operating temperature causes the viscosity to decrease, so the flux increases.

Figure 8. M1 membrane permeation flux profile against filtration time. (A) The temperature of 30°C. (B) The temperature of 35°C.

3.4. The effect of operating pressure and temperature on phospholipid removal
Figure 9 showed that the lowest phospholipid concentration in the permeate solution was achieved by the S2T2 treatment, which was 35.27 mg/L with a rejection value of 78% in comparison to the initial phospholipid concentration in the feed solution. It happened because, at S2T2, the permeation flux is much lower than other treatments so that more phospholipids are retained. In the previous feed flux permeation. Meanwhile, the highest phospholipid level was achieved by S3T2, which was 57.99 mg/L with a rejection value of 53.95% compared to the initial feed. This result was expected because the higher operating temperature and pressure (S3T2) reduced the solution, so the permeation flux was higher than S3T1. On the other hand, the higher phospholipid level in the permeate solution was because the Isopropanol could induce the swollen membrane, which causes the lower membrane selectivity.
Figure 9. The effect of operating pressure and temperature on the phospholipid concentration in the permeate solution.

4. Conclusions
The UF-PES flat sheet membrane (M1 and M2) was synthesized via the phase inversion method, and their characteristics were studied. According to the membrane's pure water permeation and MWCO test, the M1 membrane was categorized as a UF membrane. The M1 membrane could keep a stable flux performance during filtration of CPO-Isopropanol mixture solution at various operating temperatures (S) and pressure (T). In addition, at the S2T2, the M1 membrane gave the higher phospholipid removal, about 78%. However, the phospholipid removal was considerably lower because the M1 membrane still has a larger pore size (above 100 kDa). The ideal UF-PES membrane pore size should be in the range of 20-40 kDa for the higher phospholipid removal. Furthermore, a comprehensive study is needed to determine the optimal composition ratio of CPO-Isopropanol mixtures as the feed solution, giving higher phospholipid removal.

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