The Application of Nanofiltration Membrane for Palm Oil Mill Effluent Treatment by Adding Polyaluminium Chloride (PAC) as Coagulant

Jhon Armedi Pinem, Imanuel Tumanggor, Edy Saputra*

Chemical Engineering Department, Engineering Faculty, Universitas Riau Binawidya Campus, Jln. H.R Soebrantas Km 12,5 Simpang baru Panam, Pekanbaru

*E-mail: edysaputra@unri.ac.id

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Abstract

The rapid development of Crude Palm Oil (CPO) production has led to an increase in the production of Palm Oil Mill Effluent (POME) as well. POME will cause problems in the environment because it contains high contaminants. This study aims to investigate the effect of the coagulant Polyaluminium Chloride (PAC) variations and the membrane's operating pressure on the POME treatment process using the nanofiltration membrane (NF) with the coagulation-flocculation process as pre-treatment. The PAC was used in the coagulation-flocculation process with variations in concentration (5.0; 5.5; 6.0; 6.5; 7.0 g/L). The process was completed by a rapid stirring of 200 rpm for 5 minutes, followed with slow stirring at 60 rpm for 15 minutes and settling time for 30 minutes. The process of membrane nanofiltration was carried out for 60 minutes with variations in operating pressure (8.0; 9.0; and 10 bars). In each treatment process, effluent quality testing was carried out with Biochemical Oxygen Demand (BOD5), Chemical Oxygen Demand (COD), Total Suspended Solid (TSS) and oil/fat as parameters. In addition, an analysis of permeate fluxes and rejection of NF membrane was also carried out. The results of the analysis suggested that the best coagulant doses are 6.0 g/L with the reduction percentage of BOD, COD, TSS and oil/fat at 78.85%; 68.57%; 92.77% and 92.31% respectively. The highest percentage of NF membrane rejection was found at a pressure of 10 bar, which is equal to 94.71%; 94.86%; 97.92% and 95% respectively for BOD, COD, TSS and oil/fat with a flux value of 7.16 L/m2.hours.

Keywords: palm oil mill effluent, nanofiltration, coagulation, flocculation, polyaluminium chloride

1. Introduction

Palm oil is one of Indonesia's main agricultural commodities that has grown with 12.3 million hectares of plantations, producing 35.4 million tons of crude palm oil (CPO) in 2017 (Adi, 2017). Plantation of palm oil are expected up to 13 million hectares with total production up to 40 million ton in 2020. The increased production of palm oil will likely cause problems for the environment, particularly the liquid waste generated from palm oil processing industry in the form of Palm Oil Mill Effluent (POME), which is difficult to degrade. POME is produced at the boiling, sedimentation, decantation and centrifugation stages carried out during the CPO clarification process. Every ton of CPO produced generates between 22.5 – 3.0 m³ POME (Shintawati et al., 2017).

POME is a colloidal suspension consisting of 95-96% water, 0.6-0.7% oil/fat and 4-5% total solids. It is removed from factories as a dark brown, thick liquid with a distinctive smell at 80- 90°C and has a pH value between 4.0-5.0. In addition, the content of POME contaminants ranges between 23,000-32,000 mg/L and 25,000-36,000 mg/L for BOD (biochemical oxygen demand) and COD (chemical oxygen demand) respectively (Rahardjo, 2005). The figures have not met the criteria for the quality standard of palm oil industry effluent as stated by the Regulation of Ministry of Environment number 5 of 2014, especially with COD far above 350 mg/L and BOD above 100 mg/L. Without proper processing and compliance with the said standards, POME will potentially pollute the environment by deteriorating soil and air quality (Shintawati et al., 2017).

CPO factories generally process POME conventionally in a series of open aerobic-anaerobic ponds, followed by land applications (Hasanuddin et al., 2015). Conventional treatment methods have several disadvantages. They store waste up to 20-200 days in disposal area (Bala et al., 2014); (Poh and Chong, 2009), and require a...
large area to accommodate the waste generated every day and turn to be less environmentally friendly because of releasing a lot of methane gas into the atmosphere (Wahyun et al., 2016).

Membrane technology is a technology that offers several benefits to overcome the current weaknesses in waste treatment. The technology can be constantly produced; it has a good quality of the permeate produced, and it can be used continuously, thereby saving time and energy (Mulder, 1996). In this research, pretreatment was carried out by coagulation-flocculation using Polyaluminum Chloride (PAC) coagulant, the aim of which was to reduce the membrane workload so that the membrane will last longer.

Research on POME processing has been done in the past. Idris et al. (2010), for example, studied the use of the UF membrane system for tertiary treatment of biologically treated POME with the effect of MWCO and transmembrane pressure. The pretreatment process was carried out coagulation using ferric chloride and polyacrylamide as the flocculant aid in the concentration of 100 mg/l for both. After going through the pretreatment process, POME was treated with an ultrafiltration membrane of 1 kDa and 5 kDa in which under multiple pressures (0.5; 1.0; 1.5 bar). Results showed that the pretreatment processes which consist of coagulation and adsorption showed remarkable results in reducing COD, color and turbidity up to 92.8 %, 99.3 % and 99.9 % respectively. This study also indicated that at transmembrane pressure 0.5 bar, an increase in reduction COD, color and turbidity were observed. Thus, it is concluded that the smallest MWCO of the membrane at transmembrane pressure 0.5 bar gave a better reduction of pollutants from the pretreated POME. Also, it can be concluded that increasing transmembrane pressure leads to a corresponding increase in permeate flux which starts to level off at higher transmembrane pressures.

Ahmad and Chan (2009) conducted a study entitled "Sustainability of Palm Oil Industries: An innovative treatment via membrane technology". They integrated the membrane process with chemical-physical treatments; that is, the coagulation-flocculation process as pretreatment and the combination of ultrafiltration membrane (UF) and reverse osmosis (RO) processes as post-treatment. The separation process on the UF membrane was done at 25°C and 2 bar pressure, the same temperature was done also on the RO membrane but with 40 bar pressure. This pretreatment used moringa oleifera seed as coagulants were able to reduce 56% TSS; 98% oil and fat; and 70% COD. Furthermore, the ultrafiltration process was able to reject 25% TSS; 99.9% oil and fat; and 17.3% COD. The final process on the RO membrane rejected 98% TSS; 99.9% oil and fat; and 99.3% COD. The value of the POME from the treatment is below the POME quality standard according to the US Environmental Protection Agency (USEPA).

Said et al. (2015) in his study entitled "Investigation of Three Pre-Treatment Method Prior to Nanofiltration Membrane for Palm Oil Mill Effluent Treatment" varied pre-treatment methods on POME including ultrafiltration, adsorption and decantation. In this study, a post-treatment was done with the method of nanofiltration (NF) using NF-1 and NF-ASP30 membrane modules operated under 10 bar pressure. The results indicated that the combination of adsorption and nanofiltration generated the highest reduction in COD, TSS, color and POME turbidity, but in terms of cost and time, the combination of ultrafiltration and nanofiltration was found to be better. In addition, the NF-1 performed better than NF-ASP30 in that it rejected 98.70% COD; 99.95% TSS; 99.85% color (PtCo); and 99.99% turbidity (Ntu).

Arifin and Sri (2014) studied varied pre-treatment methods on water treatment with and without pre-treatment of ultrafiltration coagulation. Ultrafiltration membranes have been characterized by measuring ultrafiltration, flux, permeability (Lp), and MWCO with various molecular weights of dextran SEM analysis was carried out on the membrane surface and cross-section of the membrane. Optimum coagulation process was carried out using a jar test tool, obtained optimum conditions at 50 ppm Al2(SO4)3 and pH 7. The colour rejection index obtained by coagulation is 85% and without coagulation is 62% From this study it can be concluded that the percentage of rejection is higher in the membrane with the coagulation treatment in the membrane process.

In a study by Aprilia et al. (2013) entitled "The Combination of the Coagulation Process and Ultrafiltration System with Polycrylonitrile Membranes for Colored Water Purification" states that a serious problem encountered in ultrafiltration membranes is the tendency for flux to decrease throughout the operating time. This can prevent the subsequent separation of water going through the membrane. Therefore, treatment is carried
out using a combination of coagulation and ultrafiltration membrane methods. Preliminary treatment with coagulation can improve membrane. This study aims to examine the performance of the coagulation-flocculation process by using PAC coagulants and the performance of nanofiltration membranes (NF) in removing BOD5, COD, TSS and oil/fat effluent from CPO factories. The resulting flux and rejection are higher than membranes without coagulation pretreatment.

Idris et al. (2010), a combination of coagulation process using ferric chloride and flocculation process using polyacrylamide as an adsorbent was found to significantly reduce COD, color and turbidity by 97.9%, 99.3%, and 99.9% respectively.

Similar research by Ahmad et al. (2005) concluded that the pre-treatment process involving coagulation-flocculation with activated carbon adsorption successfully reduced 99.9% suspended solids, 95% oil and fat, 86.3% BOD and 85% COD before POME was ultimately treated with the NF membrane. So, this study is going to know an optimum condition to treatment POME effluent by adding polyaluminium chloride (PAC) as a coagulant and study the permeability and rejection of the nanofiltration membrane.

2. Materials and Method

2.1. Materials

POME was taken from the tertiary pond, CPO factories of PTPN V Sei Galuh located in Tapung, Kampar Regency, Riau. Then, the POME stored in a refrigerator at 4°C. POME had treated using PAC coagulants. The POME characteristics are outlined in Table 1. This study employed the commercial membrane module NF-1812-150, from Kusatsu Toray Nanofiltration Membrane produced by PT. Indotara Persada (see Table 2).

2.2. POME Pretreatment Procedure

The pre-treatment of POME includes the process of coagulation-flocculation using PAC coagulants with the concentration varied at 5.0; 5.5; 6.0; 6.5 and 7.0 g/L. The coagulant was mixed with POME by using fast stirring at 200 rpm for 5 minutes and slow stirring at 60 rpm for 15 minutes. After that, the solid is allowed to settle for 30 minutes so that it is separable from the effluent. The effluent from the pre-treatment process was taken as a sample to analyze its BOD5, COD, TSS and oil/fat.

Table 1. Characteristics of POME Initial Samples

| No. | Parameter     | *Quality standards | **Analysis Results |
|-----|---------------|--------------------|-------------------|
| 1   | BOD5 (mg/L)   | 100                | 10.005            |
| 2   | COD (mg/L)    | 350                | 22.115            |
| 3   | TSS (mg/L)    | 250                | 4.260             |
| 4   | Oil or fat (mg/L) | 25         | 260               |
| 5   | pH            | 6.0 – 9.0          | 4.6               |

Source: * The Regulation of Ministry of Environment, the Republic of Indonesia, Number 5, 2014 ** Results of Laboratory Testing in Unit of Construction Materials Testing, Public Works and Spatial Planning, Riau Province

Table 2. Membrane Characteristics

| Design Specifications | Capacity and Model |
|-----------------------|--------------------|
| Membrane Material     | Poly (Piperazine-Amide) composite |
| Average Rejection     | 80%                |
| Elements              | Spiral wound       |
| Configuration         | K 0.32 m²          |
| Pre-filtration        | 50 – 150 µm        |
| Maximum pressure      | 300 Psi (21 bar)   |
| Flux Score            | 150 GPD            |
| Operating             | 5 – 45°C           |
| Temperature           | PH Range 3 – 10    |
| Pore Diameter         | 0,001 µm           |

2.3. Nanofiltration Procedure

In this study, the operating pressure was varied at 8.0; 9.0 and 10.0 bars. About 50 ml of permeate sample was taken every 5 minutes over 60 minutes of operating time, the aim of which was to analyze the amount of BOD5, COD, TSS and oil/fat of the permeate. Backwashing for 30 minutes using aquades was done every time the variable was changed. Concentrated on solute each sample was measured by the third party.

According to Munandar et al. (2016) research showed the efficiency of COD absorption at nano-size was higher than the size <35 microns. Nanoparticles have several advantages including the ability to penetrate a variety of spaces that cannot be penetrated by large particles, the ability to penetrate higher cell walls and flexibility that can be combined with various other technologies that will open up great potential to be developed in various needs and goals. Another advantage is that there is an increase in the affinity of the system due to an increase in the contact surface area by the same amount.
2.4. Analysis

In analyzing the wastewater parameters, the authors relied on the standards ruled by the Indonesian National Standard (SNI) for BOD5 (SNI 06-6989.72: 2009) Lenore S.C et al. (2005), COD (SNI 06-6989.73: 2009) Lenore S.C et al. (2005), TSS (SNI 06-6989.3: 2004) Lenore S.C et al. (1998), and oil/fat (SNI 06-6989.10: 2011) Lenore S.C et al. (2005).

For the analysis of the structure, morphology and distribution of membrane pore before and after the nanofiltration process, Scanning Electron Microscopy-Energy Dispersive X-ray (SEM-EDX) was used. The rate of permeate flow, flux and membrane selectivity were also analyzed.

Flux is the velocity of flow through the membrane, calculated through Equation 1.

\[ J = \frac{V}{At} \]  

(1)

With,

- \( J \) : liquid flux (L/m².hour),
- \( V \) : permeate volume (L),
- \( t \) : permeate time (hour) and
- \( A \) : the width of membrane surface (m²)

Selectivity describes the ability of membranes to separate one species from another. The value of rejection is determined by Equation 2.

\[ R = \frac{C_p}{C_f} \times 100\% \]  

(2)

With,

- \( R \) : rejection percentage
- \( C_p \) : concentration of dissolved substance in the permeate (ppm), and
- \( C_f \) : the average concentration of the dissolved substance in the feed and retentate (ppm).

3. Results and Discussion

3.1. Pretreatment Performance

The coagulation-flocculation process had been used extensively as a pre-treatment for removing suspended particles and coloring material in primary treatment before biological treatment. As shown in Figure 1, the coagulation-flocculation process has a significant effect on the removal of TSS and oil/fat rather than BOD and COD because the coagulation-flocculation treatment of liquid waste aims at removing suspended solids by turning flocks into macrophages so.

Figure 1 also shows that an increase in the coagulant dose led to a higher percentage of removal until reaching the optimal point of POME destabilization or the removal of waste pollutants. About 6.0 g/L of coagulant was required to achieve the highest reduction efficiency, which was the removal of BOD, COD, TSS and oil/fat by 78.85%; 68.57%; 92.77%; and 92.31% respectively. When the coagulation process ended, the positively charged polyelectrolyte adsorbent formed macro flock particles with slow stirring. When the flocks reached the optimum size and strength, the sedimentation process was carried out.

Figure 1. Effects of coagulant on the removal of BOD, COD, TSS and oil/fat

The addition of the composition of PAC as a coagulant affects the percentage of removal. In Figure 1, there is an increased removal percentage for the composition of the coagulant from 5.0 g / L to 6.0 g / L. It caused PAC had an excellent ability to remove so that it can reduce the value of the tested sample parameters. This is because PAC coagulants have bound and precipitated most of the particulates in wastewater. Especially in COD, the decrease in COD in wastewater is caused by floc formed by the ions of organic compounds that bind to positive coagulant ions. Molecules in the waste are formed into floc, colloidal particles in the waste are binding particles or other compounds that are in the waste. With a decreasing number of particles, the oxygen needed to oxidize organic compounds also decreases, so the COD value after coagulation is also low. In other cases, when PAC coagulant with composition 6.5 g/L and 7.0 g/L decrease removal percentage. It caused an overdose of PAC coagulants composition would cause a slight decrease in removal efficiency, because of the reversal of surface charge and the stabilization of the coagulated particles. After the dose of PAC exceeds the saturation of the polymer bonds, the excess PAC is likely to break the polymer bonds between the
particles, thus showing an increase in residual turbidity.

In this study, the percentage of organic reduction was compared with that of previous studies that used pre-treatment processes such as coagulation-flocculation and adsorption. Therefore, compared to the results of previous studies using the coagulation-flocculation process as pre-treatment, the results obtained in this study are better. However, the combination of the coagulation-flocculation process with activated carbon adsorption is more effective at reducing organic POME before proceeding to the processing with membranes.

### 3.2. Characteristics of the Quality of the Produced Permeate

In general, the pre-treatment process with PAC coagulant by 6 g/L had reduced pollutants in BOD5, COD, TSS and oil/fat compared with those of their initial sample before the NF process. Figure 2 shows that the effect of operating pressure on the amount of BOD, COD, TSS and oil/fat removed is increasing with increasing pressure from 8 bar to 10 bar. Thus, it is consistent with the finding of research conducted by Wu et al. (2007) and Wahab et al. (2009) that the increase in operating pressure used in the membrane filtration process would increase membrane selectivity, thereby raising the percentage of BOD, COD, TSS and oil/fat removed. This is attributable to the area of fouling (clogging), which is the area formed by the accumulation of particles unable to pass through the membrane. The fouling area then acts as another layer or additional filter that increases resistance to organic materials going to pass through the membrane (Idris et al., 2010).

Figure 2 displays the highest scores of NF membrane selectivity at 10 bar pressure equal to 94.71%, 94.86%, 97.92%, and 95% for BOD, COD, TSS and oil/fat respectively. The nanofiltration membrane was able to reduce the amount of TSS and oil/fat below the quality standard set by the government in the regulation of Ministry of Environment Number 5 of 2014 concerning the quality standards of palm oil industry liquid waste, which is below 250 mg/L and 50 mg/L respectively for TSS and oil or fat. However, the amount of BOD and COD after the nanofiltration membrane process was still above 100 mg/L and 350 mg/L, slightly higher than the established standards for BOD and COD. Therefore, the nanofiltration membrane process still requires an additional pretreatment process to eliminate more BOD and COD in POME because of the coagulation-flocculation processes (chemical processing) and NF membrane processes (physical processing) were not optimal in reducing the amount of BOD and COD. An extra pretreatment with biological treatment methods is necessary to reduce the amount of BOD and COD effectively.

![Figure 2](image.png)

**Table 3. POME Characteristics at Each Processing Stage**

| Parameter | Coagulation-Flocculation | Nanofiltration |
|-----------|--------------------------|----------------|
| BOD5 (mg/L) | 2116 | 112 |
| COD (mg/L) | 6950 | 357 |
| TSS (mg/L) | 308 | 6.4 |
| Oil/fat (mg/L) | 20 | 1 |

A comparable experiment by Abdullah et al. (2017) on the processing of Aerobically-Treated POME using the commercial membrane NF90 from DOW FILMTEC (United States) at 10 bar resulted in the reduction of 78.66% conductivity (μS), 98.92% color (American Dye Manufacturer’s Institute), and 87.15% total organic carbon (ppm). Therefore, the results obtained in this study are better than those of previous studies.

Table 3 and Figure 3 clearly shows that after all stages (coagulation-flocculation and nanofiltration), the amount of BOD, COD, TSS and oil/fat in POME successfully reduced are quite large (98.88%, 98.39%, 99.85%, and 96.00%) and comparable to results of other studies. For instance, the experiment by Said et al. (2015) using adsorption and nanofiltration (NF-1) as pre-treatment processes successfully rejected 98.70% COD,
99.95% TSS, 99.85% color (PtCo); and 99.99% turbidity (NTU). In addition, Ahmad et al. (2006) who also treated CPO factory effluent by the coagulation-flocculation process succeeded in reducing TSS parameters and oil/fat by more than 95%. Finally, the work of Ahmad and Chan (2009) employing coagulation-flocculation and ultrafiltration membrane processes followed by nanofiltration was able to cut off 98% TSS; 99.9% oil and fat, and 99.3% COD.

![Figure 3. The reduction of BOD5, COD, TSS and oil/fat concentration at each processing stage](image)

### 3.3. Permeate Flux

In this study, the process of the nanofiltration membrane was operated with 8 bar, 9 bar and 10 bar pressure. Figure 4 demonstrates clearly that the pressure noticeably increased the permeate flux. The higher the operating pressure, the bigger the figures of permeate flux. This finding is expected since theoretically, the pressure is a driving force for the nanofiltration process (NF), so a rise in pressure will lead to an increase in total flux. According to Rosnelly (2012) research, a common phenomenon that often found in a membrane separation process was if the membrane flux is large then rejection will below, and vice versa if the rejection is high then the flux will also below.

The highest average flux value was obtained at a pressure of 10 bar (7.16 L/m²-hour). The values obtained in this study are comparable to those of previous research as shown in Table 4. This flux is relatively lower compared to the commercial membrane. The difference in flux values is due to the different quality of POME waste feed used in the nanofiltration process. The quality of the waste feed affects the resulting flux – the higher the amount of waste pollutants, the smaller the flux produced. This owes to fouling, membrane pore-clogging or greater membrane workload (Notodarmojo et al., 2004). The average flux in this study was 7.16 L/m²-hour with 2116 mg/L BOD5, 6950 mg/L COD, 308 mg/L TSS, and 20 mg/L oil/fat, while the study by Said et al. (2015) resulted in 8.95 L/m²-hour and 12.62 L/m²-hour average flux value with feed quality 416 mg/L BOD5, 645 mg/L COD, 105 PtCo (Color), and 2203 NTU turbidity.

![Figure 4. Effect of operating pressures on the membrane flux](image)

| Membrane                  | Operating Pressure, bar | Flux, L/m²-hour | Ref         |
|---------------------------|-------------------------|-----------------|-------------|
| NF270 nanofiltration membrane, Dow FILMTEC™, USA) | 10                      | 9.01            | Tan et al.  |
| NF-1, Amfor Inc., China   | 10                      | 8.95            | Said et al. |
| NF-ASP30, Amfor Inc., China | 10              | 12.62           | Said et al. |
| Nanofiltration membrane   | 10                      | 7.16            | This Work   |

### 3.4. Membrane Resistance

Figure 5 illustrates that the permeate flux decreased as the pressure was raised from 8 bar to 9 and 10 bar and with the passage of nanofiltration membrane operating time. The acquired data are consistent with those found by Wu et al. (2007), and the decrease owes to the case of fouling and membrane resistance. Both of which generally occur due to pore clogging, the formation of a gel layer, or concentration polarization. Pore blockage happens because of the asymmetrical membrane pore structure.

Wibisono et al. (2018) stated that there would be a decrease in membrane permeability and flux values with the addition of extract which in this study used Moringa extract. This study concluded that the addition of Moringa extract
was able to inhibit bacterial growth by 39.7%. On the other hand, membrane permeability has decreased from 29,479 to 19,007 mL/cm² minutes which has the potential to increase the rejection rate of the result of the microfiltration membrane.

It is also visible from Figure 5 that the decrease in flux occurred rapidly at 9 bar and 10 bar pressure, while the flux was more stable with a slow decline at 8 bar. This phenomenon is caused by increasing operating pressure. The higher the pressure, the faster the flow of POME waste feed to be filtered, speeding the process of pore-clogging and formation of a gel layer. Figure 6 displays proof of this pore blockage.

Figure 7 was processed using ImageJ 1.52a software to estimate the number of pores, pore area and pore diameter of the commercial nanofiltration membrane before and after being used for the membrane filtration process. The estimated pore area is between 0.01- 0.10 µm² and the pore circularity (the degree of closeness of the pore shape to the circle) ranges between 0.50-1.00. After analysis, the amount of pore nanofiltration membranes before use was 459/445.5 µm² or 1.03 pores/µm² with 9,432 µm² total area, while that after use was 233 every 445.5 µm² or 0.52 pore/µm² with a total area of 4.359 µm². This means there was a pore blockage during the membrane operation time. The amount of membrane pores obtained through ImageJ software is given in Figure 8.

EDX (energy dispersive x-ray) is an analytical technique used to determine the elements and chemical characterization of a sample, so the elements that cause fouling on commercial nanofiltration membranes after use can be identified. Figure 8 and Figure 9 show the results of EDX analysis of nanofiltration membrane samples before and after use, displaying the differences in components and composition (% of weight) on the membrane. After the process of nanofiltration membrane, the authors discovered the addition of other elements including silica oxide (SiO) by 0.29% of
weight, alumina (Al₂O₃), Sulfite (SO₃), Chloride (Cl), Potassium Oxide (K2), Copper (II), and Oxide (CuO). These elements come from POME waste pollutants that did not escape and hence trapped in the pore membrane, leading to the occurrence of fouling (Wahyuni et al., 2017).

4. Conclusion

The optimal conditions for removing pollutants in palm oil industry liquid waste through the coagulation-flocculation process were obtained at a coagulant dose of 6.0 g/L that cut off up to 78.85% BOD, 68.57% COD, 92.77% TSS, and 92.31% oil/fat. Meanwhile, the highest rejection percentage of the NF membrane process was obtained at a pressure of 10 bar (94.71% BOD, 94.86% COD, 97.92% TSS, and 95% oil/fat) with an average flux value of 7.16 L/m².hour.

The combination of the coagulation-flocculation process and membrane technology in the CPO effluent treatment effectively reduces the amount of TSS and oil/fat below the quality standard (6.4 mg/L and 1 mg/L). Nevertheless, POME processing using NF membranes can be combined with conventional processing to meet quality standards.

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