NON-DISSOLVED SOLIDS REMOVAL DURING PALM KERNEL OIL ULTRAFILTRATION

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Abstract

Performance of polypropylene hollow fiber ultrafiltration membrane during non-dissolved solids (NDS) removal from palm kernel oil is investigated. The filtration is operated at difference feed temperature and pressure to study the effect of both parameters on membrane performance. From the experimental results, it can be concluded that polypropylene hydrophobic hollow fiber membrane can be used for palm kernel oil NDS removal. Temperature and trans-membrane pressure have proportional effect to permeate flux. In contrast, they have inverse effect to rejection of NDS. During the experiment, permeate fluxes and rejections of NDS varied from 3.4 to 8.7 L/m^2.h and from 51% to 94%, respectively. The best operating conditions suggested are feed temperature of 30°C and TMP of 1 bar which produce the highest NDS rejection. In addition, the permeate quality can meet the requirement of standard NDS content even at its lowest rejection level which shows the remarkable performance of membrane filtration.

Keywords: *non-dissolved solids; palm kernel oil; polypropylene membrane; ultrafiltration*

Abstrak

PENYISIHAN PADATAN TAK TERLARUT PADA PROSES ULTRAFILTRASI MINYAK INTI SAWIT. *Makalah ini menyelidiki tentang kinerja membran ultrafiltrasi hollow fiber selama proses penyisihan padatan tak terlarut (NDS) dari minyak inti sawit. Filtrasi dioperasikan pada temperatur umpan dan tekanan yang berbeda untuk mempelajari pengaruh dari parameter tersebut terhadap kinerja membran. Dari hasil eksperimen dapat disimpulkan bahwa membran hollow fiber hidrofobik polipropilen dapat digunakan untuk penyisihan NDS dari minyak biji sawit. Temperatur dan tekanan memiliki pengaruh proporsional terhadap fluks permeat. Sebaliknya, parameter tersebut memiliki pengaruh yang berkebalikan terhadap rejeksi NDS. Selama eksperimen, fluks permeat bervariasi dari 3,4 hingga 8,7 L/m^2.h dan rejeksi NDS dari 51% hingga 94%. Kondisi operasi terbaik yang disarankan adalah temperature 30^{o}C dan tekanan operasi 1 bar yang menghasilkan rejeksi NDS tertinggi. Selain itu, kualitas permeat dapat memenuhi syarat standar kandungan NDS meskipun pada kinerja filtrasi dengan rejeksi terendah yang menunjukkan kinerja yang sangat baik dari filtrasi membran.*

Kata kunci: *padatan tak terlarut; minyak inti sawit; membran polipropilen; ultrafiltrasi*

INTRODUCTION

Palm kernel oil has a relatively higher melting point compared to other vegetable oils, due to the 85% saturated fatty acids present in the oil. The main components of palm kernel oil are palmitic, oleic, and linoleic fatty acid. Uniquely, palm kernel oil composition is very similar with coconut oil (Pantzaris and Ahmad, 2004). Therefore, palm kernel oil stearin (the solid fraction) is highly suitable to be used as cocoa butter substitute.

Palm kernel oil contains minor components such as non-dissolved solids (NDS), phospholipid (gum), free fatty acid (FFA), sterol, protein, color pigments, and oxidation products. Some of these components that considered as impurities are NDS, phospholipid, FFA, oxidation products, and sometimes the color pigments. In order to avoid quality deterioration which attributed to these components, palm kernel oil purification process is required.

As one of the internationally traded food products, vegetable oil has an international standard of quality which is determined by WHO (World Health Organization) and FAO (Food and Agriculture Organization). The quality standard for palm and palm kernel oil is CODEX STAN 210-1999. This includes general quality characteristics (including insoluble solids content), fatty acids composition, physical and chemical characteristics, and antioxidant level in the oil.

Conventional vegetable oil processing is identical with high energy consumption, loss of oils and nutrients, high water and other chemicals demand, and generation of effluent in large quantity (Coutinho *et al.*, 2009). Membrane separation is a potential alternative to overcome those drawbacks. Lower energy demand, shorter processing steps, higher efficiency, and better oil quality are some advantages of membrane processing for vegetable oils.

The utilization of membrane processing technology in vegetable oil refinery has been extensively studied. Membrane can be used in degumming process (Subramanian *et al*., 1999, Basso *et al*., 2009, Alicieo *et al*., 2002, Hafidi *et al*., 2005, Ribiero *et al*., 2008). Other studies also evaluate about membrane flux behavior of several vegetable oils (Manjula *et al*., 2011, Alicieo *et al*., 2002), deacidification (Firman *et al*., 2013), decolorization (Reddy, 2001), and pigment concentration from the oil (Chiu *et al*., 2009).

The studies of membrane for vegetable oil refining to reduce the processing steps have also been reported. For example, ultrafiltration membrane can be used in a simultaneous clarification and degumming process (Kusumah and Sadeli, 2007), deacidification and solvent recovery (Firman *et al*., 2013), simultaneous degumming, deacidification, water and soap removal (Hafidi *et al*., 2005), and also simultaneous deacidification, degumming, and color removal (Subramanian *et al*., 1999). This study focuses on the clarification of palm kernel oil using hydrophobic polypropylene hollow fiber membrane. Performance of membrane during filtration is investigated according to the permeate flux and NDS rejection.

MATERIALS AND METHODS

Materials

Crude palm kernel oil is supplied from PT Agricinal Bengkulu, Indonesia (NDS content: 0.08% wt; density: 0.907 kg/l). The refined oil used for membrane conditioning and initial performance testing is a commercial refined palm oil (Bimoli, PT. Salim Ivomas Pratama, Tbk, Surabaya). Other materials such as n-hexane that is used for diluting the oil sample and Whatmann glass fiber filter for NDS analysis are obtained from Chemical Engineering Operational Laboratory, Institut Teknologi Bandung. Polypropylene hydrophobic hollow fiber membrane is obtained from GDP Filter, Indonesia. The membrane pore diameter is 0.05 μm and the membrane fiber diameter is 0.35 mm. A 30 cm membrane module consists of 150 hollow fibers with a dead-end configuration. The filtration system is equipped with a pump and a pressure indicator that are placed in membrane inlet stream. The experimental set up is shown in Figure 1.

Experimental Procedure

The experiment comprises of three steps, which are: (1) initial membrane performance testing with refined oil, (2) crude palm kernel oil sample pre-treatment and conditioning, and (3) non-dissolved solids removal of the crude palm kernel oil sample.

Initial membrane performance testing is conducted to check the module and membrane leakage. The refined oil is poured into sample container and pumped into membrane module. The oil is transferred into membrane module and permeated at dead-end filtration mode by flowing the oil from shell side into lumen side. Filtration is conducted under room temperature and within a range of trans membrane pressure (TMP) which will be used in crude palm oil clarification process. Afterward, the membrane flowrate is observed by collecting permeate volume in a period of time at each TMP. Any leakage will be observed when a very high flowrate is occurred at a relatively low operating pressure.

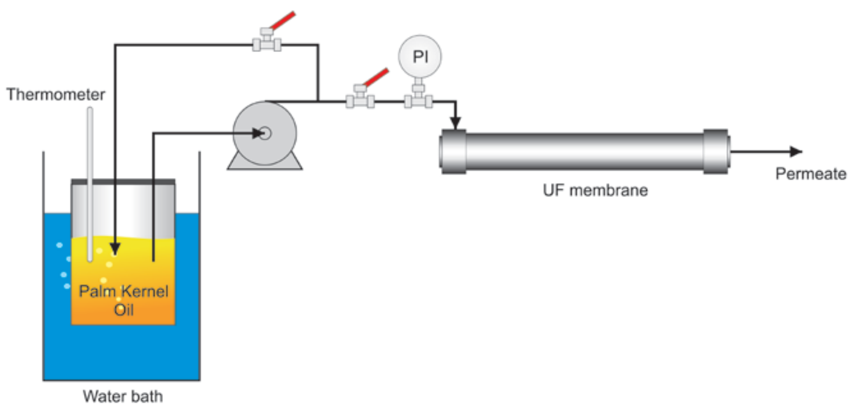


Figure 1. Experimental set up

In the pretreatment step, sample (crude palm kernel oil) is agitated for about 5 minutes to obtain fine distribution of NDS content. Afterward, 700-1000 mL oil sample is poured into sample container, which then placed in the water bath to adjust the oil temperature to a desired value. The oil temperature is maintained throughout the operation.

Crude palm kernel oil that has been conditioned is pumped through the membrane module. The dead-end module configuration is conducted by flowing the crude oil from membrane shell side to the lumen side. This flow configuration is applied to prevent the blocking of lumen side by the NDS component. This process is continuously operated until it reaches a constant permeate flux. Each run takes up to 30 minutes.

Data Collection and Analysis

During the clarification process, 5 mL of permeate is collected every 5 minutes and the time needed for each sample collection is recorded. The permeate flux (J) is calculated according to the following equation,

$$J = V/(A.t) \quad (1)$$

where V is volume of permeate, t is collection time, and A is membrane total surface area.

NDS rejection is the percentage of NDS removed during the clarification. A 100 mL of permeate sample is collected, and then diluted with n-hexane to make 30% n-hexane micella. The mixture is filtered in a vacuum filtration system. NDS rejection is calculated with the following equation,

$$\text{Rejection} = (m_i - m_f)/m_i \times 100\% \quad (2)$$

where m_i represents amount of NDS (gram) before the clarification process and mf represents amount of NDS after the process.

All data obtained are processed using Minitab software (trial version) to construct the surface plot diagram. The surface plot diagram represents the effect of both operating variable on permeate flux and NDS rejection percentage.

RESULTS AND DISCUSSION

Permeate Flux

Flux is an important parameter that is being used to determine the feasibility of membrane processes for industrial application, besides membrane selectivity (Arora *et al*., 2006). High permeate flux is desired in industrial membrane processes to ensure that the processes are economically viable. Figure 2 presents the permeate flux obtained at different TMP and temperature. As shown in Figure 2, the permeate flux improves with an increase in TMP. Since TMP becomes higher, the driving force for oil diffusion into the permeate side also increases. When there is no compaction of membrane at lower pressure range, permeate flux is controlled by pressure (Lin *et al*., 1997). The result obtained in this experiment indicates that within the TMP range (1-2 bars), there is no membrane compaction observed. Therefore, increasing TMP gives greater driving force for the oil to diffuse through membrane pores. At higher temperature, the permeate flux increases more rapidly which attributed to viscosity reduction as the feed temperature rises (Lin *et al*., 1997). For example, by increasing the feed temperature from 30°C to 50°C, the permeate flux increases from 6.3 to 8.7 L/m^{2}h. In that range of temperature, palm kernel oil viscosity decreases more than a half of the initial temperature, from 44.0 to 21.0 cP (Timms, 1985). In membrane process application, operating at high temperature is in line with flux, since it enhances the mass transfer and thus increases the permeation rate (Cheryan, 1986). However, it should be noted that high temperatures can only be applied when the quality of the oil regarding its thermally sensitive materials is not a main concern (Bottino *et al*., 2003). The feed temperature has a small effect to permeate flux in a low TMP (1 bar). On the other hand, TMP has a better influence to the flux. Thus, the higher permeate flux can be achieved at combination of high feed temperature and high TMP.

Rejection of Non-dissolved Solids

Membrane retentivity towards impurities is the main concern in membrane performance evaluation. There are several factors that affect membrane retentivity, such as membrane type, molecular size of solute, and micro-environmental conditions such as pH, temperature, and concentration of solutes (Lin *et al*., 1997). In this experiment, NDS is the main impurity that is rejected by the membrane.

Figure 3 illustrates how TMP and temperature affect the rejection of NDS. The increase of TMP leads to reduction of rejection percentage. The rejection percentage decreases from 94% to 51% with rising TMP from 1 bar to 2 bar. The increasing TMP decreases NDS rejection due to both concentration polarization and/or the shear-induced solute distortion at a higher TMP (Zeman and Wales, 1981). A higher TMP relates to a higher oil velocity as well. It does not facilitate the maximization of the concentration polarization, thereby decreasing the rejection percentage. At the same TMP, an increase of feed temperature leads to a decrease of NDS rejection percentage. The rejection percentage drops from 93% to 60% as the feed temperature rises from 30°C to 50°C in constant TMP (1.5 bar). The increasing of feed temperature leads to decreasing viscosity of palm kernel oil. Thus it facilitates mass transfer improvement and contributes to selectivity deterioration as well. Meanwhile, oil viscosity is increased and mass transfer rate is reduced at lower temperature and TMP which resulting in lower shear rate. This phenomenon causes formation of polarization layer on the surface of membrane. The deposition of impurities on the membrane surface provides a polarization layer resulting an increase in membrane rejection (de Moura *et al*., 2005). Although the formation of polarization layer lowers the permeate flux, it contributes to enhance the NDS rejection due to the improvement of the membrane selectivity.

The productivity of a membrane process is measured mostly by its permeate flux and impurities rejection (de Moura, *et al*., 2005). The preferred membrane process is the one with high permeate flux and solids rejection. However, to obtain either highest permeate flux or solids rejection, the other membrane performance parameter is usually compromised.

Figure 4 (a) and (b) show surface plot of permeate flux and rejection as a function of feed temperature and TMP for palm kernel oil clarification process. According to Figure 4 (a), the best permeate flux in this experiment is 8.7 L/m^{2}h, which is acquired at the highest feed temperature (50°C) and TMP (2 bar).

Meanwhile, the best rejection performance in this experiment is 94%, which is obtained at the lowest feed temperature (30°C) and TMP (1 bar) as depicted in Figure 4 (b). It is obvious from the surface plot that the NDS rejection is influenced more significantly by combination of both pressure and temperature than those encountered in permeate flux. In addition, the most determining operating parameter is membrane rejection towards impurities. Thus, the best operating conditions suggested are feed temperature of 30°C and TMP of 1 bar.

However, in the lowest NDS rejection (0.038%, see Table 1), the NDS content is lower compared to the standard given in the CODEX STAN 210-1999 (NDS content: 0.04%). Therefore, it clearly shows that polypropylene based ultrafiltration membrane provides remarkable clarification performance related to NDS removal for palm kernel oil even at its lowest rejection.

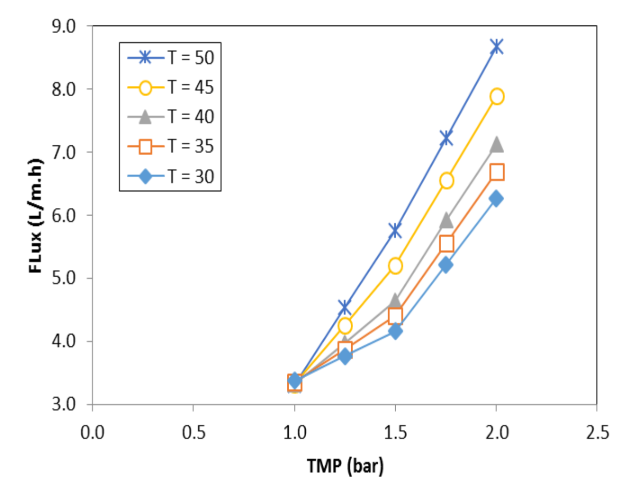


Figure 2. Effect of trans-membrane pressure on the permeate flux

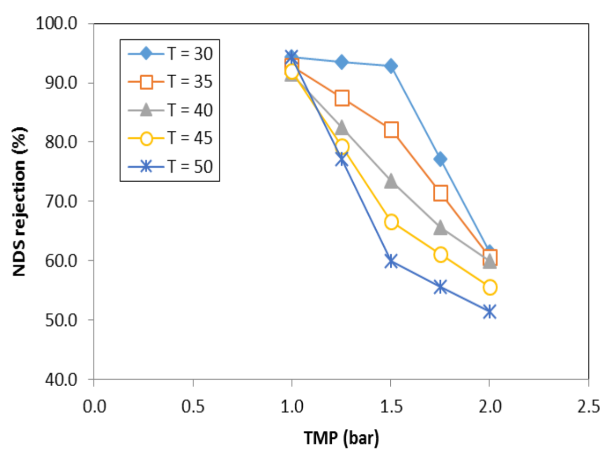
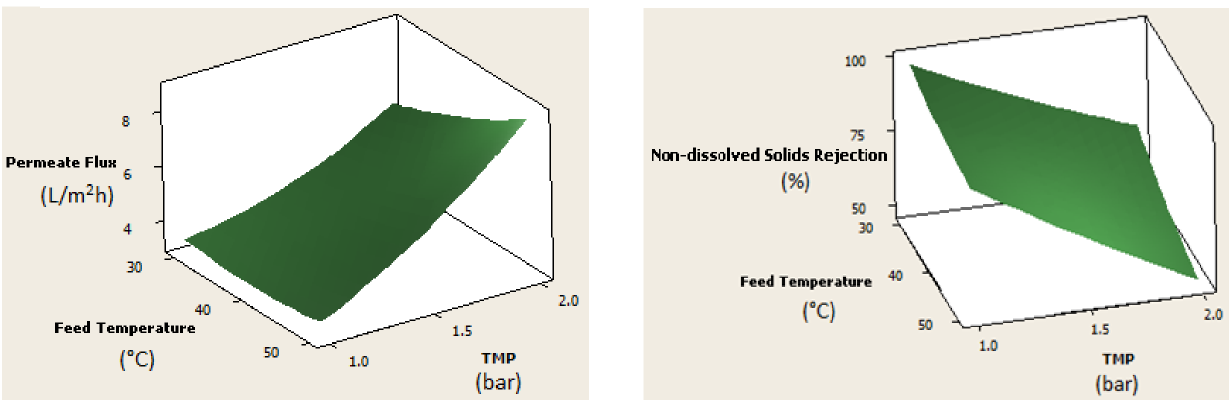


Figure 3. Effect of trans-membrane pressure on NDS rejection



(a) (b)

Figure 4. Surface plot: (a) Effect of temperature and TMP on permeate flux, (b) effect of temperature and TMP on NDS rejection

Table 1. Ultrafiltration performance parameters at various operating conditions

Temperature (°C)	TMP (bar)	Permeate flux (L/m^2.h)	NDS rejection (%)	NDS content in permeate (%)
30	1.00	3.38	94	0.004
30	1.25	3.77	93	0.005
30	1.50	4.16	93	0.006
30	1.75	5.22	77	0.018
30	2.00	6.27	61	0.030
35	1.00	3.35	93	0.006
35	1.25	3.87	87	0.010
35	1.50	4.40	82	0.014
35	1.75	5.55	71	0.022
35	2.00	6.70	61	0.030
40	1.00	3.32	91	0.007
40	1.25	3.98	82	0.014
40	1.50	4.69	72	0.021
40	1.75	5.93	66	0.026
40	2.00	7.12	60	0.031
45	1.00	3.31	92	0.006
45	1.25	4.26	79	0.016
45	1.50	5.20	67	0.026
45	1.75	6.55	61	0.030
45	2.00	7.90	56	0.034
50	1.00	3.31	94	0.004
50	1.25	4.54	77	0.018
50	1.50	5.76	60	0.031
50	1.75	7.22	56	0.034
50	2.00	8.67	51	0.038

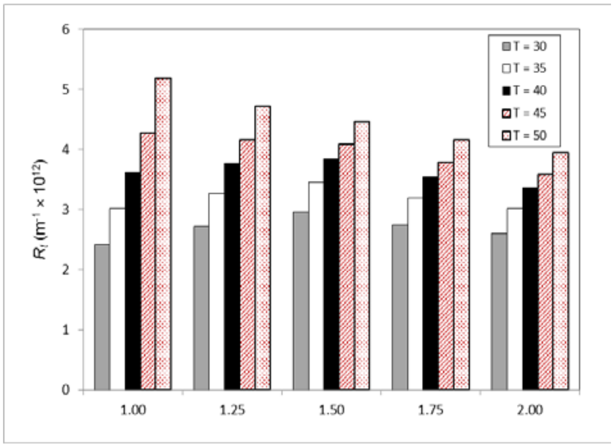


Figure 5. Hydraulic resistance (R_t) during palm kernel oil filtration

Performance of polypropylene ultrafiltration membrane during NDS removal from palm kernel oil is further investigated with total resistance (Rt) analysis. The total resistance is evaluated from the following equation,

$$J = \Delta P/(\mu \times Rt) \qquad (3)$$

where ΔP is TMP and μ is oil viscosity. The palm kernel oil viscosities in different temperature are obtained from literature (Timms, 1985). The effect of both temperature and TMP on hydraulic total resistance is illustrated in Figure 5. The higher feed temperature increases total resistance while the higher TMP reduces it. As previously explained, the higher TMP gives the improvement for permeation which is attributed to the higher driving force provided. Furthermore, the improvement of permeation rate can reduce the possibility of polarization layer formation thus it reduces total resistance. The results also indicate that total resistance is significantly reduced by applying high TMP when the feed temperature is increased as well. On the other hand, the feed temperature gives negative effect to the total resistance since the Rt increases with the increase of feed temperature. Moreover, the effect of temperature on Rt is declined when the TMP is higher. It possibly implies that the higher feed temperature makes the oil permeate easily through the membrane pores. However, the more oil amount entrapped in the membrane pore gives the more mass transfer resistance. In membrane filtration, some particles could interact with the membrane by adsorption or locating in pores which can be both surface and internal interactions (Chen *et al*., 1997). This internal interaction may gives the addition to mass transfer resistance. Therefore, in order to drive the oil coming out from the membrane pore to the permeate side, the more driving force is required.

Case Study for a 10 m^3/h Clarification Plant

Case study of a 10 m^3/h plant is considered to evaluate theoretical required membrane area (Areq), power consumption (P), and specific energy consumption (Esp) for various TMP and feed temperature (T). These parameters contribute to capital and opeарational cost. Power consumption and specific energy consumption comprise of electrical and thermal energy which are mainly used for pumping and heating, respectively. A summary of the analysis is presented in Table 2.

Table 2. Technical analysis of 10 m^3/h clarification plant

TMP (bar)	T (°C)	A_{req} (m^2)*	P (kW)**	E_{sp} (kWh/m^3)
1.00	30	2962	34.10	3.41
1.25	30	2654	34.21	3.42
1.50	30	2404	34.33	3.43
1.75	30	1917	34.44	3.44
2.00	30	1595	34.56	3.46
1.00	35	2988	68.21	6.82
1.25	35	2582	68.33	6.83
1.50	35	2273	68.44	6.84
1.75	35	1802	68.56	6.86
2.00	35	1493	68.68	6.87
1.00	40	3015	102.81	10.28
1.25	40	2514	102.93	10.29
1.50	40	2132	103.04	10.30
1.75	40	1686	103.16	10.32
2.00	40	1404	103.27	10.33
1.00	45	3018	137.89	13.79
1.25	45	2349	138.00	13.80
1.50	45	1923	138.12	13.81
1.75	45	1527	138.24	13.82
2.00	45	1266	138.35	13.84
1.00	50	3020	173.45	17.34
1.25	50	2204	173.56	17.36
1.50	50	1736	173.68	17.37
1.75	50	1385	173.80	17.38
2.00	50	1153	173.91	17.39

* Theoretical required membrane area is calculated from flux

** Power consumption is calculated from pump and heat exchanger; Pump efficiency is 60 %; Heat exchanger efficiency is 80 %; PKO specific heat is taken from Timms, 1985; Tf_{eed} = 25 °C

An increase in feed temperature enhances the permeat flux, so decreasing the required membrane area and, therefore, the investment cost for membrane. However, a high feed temperature requires more heat energy from heat exchanger which in turns increases the operational costs. Consequently, an optimization between membrane costs and energy costs must be considered while increasing the feed temperature.

On the other hand, an increase in TMP (within this range) significantly improves the permeate flux while gives a negligible effect to electrical consumption. Thus, the improvement of permeate flux by increasing TMP is more attractive for reducing capital cost related to required membrane than those encountered in feed temperature. However, it should be noted that increasing both TMP and feed temperature resulting in rejection deterioration (as explained in previous section). Therefore, an optimization between costs and product quality must be considered.

CONCLUSION

This study indicates the effectiveness of membrane processes in the palm kernel oil treatment. From the experimental results it can be concluded that polypropylene hydrophobic hollow fiber membrane can be used for palm kernel oil clarification which is related to NDS removal. The operating conditions, which are feed temperature and TMP, influence membrane performances, both permeate flux and rejection of NDS in different ways. Temperature and TMP have proportional effect to permeate flux. In contrast, they have inverse effect to rejection of NDS. During the experiment, permeate fluxes and rejections of NDS varied from 3.4 to 8.7 L/m^2.h and from 51 to 94%, respectively. The best operating conditions suggested are feed temperature of 30°C and TMP of 1 bar which produce the highest NDS rejection. In addition, the permeate quality can meet the requirement of standard NDS content even at its lowest rejection level which shows the remarkable performance of membrane filtration.

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