


Concentrating emulsified oily wastewater by integrated membrane technology

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ABSTRACT

The effects of operating pressure, feed temperature, oil content of feed solution, and membrane surface flow rate on membrane flux, concentration multiple, and average particle size of oil droplets in the concentrated solution during the single-stage membrane concentration process were investigated. The experimental results show that within a certain operating range, the membrane flux increases with the increase of operating pressure, feed temperature, and membrane surface flow rate, while it decreases continuously with the increase of feed oil content. Optimal conditions for single stage concentration based on membrane flux, concentration factor, and average particle size of oil droplets in the concentrated solution were determined. Then, on the basis of single-stage membrane concentration conditions, three different pore size separation membranes are combined in different ways. After concentration through multi-stage membranes, the particle size of the oil droplets in the concentrate is greater than 20 μm , which is beyond the particle size range of the emulsified oil, and the concentration of the oil in the concentrate is 20–30 times that of the original oil. The method can realize the recovery of oil resources in the emulsified oil-containing wastewater.

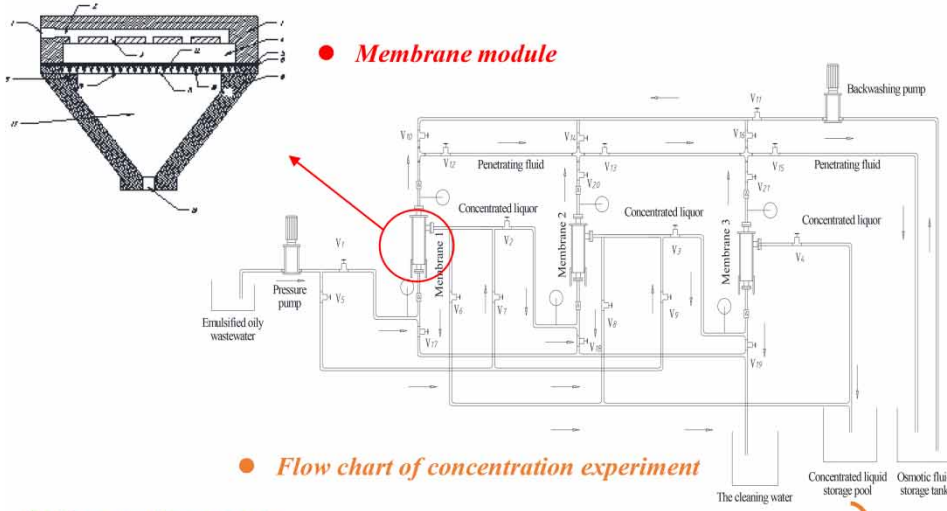
Key words: concentration multiple, emulsifying oily wastewater, integrated membrane, membrane flux, oil droplet size

HIGHLIGHTS

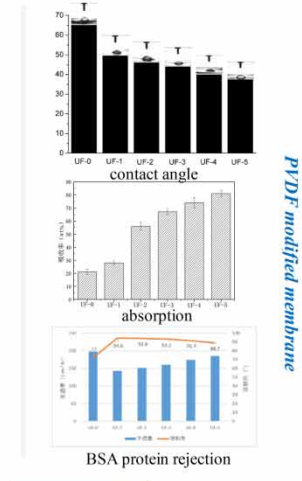
- Under the optimal membrane separation conditions, the concentration rate reaches the highest.
- An integrated membrane can effectively concentrate oily wastewater.
- The oil in the concentrated wastewater can be better recovered.

GRAPHICAL ABSTRACT

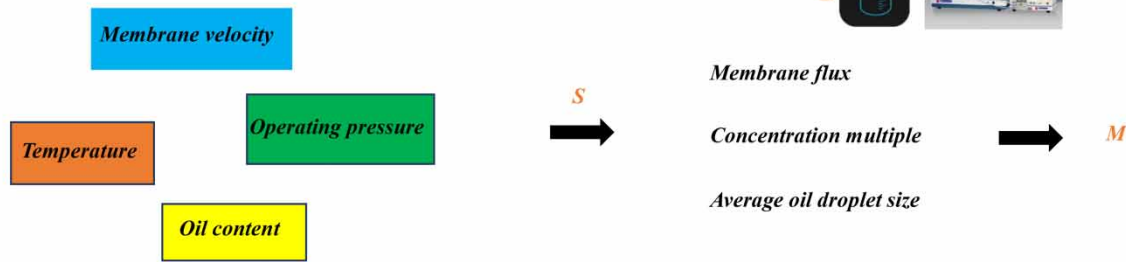
Abstract



1. Primary device



2. Research contents



1. INTRODUCTION

Membrane separation technology is an efficient and environmentally friendly separation process. Different from traditional filtration, the membrane has a selective separation function, which can realize separation (Zhang *et al.* 2021), purification (Alias *et al.* 2019), and concentration (Tanudjaja *et al.* 2019) of different components of solid-liquid. The separation process is a purely physical process, which has the characteristics of no phase change and energy-saving, and is widely used in the treatment of emulsified oily wastewater. In addition, membrane separation technology is widely used in municipal (Zhao *et al.* 2021), petrochemical (Ahmad *et al.* 2020), food processing (Wang *et al.* 2020), and other wastewater treatment processes and environmental protection industries.

Helali *et al.* (2020) used polyamide-imide (PAI) to prepare hydrophilic and underwater ultra-hydrophobic microfiltration membranes by the non-solvent-induced phase conversion method, and modified them by polyvinylpyrrolidone and polyethylene glycol, further improving their hydrophilic properties. The experimental results showed that the modified membrane showed a 98% oil rejection rate, and the permeation flux was as high as 210 L/(m²·h). Under the condition of polyethylene glycol (PEG) 6 kDa and polyvinylidene fluoride (PVP) 10 kDa ratio, the underwater oil contact angle of the modified membrane was more than 150°, and the flux recovery rate was as high as 98%. Yuliwati *et al.* (2011) modified a polyvinylidene fluoride (PVDF) ultrafiltration membrane with LiCl·H₂O and TiO₂ by the phase conversion method in a study. The wastewater used in the experiment was oily wastewater from the refinery. The experimental results showed that when the concentration of TiO₂ was 1.95%, the hydrophilicity of the membrane and the porosity of the membrane reached the highest. It was also found that when pH = 6.9, the maximum flux was 82.5 L/(m²·h), and the retention rate was 98.8%. This study shows that PVDF ultrafiltration membrane can treat oily wastewater from oil refineries.

From the above documents, it can be seen that in the process of wastewater treatment through the single-stage membrane at home and abroad, more research is focused on the preparation and modification of membrane materials by preparing the

corresponding hydrophilic membrane to improve the separation performance of the separation membrane, reduce the pollution rate of the separation membrane, and achieve a better separation effect. However, there are few studies on the concentration of oil on the side of the intercepted liquid, the size of oil droplets, and subsequent treatment issues after membrane treatment (Zhou *et al.* 2008, 2017; Chen *et al.* 2019; Lee *et al.* 2020). In the engineering application of membrane technology to treat oily wastewater, the post-treatment of intercepted liquid cannot be avoided. Through the comprehensive consideration and research of membrane separation process parameters, the reasonable integration of membrane process, and membrane pollution, the effective separation of intercepted liquid after membrane treatment can be achieved, so that the permeate can meet the requirements of drainage or recycling and achieve the recovery and reuse of oil, which is of great significance for environmental protection and effective use of energy.

In this paper, emulsified oil is prepared with diesel oil and water, and oily wastewater is simulated. The effects of several aperture separation membranes on the treatment of emulsified oily wastewater under different conditions are discussed. The reasonable combination and optimum process for oil concentration are studied.

2. EXPERIMENT PART

2.1. Experimental materials and instruments

2.1.1. Experimental instruments

The instruments used were electronic balance AE224; pure water machine GT-30; electric mixer JJ-1A; laser particle size analyzer BT-9300H; vacuum drying box DZF-6020; electric heating constant temperature water bath pot HWZ-2; glass dryer 450 mm.

2.1.2. Experimental materials

Deionized water (DI water), Tween 80 (Tween80), anhydrous sodium sulfate (Na_2SO_4), hexane (C_6H_{14}), 98% sulfuric acid (H_2SO_4), and sodium chloride (NaCl) were all analytically pure; microporous membrane (PP, pore size: 0.22 μm ; 0.45 μm ; 0.8 μm); plate membrane module; diesel fuel.

2.2. Preparation of emulsified oily wastewater

Around 1 g of diesel oil as base oil is weighed by using an electronic balance, 1,000 mL of DI water is added, and 0.1 g of Tween 80 as a surfactant is added and placed in an electric stirrer with 2,500 r/min stirring speed for 50 min. The obtained white emulsion is emulsified oil and stored in a glass dryer at room temperature.

2.3. Measurement of oil droplet size

Oil droplet size is one of the important parameters to identify the stability of emulsified oil (Matos *et al.* 2016; Shi *et al.* 2019). In the membrane concentration experiment, the concentration effect of emulsified oil under different operating conditions was investigated by measuring the average particle size of oil droplets in the concentrated solution. In this experiment, the BT-9300H laser particle size analyzer was used to measure the particle size of oil droplets. The refractive index of diesel oil is 1.45 when the background value is 1–4; the system background calibration is carried out and then the prepared emulsified oily wastewater is added. When the shading rate is 15–20, the measurement is carried out.

2.4. Determination of oil concentration

The oil content in oily wastewater can be determined by the gravimetric method, fluorescence spectrophotometry, ultraviolet spectrophotometry, and infrared spectrophotometry (Wei 2011; Padaki *et al.* 2015; Khorshid *et al.* 2021). After membrane concentration, the oil concentration in the concentrate will become larger, and the weight measurement method has a wide range of measurements and is not limited by the oil standard, so this experiment chooses the cumbersome weight measurement method.

The concentration of oil in oily wastewater is calculated by the following formula:

$$C = \frac{(m_1 - m_2) \times 10^3}{V} \quad (1)$$

where C is the oil concentration in oily wastewater (mg/mL); m_1 is the total weight of the beaker and oil (g); m_2 is the weight of the beaker (g); V is the collected water sample volume (mL).

The concentration multiple in the concentration experiment of oily wastewater was calculated by the following formula:

$$N = \frac{C_1}{C_0} \quad (2)$$

where N is the concentration multiple; C_1 is the concentration (mg/mL); C_0 is the raw material concentration (mg/mL).

2.5. Calculation of membrane flux

After calculation, the effective filtration area of the membrane in the membrane pool was 113.0973 cm². In the concentration process, the membrane flux is an important indicator to investigate the membrane separation performance, which is calculated by the following formula:

$$J = \frac{V}{AT} \quad (3)$$

where V is the permeation liquid product (L); A is the filtration area (m²); T is the filtration time (h); J is the membrane flux (L/(m²·h)).

2.6. Concentrator and process

The concentration device is a set of integrated membrane concentration equipment designed by the laboratory for oily wastewater treatment, and the experimental process is shown in Figure 1. The system includes different membrane pore size units, and each unit can operate independently or continuously for multi-stage operations according to the experimental research needs, mainly concentrating emulsified oily wastewater, and concentrating different stages according to different needs. In addition, the core device of the concentration experiment is the cross-flow filtration plate membrane module. The cross-flow filtration plate membrane module of the experimental device is completed by self-made in the laboratory. The membrane pool profile is shown in Figure 2. Before the experiment starts, it is necessary to compact the filter membrane, open the feed

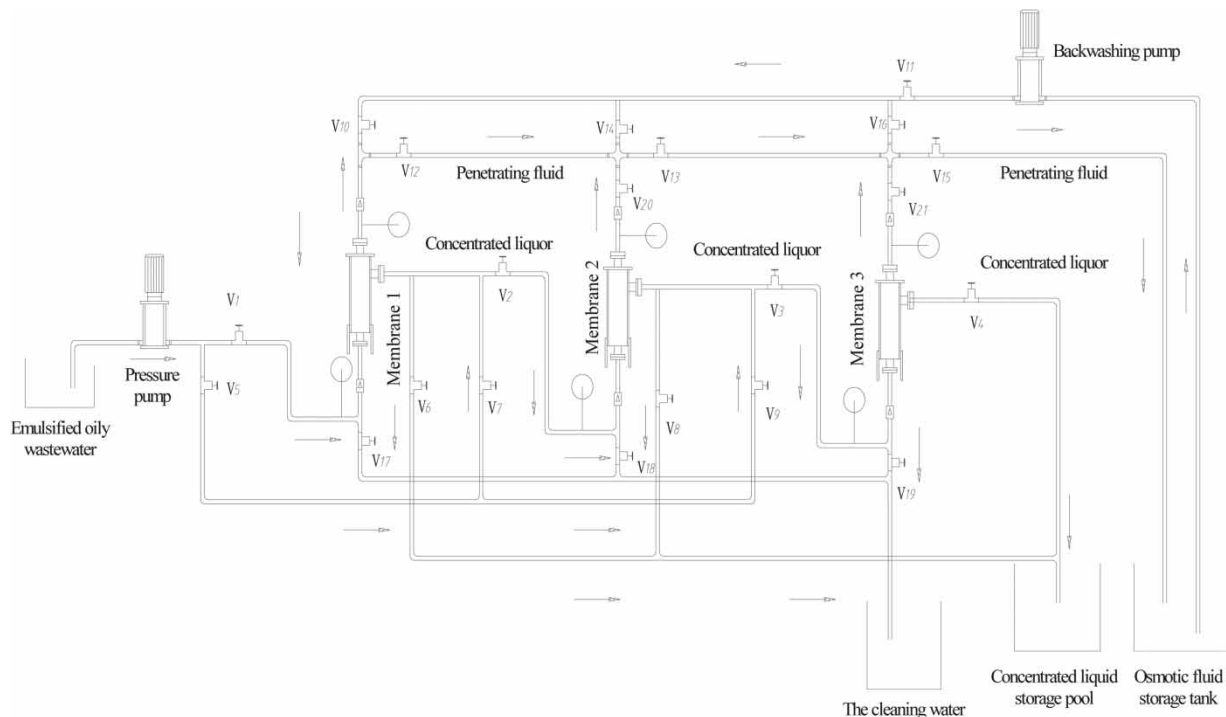


Figure 1 | Flow chart of the concentration experiment.

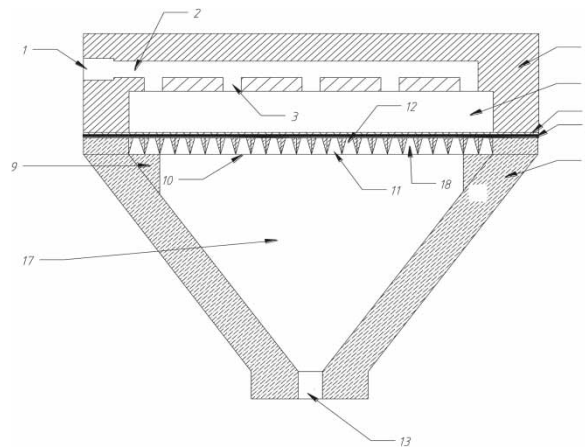


Figure 2 | Sectional view of the membrane tank in the concentration process. 1 – Raw material liquid inlet; 2 – feed liquid channel; 3 – feed liquid port; 4 – cavity; 5 – gasket; 6 – diaphragm; 7 – upper cover; 8 – lower cover; 9 – support table; 10 – support plate; 11 – permeate channel outlet; 12 – permeate channel entrance.

pump and valve, and adjust the pressure to the maximum pressure that the membrane can withstand. After entering the pure water equipment for 30 min, the concentration experiment is started.

At the same time, PVDF was used as the membrane material in the laboratory, and PVP was used as the additive. The PVDF-modified membrane was prepared by adjusting the ratio of 3-(2,3-epoxypropyl) propyltriethoxysilane (KH-561) and dopamine. The surface hydrophilicity of the modified membrane is improved, and the hydrophilicity of the inner pore of the membrane is improved. The water contact angle of the PVDF-modified membrane is reduced to 37.8° , and the wettability of the membrane is also excellent, with the highest water absorption of 81%. The membrane water flux reaches $174 \text{ L} \cdot \text{m}^{-2} \cdot \text{h}^{-1}$, and the protein retention rate reaches more than 90%. The comprehensive performance of the membrane is effectively improved, making the treatment effect of the system relatively stable. The hybrid coating forms a layer of water film on the membrane surface, which makes the membrane have good anti-pollution performance. The minimum attenuation coefficient of the modified membrane can reach 0.19. The characteristics of the filter membrane are shown in Figure 3. In order to ensure that the membrane system can operate stably for a long time, the stainless steel multi-stage centrifugal pump with good performance is adopted, and the driving force is very stable. Through online monitoring of various indicators of the membrane system, such as temperature, pH value, flow, and pressure, the whole process of membrane separation can be controlled systematically.

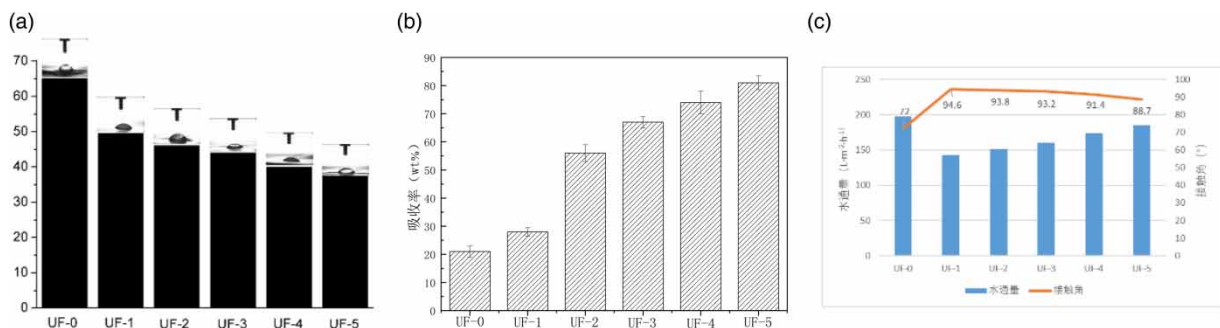


Figure 3 | Change of water contact angle of PVDF film before and after modification (UF-0 is before modification and UF-1 to UF-5 is after modification) (a), absorption change of PVDF membrane before and after modification (UF-0 is before modification and UF-1 to UF-5 is after modification) (b), pure water flux and bovine serum albumin (BSA) protein rejection of PVDF membrane before and after modification (UF-0 is before modification and UF-1 to UF-5 is after modification) (c).

3. RESULT AND DISCUSSION

3.1. Single-stage membrane concentrating oily wastewater

3.1.1. Effect of operating pressure on the concentration of oily wastewater

The feed temperature was set at 30 °C, the oil content was 1%, the feed flow rate was 150 mL/min, and the operating pressure was controlled at 0.1–0.5 MPa. The effects of operating pressure on membrane flux, oil droplet size, and concentration ratio in the concentrate were investigated, as shown in Figure 4.

Figure 4(a)–4(c) shows the effect of operating pressure on the membrane flux, the concentration ratio and the average particle size of concentrated oil droplets. From the figure, in a certain range with the increase of operating pressure, the membrane flux is also increasing. For the membrane with the pore size of 0.22 μm , when 0.1 MPa $< P < 0.4$ MPa, the membrane flux increased from 229.145 to 330.242 $\text{L}\cdot\text{m}^{-2}\cdot\text{h}^{-1}$. Similarly, for the membrane with a pore size of 0.45 μm , when the operating pressure was 0.1 MPa $< P < 0.3$ MPa, the membrane flux increased from 240.391 to 335.751 $\text{L}\cdot\text{m}^{-2}\cdot\text{h}^{-1}$, and the operating pressure of the membrane with a pore size of 0.8 μm was 0.1 MPa $< P < 0.2$ MPa. The membrane flux increased from 320.32 to 340.23 $\text{L}\cdot\text{m}^{-2}\cdot\text{h}^{-1}$. It can be seen that within a certain operating pressure range, the membrane flux of membranes with different pore sizes increases linearly with the increase of pressure, which is because there is almost no pollution in the membrane pores at the initial stage of filtration (or when the pressure is low), and the fouling layer on the membrane surface is very thin. The membrane flux increases linearly with the increase of pressure, and the variation trend of oil droplet size and concentration multiple is basically the same as that of membrane flux. With the increase of pressure to a certain extent, the increase of specific resistance of the pollution layer partially offsets the increase of membrane flux caused by the increase of pressure, which slows down the increase of membrane flux and even tends to be stable, and the change of oil droplet size and concentration multiple tends to be flat. With the further increase of pressure, the growth rate of filtration resistance exceeds that of pressure, resulting in the attenuation of membrane flux and the decrease of concentration factor.

Considering the membrane flux, the concentration ratio, the average particle size of oil droplets in concentrated solution and energy consumption, the operating pressures of the three separation membranes were 0.4 MPa (membrane pore size 0.22 μm), 0.3 MPa (membrane pore size 0.45 μm) and 0.2 MPa (membrane pore size 0.8 μm), respectively.

3.1.2. Effect of feed temperature on the concentration effect of oily wastewater

The feed pressure distributions of the three separation membranes were 0.4 MPa (membrane pore size 0.22 μm), 0.3 MPa (membrane pore size 0.45 μm), and 0.2 MPa (membrane pore size 0.8 μm). The feed oil content was 1%, and the feed flow rate was 150 mL/min. The feed temperature was controlled from 25 to 45 °C, respectively. The effects of feed temperature on membrane flux, oil droplet size in concentrate, and concentration ratio were investigated. The results are shown in Figure 5.

Figure 5(a) shows the effect of feed temperature on membrane flux. The membrane flux of the three separation membranes increased from 221.59 to 261.37 $\text{L}\cdot\text{m}^{-2}\cdot\text{h}^{-1}$ (membrane pore size 0.22 μm), from 240.39 to 296.45 $\text{L}\cdot\text{m}^{-2}\cdot\text{h}^{-1}$ (membrane pore size 0.45 μm), and from 310.32 to 371.56 $\text{L}\cdot\text{m}^{-2}\cdot\text{h}^{-1}$ (membrane pore size 0.8 μm), showing an approximate linear growth. There may be several reasons for the increase in the membrane flux: on the one hand, the viscosity of liquid decreases

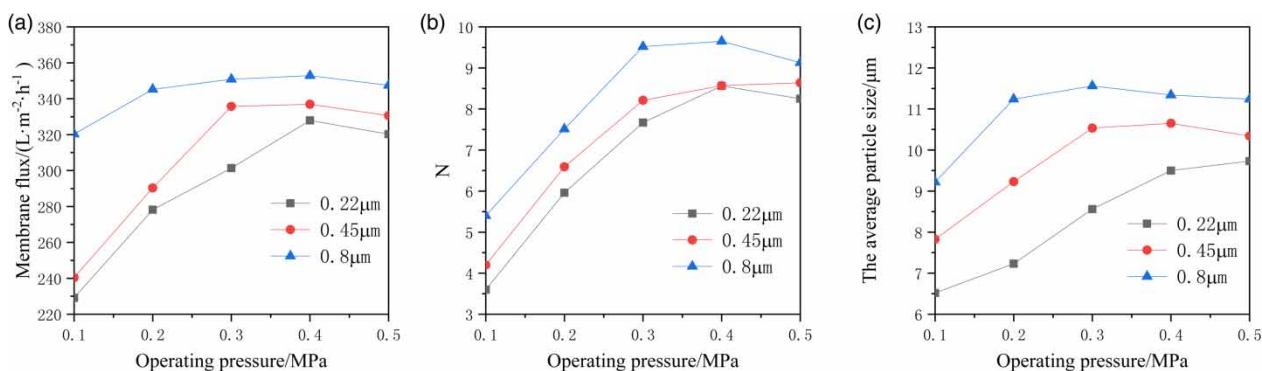


Figure 4 | Effect of operating pressure on membrane flux (a), concentration multiple (b), and average particle size (c) of oil droplets.

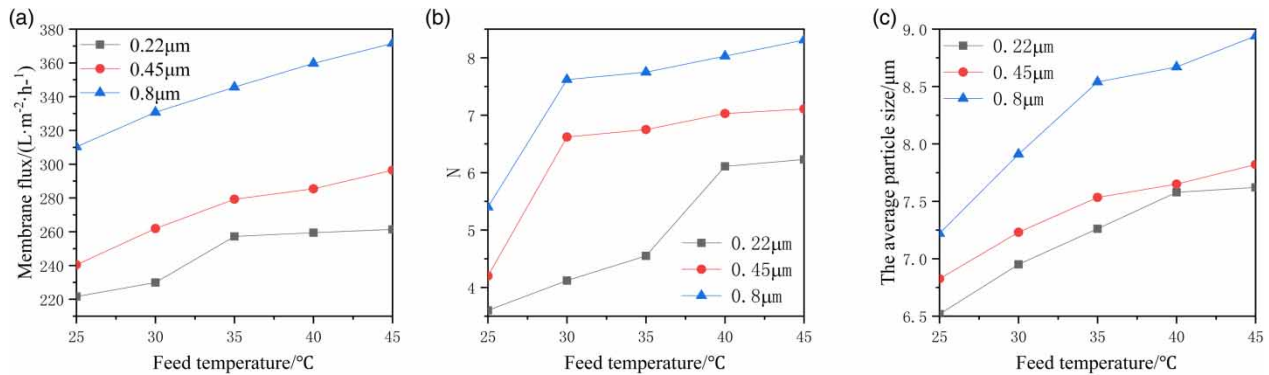


Figure 5 | Effect of feed temperature on membrane flux (a), concentration multiple (b), and average particle size (c) of oil droplets.

with the increase of temperature, which improves the flow condition of liquid; on the one hand, the increase of liquid temperature is beneficial to increase the diffusion coefficient of oil droplets, which makes the oil droplets trapped on the membrane surface return to the main fluid and reduces the speed of forming concentration polarization on the membrane surface; on the one hand, increasing the feed temperature increases the membrane pore and reduces the resistance of solvent water through the membrane pore; on the other hand, due to the existence of hydrogen bonds, water molecules exist in the form of association. With the increase of solid-liquid temperature, the movement of water molecules is accelerated, which reduces the association effect and the association size, and makes it easier for water molecules to pass through the separation membrane, thereby improving the membrane flux (Li *et al.* 2008; Wang 2014). However, it is not that the higher the temperature is, the better the membrane will be. Higher temperatures will cause damage to the internal structure of the membrane and shorten the service life of the membrane. In addition, increasing temperature will increase energy consumption. It is not economical to obtain higher membrane flux by increasing the liquid temperature. In the actual concentration process, with the increase of equipment running time, the liquid temperature will also rise.

It can be seen from Figure 5(b) and 5(c) that the concentration ratio and the particle size of oil droplets in the concentrated solution increase with the increase of temperature, but the increase is small. This is because, with the increase of temperature, the average particle size of oil droplets in emulsified oily wastewater decreases gradually. In addition, the increase of temperature leads to the increase of membrane pores. Smaller oil droplets will enter the osmotic solution through the membrane pores, and the increase of temperature will destroy the membrane pores. Oil droplets will adhere to the membrane pores when passing through the membrane pores, resulting in the blockage of the membrane pores.

Considering the membrane flux, the concentration ratio, the average particle size of oil droplets in concentrated solution and energy consumption, the appropriate temperature range of the separation membrane was 25–30 °C.

3.1.3. Effect of oil content in feed on the concentration of oily wastewater

The feed pressure distributions of the three separation membranes are 0.4 MPa (membrane pore size 0.22 μm), 0.3 MPa (membrane pore size 0.45 μm), and 0.2 MPa (membrane pore size 0.8 μm), the feed temperature is 25 °C, the feed flow rate is 150 mL/min, and the feed oil content is 1–5%, respectively. The effects of the oil content of the feed liquid on the membrane flux, the particle size of the oil droplets in the concentrate, and the concentration ratio are investigated. The results are shown in Figure 6.

Figure 6(a)–6(c) shows the effect of oil content on the membrane flux, the concentration ratio and the average particle size of oil droplets. When the oil content of the feed liquid is low, the membrane flux decreases with the increase of the oil content of the feed liquid. When the oil content of the feed liquid is high, the membrane flux decreases slightly and tends to be stable with the increase of the oil content of the feed liquid (Hu 2021). With the increase of feed liquid, the membrane surface will increase the reservoir cover surface, resulting in the decrease of membrane flux, the increase of membrane fouling and the decrease of concentration ratio. On the other hand, increasing the oil content of the feed liquid will increase the viscosity of the feed liquid, which makes the flow state of the feed liquid worse, and the oil droplets are easier to adhere to the plug hole or to the membrane surface, which accelerates the membrane fouling and the concentration polarization of the membrane surface. The above two aspects lead to the decrease of membrane flux. The lower oil content will increase the

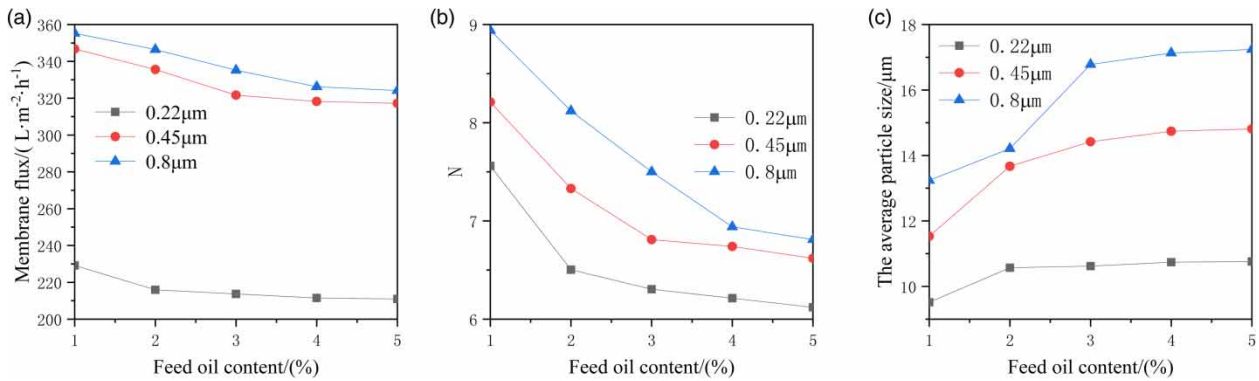


Figure 6 | Effect of feed concentration on membrane flux (a), concentration multiple (b), and average particle size (c) of oil droplets.

chance of collision between oil droplets, resulting in the re-aggregation of oil droplets and the increase of the average particle size of oil droplets. When the oil content of the feed solution is high, a small part of water molecules enters the permeate through the membrane pores, and the increase in the average particle size of oil droplets in the concentrated solution decreases.

Considering the membrane flux, the concentration ratio, the average particle size of oil droplets in concentrated solution and energy consumption, the oil content of feed liquid of the three separation membranes was 2% (membrane pore size 0.22 μm), 3% (membrane pore size 0.45 μm), and 4% (membrane pore size 0.8 μm), respectively.

3.1.4. Effect of membrane surface velocity on the concentration of oily wastewater

The feed pressure distributions of the three separation membranes were set as 0.4 MPa (membrane pore size 0.22 μm), 0.3 MPa (membrane pore size 0.45 μm), and 0.2 MPa (membrane pore size 0.8 μm), and the feed temperature was 25 °C. The feed oil contents were 2% (membrane pore size 0.22 μm), 3% (membrane pore size 0.45 μm), and 4% (membrane pore size 0.8 μm), respectively. The flow rates of the membrane surface were controlled at 150–350 mL/min, respectively. The effects of the membrane surface flow rate on membrane flux, oil droplet size, and concentration multiple in concentrated solution were investigated, and the results are shown in Figure 7.

Figure 7(a)–7(c) shows the effects of membrane surface velocity on the membrane flux, concentration multiple, and the average particle size of concentrated oil droplets. With the increase of outlet flow rate, the membrane flux first increases and then decreases. When the flow rate is low, the oil droplets stay on the membrane surface for a long time, and the oil layer gap on the membrane surface decreases. At the same time, the continuous accumulation on the membrane surface will gradually form an oil layer, resulting in low membrane flux. With the continuous increase of flow rate, the turbulent part in the cross-flow filtration membrane tank increases, which reduces the residence time of oil droplets in emulsified oily wastewater on the membrane surface, and then increases the gap between oil layers on the membrane surface, slows

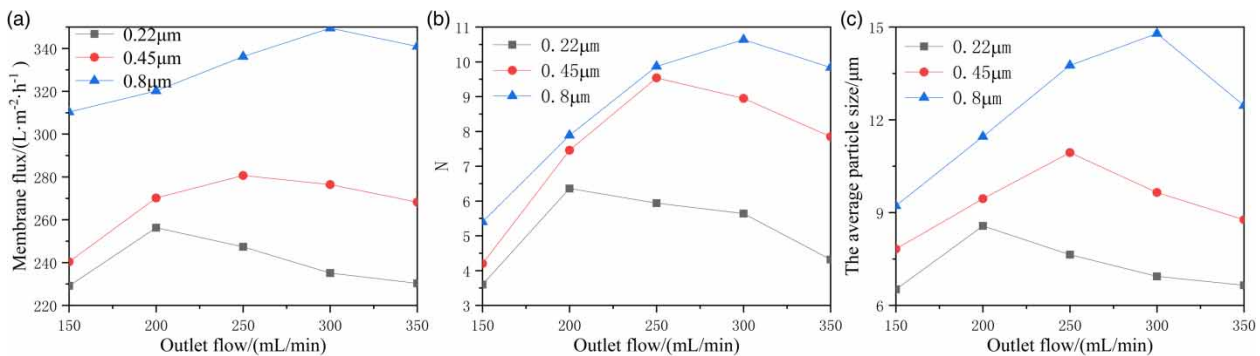


Figure 7 | Effect of membrane surface velocity on membrane flux (a), concentration multiple (b), and average particle size (c) of oil droplets.

down the membrane pollution, improves the membrane flux, and increases the concentration multiple, which increases the chance of collision and aggregation of oil droplets in the intercepted liquid and increases the particle size of oil droplets in emulsified oily wastewater. With a further increase of flow rate, the particle size of oil droplets in oily wastewater decreases, and some smaller oil droplets enter the membrane pores, resulting in the blockage of membrane pores and the decrease of membrane flux (Padaki *et al.* 2015; Golshenas *et al.* 2020). The water molecules entering the permeate through the membrane hole are reduced, the concentration ratio is reduced, and the chance of coalescence between oil droplets in the concentrate is reduced, which reduces the concentration effect.

In the actual concentration process, the appropriate flow rate can improve the membrane flux, concentration multiple, and the average particle size of oil droplets in the concentrate, but energy consumption should also be considered. Based on the above analysis, the optimal flow rates of the three separation membranes are 200 mL/min (0.22 μm), 250 mL/min (0.45 μm), and 300 mL/min (0.8 μm).

3.2. Multi-stage membrane concentrating oily wastewater

The concentration and purification of oily wastewater were further realized by the reasonable combination of filter membranes with different pore sizes. The initial operating conditions of each combination is shown in Table 1.

Figure 8 shows the effects of different membrane combinations on concentration multiple and average particle size of oil droplets. The oil content in oil-containing wastewater after three-stage concentration is about 25 times higher than that before the concentration. The average particle size of oil droplets in the concentrated solution is greater than 20 μm , which exceeds the particle size range of emulsion oil. Layering occurs after a short period, so as to achieve the purpose of recovering oil resources in oil-containing wastewater. The oil concentration in the water of the oily wastewater concentrated by the secondary membrane combination is also more than 10 times higher than that before, and the average particle size of oil droplets in the concentrated solution is also close to the maximum upper limit of the particle size range of emulsified oil. The oil content in the oily wastewater after the first-stage concentration can be increased by about eight times compared with that before the

Table 1 | Operation conditions of membrane combination

Membrane combined (μm)	Operating pressure (MPa)	Oil condition content (%)	Outlet velocity (mL/min)	Material temperature ($^{\circ}\text{C}$)
0.22–0.45	0.4	2	200	30
0.22–0.8	0.4	2	200	
0.45–0.8	0.3	3	250	
0.22–0.45–0.8	0.4	2	200	

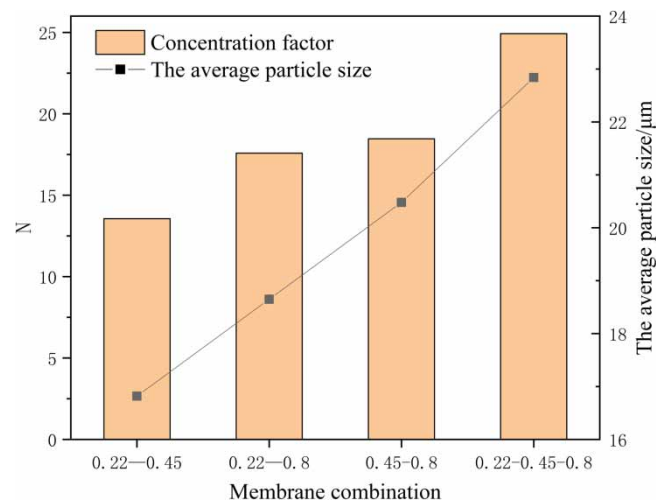


Figure 8 | Effect of different membrane combinations on concentration multiple and average particle size of oil droplets.

concentration. Whether it is primary, secondary, or tertiary concentration, the concentration ratio and the average particle size of oil droplets in the concentrated solution are greatly improved, and the recovery efficiency of oil in oily wastewater is further improved.

4. CONCLUSION

In this paper, several membranes with different pore sizes were used to treat emulsified oily wastewater. The effects of operating pressure, temperature, oil content, and membrane surface flow rate on membrane flux, the average particle size of oil droplets, and concentration factor were investigated, respectively. The reasonable operating parameters of membranes with different pore sizes were obtained, and the following conclusions were obtained.

In a certain range of operating conditions, with the increase of operating pressure, feed temperature and membrane surface flow rate, the membrane flux increases, whereas with the increase of oil content of feed liquid, the membrane flux decreases. According to the membrane flux, the concentration ratio, and the average particle size of oil droplets in the concentrate, the optimum conditions for single-stage concentration were determined as follows: the membrane pore size was 0.22 μm (0.4 MPa, 2% oil content, 200 mL/min); the membrane pore size was 0.45 μm (0.3 MPa, 3% oil content, 250 mL/min); the membrane pore size was 0.8 μm (0.2 MPa, 4% oil content, 300 mL/min); the operating temperature range was 25–30 °C.

In addition, the effects of several different membrane combinations on concentrating oily wastewater were investigated. After multi-stage membrane concentration, the average particle size and concentration multiple of oil droplets in the concentrated solution were greatly improved. After three-stage membrane combination concentration, the particle size of oil droplets in emulsified oily wastewater is greater than 20 μm , which exceeds the particle size range of emulsified oil. After a short time of static, a layer of floating oil can be seen on the surface of the concentrated liquid of emulsified oily wastewater. After a short time of static, obvious stratification occurs. It is easy to realize oil–water separation by using a liquid separation bottle (or skimmer).

ACKNOWLEDGEMENTS

This research was supported by the National Natural Science Foundation of China (21276195).

DATA AVAILABILITY STATEMENT

All relevant data are included in the paper or its Supplementary Information.

CONFLICT OF INTEREST

The authors declare there is no conflict.

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First received 29 September 2022; accepted in revised form 3 April 2023. Available online 13 April 2023