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Research on oily wastewater treatment of polyamide complex ultrafiltration membrane

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Abstract. Polyamide complex ultrafiltration membrane is a new-type high-efficient parting material with high temperature resistance, corrosion resistance, organic solvent resistance, excellent chemical stability, high separation efficiency, easiness for purification, environment friendly, and other fine features and now it has been applied to food, chemical industry, petrochemical industry, biochemical industry, pharmacy, electronic, water, wastewater treatment, and other areas and it brings huge economic and social benefit. Under the situation shortage of energy and water resource and increasing severe environment pollution in the world, membrane technology attracts high attention of the world. The Research in the Thesis is oriented at wastewater treatment problems of polyamide complex ultrafiltration membrane. It can be found in the Research that suitable operating pressure difference of membrane process is 1.0MPa with flow velocity of 5m/s. More flux and higher rejection rate of phosphate can be acquired through coagulation-micro filtration group process with penetrating fluid as circulatory wash water.

Key words. Polyamide, Complex ultrafiltration membrane, Oily wastewater, Membrane technology.

1. Introduction

With the rapid development of economy and society, oil and oil products is generally applied to all areas of national economy and people's daily life with increasing of it and its use amount. At the time of sharply increased petroleum production simultaneously, there is a daily severe tendency in polluting environment during the process of exploiting, reserving, transporting, process, and application for oils. According to statistics, there are at least 5-10 million tons oils pouring into water and flow into ocean through various ways. Oil pollution has become one of major pollutants of water. The harm of oil pollutant for ecological environment and human

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health has attracted people's great attention. Hence, oil wastewater treatment has significant meaning for protecting water resource; maintain ecological equilibrium, and promoting economic development.

Many countries in the world make regulations and restrictions for oil concentration of discharging wastewater. The maximum oil concentration of discharging wastewater in China is 10mg/L, while some foreign countries restrict it in the range of 5-20 mg/L. To protect water resource, ecological environment, and human health, and improve economic development, it is necessary and has profound meaning to positively implement pollution prevention of oils for water. Oil wastewater has extensive sources and complex components. Oil extraction, oil refining, and oil transportation in petroleum industry and chemical industry will produce oil wastewater, while ballast water and tank washing of oil tanker, cool lubricant and steel rolling water of mechanical industry, and wastewater in food industry contain a large quantity of oils. In addition, wastewater in catering, food processing, and textile industries as well as other manufacturing industries also contains a lot of oil. Environment pollution of oils presents in its severe influence on ecological system and natural environment (soil and water). It mainly consists of the following factors: suspension oil flowing into water form oil slick that impedes air reaeration and block source of dissolved oxygen in water. Because emulsified oil and dissolved oil in water with the role of aerobe need to consume dissolved oxygen in water to generate carbon dioxide and water during decomposition process, water body will be in an oxygen deficit condition with higher concentration of carbon dioxide so as to decrease pH value of water body below to a normal range and it is not suitable for fish and aquatic organism to live. Although there is adsorption and filtration of soil layers for oil contamination, oil wastewater flows to soil and oil slick can still be formed in soil so that it is hard for air to penetrate and it prevents the propagation of microorganism, and destroy granular structure of soil layer. That oil wastewater is discharged into urban water discharge pipes has influence on water discharge equipment and urban sewage handling plant. Mixed sewage flows into biological treatment structures with its oil concentration less than 30.50 mg/L, otherwise, it will influence normal metabolic processes of activated sludge and biological membrane. There are many toxic substances in oils and their decomposition products, for instance, benzopyrene and other polycyclic aromatic hydrocarbons in water or soil will be absorbed and maintained at large quantity by organism so that it will lead to distortion. If they enter into human body through food chain, it will lead to lesion of intestines, stomach, liver, kidney, and tissues and harm human health.

Complex membrane is a new-type separation membrane developed in recent years and it is compounded by dense block layer and film base with high porosity rate. Firstly, porous support membrane is always produced, and then a rather thin and effort is spared to form a dense block layer in its surface. Support layer is prepared with phase inversion and it can be high porosity rate with adjustable structure, hence, it can effectively improve flux, mechanical property, and stability of membrane. Block layer is subject to different material for changing affinity of its surface so as to improve separation rate and anti-fouling performance of membrane. It is easier to prepare ultrathin activate separating layer with composite method compared with phase inversion and composite method is intensively applied. Preparation methods for block layer include surface coating, interfacial polymerization, and layer-by-layer self-assembly and so on.

2. Characteristics of industrial wastewater

According to the property of industrial wastewater and objective of water planning for wastewater receiving, all harmful components shall be eliminated before discharging and components for elimination are concluded as follows:

(1) Solid pollutant

Solid pollutant is always indicated with "suspension" and "turbidity". Suspension is an important water quality index and a basic task for wastewater settlement. Exist OF suspension can not only muddy water but also cause blocking, abrasion of piper and equipment and hamper wastewater treatment and recycle. Turbidity is a measure for photoconduction performance of water and its value can represent content of colloid and suspension in wastewater. Solid pollutant exists in the water with three states: dissolved state (diameter is less than 1nm), colloid state (diameter is between 1nm to 100nm), and suspension state (diameter is more than 100 nm).

(2) Dissolved organic matter that can consume dissolved oxygen (aerobic pollutant)

Matter that can consume dissolved oxygen in water through biochemistry and chemical action in wastewater is collectively called aerobic pollutant. Most aerobic pollutants are organic matter, and inorganic matters include Fe, Fe2+, S2, and CN- and so on. Therefore, in general condition, aerobic pollutant indicates organic matter. In engineering practice, biochemical oxygen demand (BOD), chemical oxygen demand (COD), total oxygen demand (TOD), and total organic carbon (TOC) and other water quality indexes are subject to description.

(3) Nutrient pollutant

N and P doped wastewater is main nutrient material for plant and microorganism. When N and P concentrations in receiving water are beyond standard, it will cause water eutrophication so that algae will spread.

(4) Acid-based pollutant

Acid-based pollutant is created by acid and alkali discharged from industrial wastewater and acid rain so that pH value of water is changed. It destroys buffer action, restrain growth of microorganism, and hamper purification of water so that water quality is worse, soil is acidified, salinized, and alkalized.

(5) Toxic pollutant

Chemical matter in wastewater that can cause toxic reaction is called toxic pollutant and it mainly consists of:

1) Inorganic toxicant

Metallic toxicant: mercury, chromium, cadmium, lead, zinc, nickel, copper, cobalt, manganese, titanium, vanadium, molybdenum, and bismuth and so on. Non-metallic toxicant: arsenic, selenium, cyanide, fluorine, sulfur, nitrite and so on. It must be pointed out that many elements that can be toxicant are necessary microelement in organism, and they can be toxic when they reach a certain limited amount.

2) Organic chemical toxicant

Most of them are artificial synthetic organic compounds and it is hard for biochemical degradation, while most of them are stronger three-cause matters (cause cancer, cause mutation, and cause distortion) with huge toxicity. It mainly covers: pesticides, phenolic compounds, polychlorinated biphenyls, polycyclic aromatic hydrocarbons, and aromatic amino compounds and so on.

3) Radioactive matter

(6) Oil pollutant

It includes "petroleum" and "animal and vegetable oil". They can form oil slick on water surface for isolate the air and water surface so that reaeration condition of water is destroyed and they can attach to surface of soil particle and body surface of animal and plant. It will influence nutrient uptake and waste discharge.

(7) Biological pollutant

It indicates pathogenic microorganism in wastewater, including pathogenic bacteria, diseases, pest egg, and virus. Hygiene indexes in water quality standard include total bacterial number and total coliform bacteria groups, and the latter of them reflects pollution state of water affected by animal waste.

(8) Sensory pollutant

Matter in wastewater can evoke color difference, turbidity, foam, and stink and other phenomenona. Although there is no huge harm, but it will bring displeasure for human senses, therefore, it is called sensory pollutant.

(9) Thermal pollution

Damage of wastewater is caused by high temperature.

3. Experiment and materials

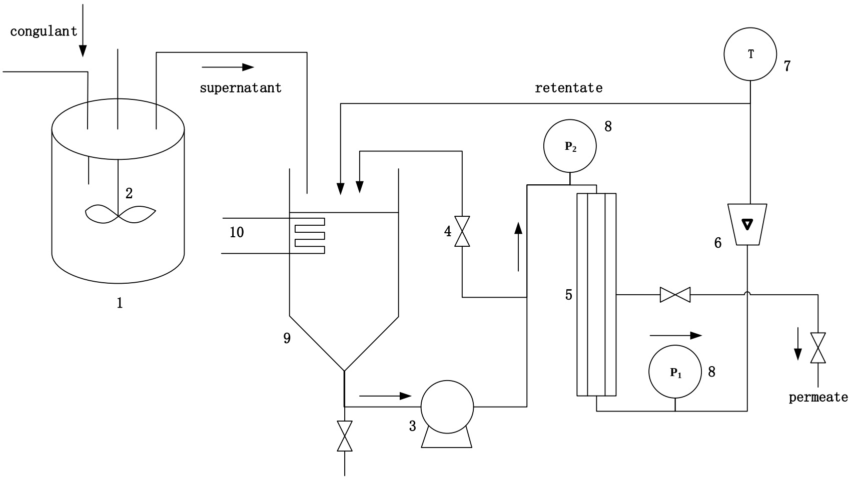
3.1. Property of wastewater

Water sample is wastewater of cathode electrophoretic paint from automobile factory with pH of 6.0-6.5, COD around 1900-2300mg/L, black grey, more solid impurities, solid content around 960mg/L, content of phosphate PO_4^3 (P)around 1.32mg/L. Experimental device of cross-flow microfiltration for polyamide complex ultrafiltration membrane available in the experiment is shown in Fig. 3-2. During membrane filtration process, when there is no attenuation for flux, the membrane filtration is in a quasi-stable state, and the flux in this stage is quasi-stable flux. Polyamide complex ultrafiltration membrane of zirconia and aluminium oxide is used in the experiment with pore diameter of 0.2μm, pore canal of 19, and pipe length of 25cm.

With potassium dichromate reflux method, $COD_{Cr}$is measured: content of PO_4^3 (P) is measured with ammonium molybdate spectrophotometric method; pH value is measured with PHS-3C precision acidity meter, and solid content of lacquer liquid is analyzed with gravimetric method, the morphology of membrane is observed in XT30 ESEM—TMP environment scanning electron microscope. All used reagent are analytical reagents.

3.2. Technical process and experimental devices

The technical process and experimental devices for wastewater treatment in the experiment are shown in Fig. 1.



1 Coagulation groove;2 Stirrer;3 Centrifugal pump;4 Side valve;5 Membrane module;6 Flow-meter;
7 Thermometer;8 Pressure meter;9 Reservoir;10 Heat exchanger;

Fig. 1. Diagram of experimental devices for the treatment of cathode electrophoretic coating wastewater with the combination process of coagulation-micro-filtration

After the coagulation treatment, the supernatant is pumped into the Polyamide Complex Ultra-Filtration Membrane System for filtration and the retention fluid is returned to the material tank. The crossflow velocity of the membrane surface is converted from the flow-meter reading; the filtration pressure difference is calculated from the inlet pressure and the outlet pressure; the permeation flux is obtained by measuring the liquid volume flowing out by stopwatch and cylinder for a certain period of time, combining with the membrane area calculation. The zirconia-polyamide complex ultra-filtration membrane tube is produced by the Greatwall New Element Membrane Technology Co., Ltd. The membrane aperture is 0.2μm and there are 19 pore canals. The pure water flux of cleaning membrane is about 800 L/ ($m^2\cdot$ h)

3.3. Experimental materials and instruments

The instruments used in the experiment are shown in table 1 respectively.

Table 1. Experimental instrument and devices

Instrument Name	Type	Manufacture
Electronic balance	JA21002	Hancping
Thermostatic magnetic heating stirrer	S21-2	SHANGHAI SILE INSTRUMENT CO., LTD.
Magnetic stirrer	82-2	SHANGHAI SILE INSTRUMENT CO., LTD.
Glass instrument airflow drier	C	Zhengzhou Greatwall Scientific Industrial and Trade Co., Ltd.
Electro- thermostatic blast oven	DHG-9146A	Shanghai Jinghong Experimental Equipment Co., Ltd.
Vacuum drying oven	DZF-6050	Shanghai Jinghong Experimental Equipment Co., Ltd.
Polyamide complex device		Laboratory homemade
Scanning electron microscope	S3000N	Hitachi
Digital polarized light microscope	BA300 pol	Motic
Universal material testing machine	WDW3020	Changchun Kexin Experimental Instrument Co., Ltd.
Positron flooded test set		American EG&G Company
Plate vulcanizing machine	XLB-D-350*350*2	Shanghai First Rubber Machinery Factory
Nano-particle size and potentiometricanalyzer	Nano ZS	British Malvern Company
Membrane filtration performance testing device		Laboratory homemade

3.4. Polyacrylonitrile (PAN) base film of polyamide complex ultra-filtration membrane

Prepare the polyamide complex ultra-filtration membrane. The photo of SEM which prepares the polyamide complex ultra-filtration membrane with the polyamide compound method has been shown in Fig. 2A. It can be seen from the figure that the PAN polyamide complex ultra-filtration membrane has a smooth surface and the average diameter of polyamide complex ultra-filtration membrane is about 300nm. After PAN nano polyamide complex ultra-filtration membrane prepared by polyamide composite is under the cold-pressing process of 5MPa pressure, then the polyamide complex ultra-filtration membrane non-woven with closer arrangement of polyamide complex ultra-filtration membrane can be obtained. As shown in Fig. 2 B, the PAN nano-polyamide complex ultra-filtration membranes after the cold-pressing process is tightly arranged and the form of polyamide complex ultra-filtration membrane is not changed. The surface is more flat and it is more suitable to be the base film of complex membrane.

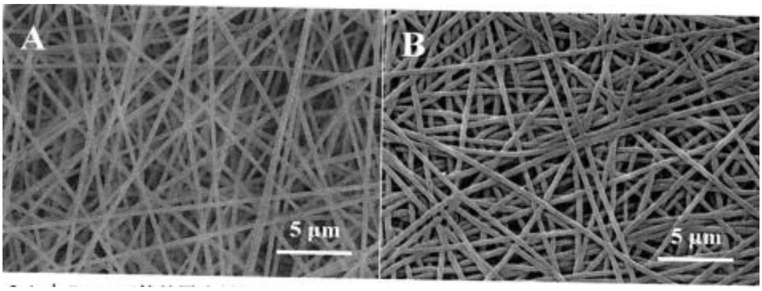


Fig. 2. Polyamide 66 composite ultra-filtration membrane [(A) the photo of stoste (B) the photo of nano-polyamide composite ultra-filtration membrane in cold-pressing process]

4. Result analyses

In previous studies, according to the beaker coagulation experiments of aluminum sulfate $AL_2(SO_4)_3 \cdot 18H_2\circ$, poly aluminum (PAC), ferric chloride, coagulant A and other treatment agents, it is found that coagulant A as a flocculant for the treatment of electrophoretic coating wastewater had a fast settling speed and a high removal rate of COD_{cr}, so it is used as the main coagulant in the following experiments.

4.1. Effects of PH value

Table 2. Effect of pH value

PH	Flocculation Condition	Sedimentation Rate	Coagulation Removal Rate	Membrane Removal Rate
1.5	Claret-red gelatinous suspension	Slow	61.9	81.2
2.6	Light-red floc	Slow	56.2	
3.5	Light-grey turbidity	No obvious stratification	50.1	67.8
5.1	Grey dreg	No obvious stratification	42.9	
6.7	Big grey-white floc unit	Fast	63.2	85.3
8.5	Big and little more grey-white floc unit	Fast	61.8	80.5
12.3	Big and more grey-white floc unit	Fast	63.2	84.8

In the process of coagulation, there is always a relative optimal pH value, so that the coagulation reaction is the fastest and the solubility of the floc is the smallest. The pH value of the incoming electrophoresis coating wastewater is 6.0~6.5. In the process of coagulation, the pH value is adjusted by adding acid and alkali to study the effect of pH value on the results of coagulation and separation, as shown in table 2. From table 2, pH value in the condition of strong acidic or strong alkaline, the removal rates of $\mathrm{COD_{cr}}$ is over 80%. But it is found in the experiment that when the pH value is lower than 3, the sedimentation rate of the floc is very slow. When the pH value is higher than 12, the more doses are added and the more floc would be formatted. When the pH value is between 6.5~8.0, the coagulation reaction speed is fast and the formation of floc is large. Coagulant A, polyelectrolyte and etc. as flocculant, when the pH value is 6.7, the contribution of coagulation process for the removal rate of $\mathrm{COD_{cr}}$ in the electrophoretic coating istewater is 63%. Then the microfiltration of polyamide complex ultrafiltration membrane made the removal rate of $\mathrm{COD_{cr}}$ in istewater increased to 85%.

4.2. Effects of operating parameters in membrane process

(1) Effect of differential pressure

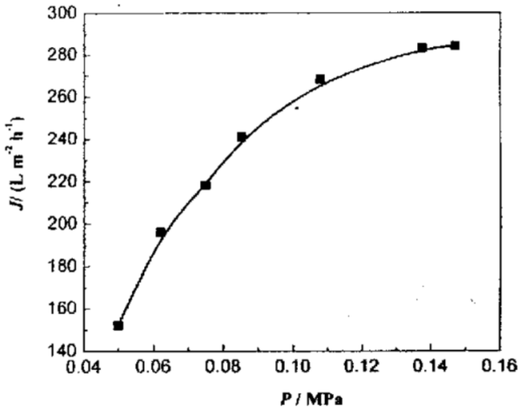


Fig. 3. Effect of pressure on filtration flux

Micro-filtration is a pressure-driven membrane process and the operating differential pressure would directly affect the membrane flux. The conditions of membrane flux changed with differential pressure while cathode electrophoretic coating wastewater is treated by adopting zirconia micro-filtration membrane had been shown in Fig. 3 when the temperature is at 30° and the crossflow velocity on surface of membrane is 4m/s. As shown in the figure, when the differential pressure increased from 0.05Mpa to 0.14Mpa, the flux would increase with the increase of differential pressure; after 0.14Mpa, the flux would basically remain unchanged. It shows that the osmosis resistance increased by further increase of driving force and increasing pollution of the surface of membrane. In industry practice, it is suitable for differential pressure to be chosen between 0.08-0.14Mpa. Because the increase of differential pressure will lead to the increase of power consumption, for comprehensive consideration, the operating differential pressure of 0.10Mpa will be chosen in this experiment.

(2) Effect of crossflow velocity on the surface of membrane

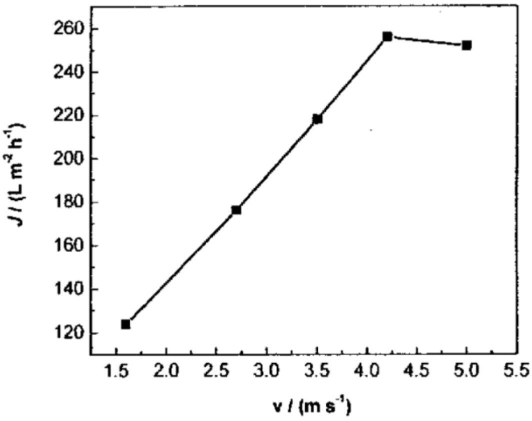


Fig. 4. Effect of crossflow velocity on filtration flux

It is generally believed that increasing the flow rate can increase the flux, but it also means the increase of circulation and energy consumption. When the temperature is at 30 ° and the differential pressure is 0.10MPa, the relationship between the cross-flow velocity and the membrane quasi stable flux is shown in Fig. 4. As shown in the figure, when the flow rate is lower than 4.2m/s, the flux increases as the flow velocity increases: But when the flow rate exceeds 4.2m/s, the flux which increased with the flow rate increases slowly or even decreases, which is similar to the phenomenon of the ultra-filtration process for cathodic electrophoretic coating. It shows that the resistance of membrane fouling increases with the increase of crossflow velocity. The crossflow velocity of 4.2m/s is chosen by the experiment.

(3) The relationship between membrane flux and cycling time

With the operating conditions of differential pressure of 0.10MPa, membrane crossflow velocity of 4.2m/s and temperature of 16~30°, the relationship between membrane flux and cycling time is shown in Fig. 5-8. At the same time, the relationship between membrane flux and cycling time when there is no coagulation pretreatment is also shown in the figure. From Fig. 5 we can see that the steady flux is obviously increased. Because the electrophoretic paint contained in the cathodic electrophoretic wastewater is colloidal solution of cationic resin type, the wastewater is entered the membrane tube without treatment, in which the charged particles are easily adsorbed and deposited in the membrane surface and the membrane hole (see Fig. 5, the ESEM of the contaminated film surface), so that the flux is reduced sharply; because of electrolyte compression double layer, adsorption, bridging and other functions, so that the wastewater with pretreatment makes the particles in waste water out of stability, accumulated and settled. After the quiescence, the supernatant are taken to the membrane tube. There are little small particles adsorbed or deposited on the membrane surface (see Fig. 5). The cake layer thins and the filtration resistance decreases so that stable flux increases. When the crossflow velocity on membrane surface is 4.2m/s, cross-membrane differential pressure is 0.10MPa and the temperature is 30°, the membrane stability flux is about 250 L/ (m^2· h). It is reported in the literature that the permeate flux can be stabilized in 2.5~3.5 L/ (m^2· h) while the cathodic electrophoretic coating is adopted the membrane treatment of hollow polyamide complex ultra-filtration membrane of charge-positive CML under the conditions of the pressure of 0.13MPa, the paint fluid flow of 200~300ml/s and the paint temperature of 30°. The amount of paint penetration of the tube-charged ultra-filtration membrane is about 20 L/ (m^2· h). Through comparison, the stable flux is higher when cathodic electrophoretic coating wastewater is treated by the micro-filtration of coagulation-polyamide complex ultra-filtration membrane, which is conducive to engineering practice.

4.3. Membrane cleaning

In the experimental process, although the appropriate operating conditions has been chosen, after a long period of operation, there will still be the phenomenon that the membrane infiltration flux decreases with the increase of practice and the membrane fouling is produced so that membrane tube must be cleaned. For different treatment systems, we selected a single cleaning agent: Acid (0.5% citric acid and 0.5% amino sulfonic acid), alkali (0.2% sodium hydroxide and 0.2 sodium carbonate) and mixed cleaning agent (0.5% amino sulfonic acid +1mL/L surfactant) to clean for 20min. The experimental results are shown in table 3.

Table 3. Cleaning effects with different cleaning agents

Cleaning Agent	Cleaning Condition	Flux Recovery Rate/%
0.2% sodium hydroxide	25 °C, 20min	38.1
0.2 sodium carbonate	25 °C, 20min	39.6
0.5% amino sulfonic	25 °C, 20min	51.2
Mixed cleaning agent	25 °C, 20min	58.3
Mixed cleaning agent	25 °C, 20min	76.8
Mixed cleaning agent	35 °C, 20min	80.2
Mixed cleaning agent	45 °C, 20min	84.2
Mixed cleaning agent	45 °C, 30min	82.6
Mixed cleaning agent	45 °C, 40min	81.1

It is shown in table 3 that the flux recovery rate is higher and the cleaning effect is better when the mixed cleaning agent is chosen to clean. In addition, for the same cleaning agent, the temperature increase accelerates the reaction rate, which is beneficial to the recovery of flux; the cleaning time shall not be too long, otherwise paint, tree wax and so on will be re-dissolved to adsorb on the membrane surface. The effective membrane cleaning method obtained from the experiment is: (1) Rinse with clean water for 15min; (2) Rinse with 0.5% amino sulfonic acid + emulsifier cleaning liquid for 20min; (3) Rinse with clean water for 20min. The flux can be recovered to 84% of the initial flux of the membrane tube. If the membrane tube is soaked in 0.5% amino acid solution for 12h, then rinse with water for several times and the flux can be recovered to about 60%.The more optimized cleaning method is needed further exploration.

5. Conclusion

Although activated carbon has a very high adsorption capacity on organic pollutants and is often used to remove organic pollutants in industrial wastewater, the activated carbon is expensive and is difficult in regeneration. What's more, it is easy for the emulsified oil droplets to block the pore canals of activated carbon surface, so that the pores will lose the activity; polyamide complex ultra-filtration membrane is cheap and easy to get and it is an economical and effective new type of adsorbent, which has a very important significance for the removal of organic pollutants in industrial wastewater and the remediation of groundwater environment. In today's world, with the situations of energy shortages, water scarcity and increasingly serious environmental pollution, the membrane technology has been highly regarded by all countries. The problem of oily wastewater treatment of polyamide complex ultra-filtration membrane is mainly studied in this paper and the effectiveness of the proposed method has been verified in the experimental results.

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