

Study on application of new membrane separation technology in advanced treatment and recycling of municipal sewage

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Abstract: A new type of anti pollution ultrafiltration membrane based on graphene oxide (GO) doped polyvinylidene fluoride (PVDF) was developed to address the problems of poor anti pollution, high energy consumption, and low resource recovery efficiency of traditional membrane technology in the deep treatment and recycling of urban sewage. A coupled process of "anaerobic/aerobic (A/O) - membrane bioreactor (MBR) - nanofiltration (NF)" was constructed to achieve efficient sewage purification and synchronous recovery of nitrogen and phosphorus resources. The results showed that the hydrophilicity and flux of GO-PVDF membrane were significantly improved, the stable flux reached 195 L/(m²·h), and the flux recovery rate after pollution reached 94.2%, which was about 15% higher than that of the original membrane. The removal rates of COD, NH-N, TP and turbidity by the integrated process exceeded 88%, 93%, 95% and 98% respectively, and the effluent quality met the standard of reuse of urban recycling water-water quality standard for industrial uses (GB/T 19923-2005). Nitrogen and phosphorus were recovered from the concentrated water side of NF by struvite crystallization, and the product purity was high. After introducing the energy recovery device, the net energy consumption of the system is reduced by 43% compared with the traditional process. This study provides technical support for low-carbon and high-efficiency regeneration and resource utilization of urban sewage.

1. Introduction

Although the traditional sewage treatment process can effectively remove organic matter and suspended solids, the removal efficiency of nitrogen and phosphorus nutrients, trace organic pollutants and emerging pollutants is insufficient, which makes it difficult for the reclaimed water quality to meet the strict standards of industrial high-precision water use, ecological water replenishment or drinking water source replenishment ^[1-2]. By converting sewage into recycled water, not only can the pressure on water resources be alleviated, but also the pollution of sewage discharge to the ecological environment can be reduced. However, the existing reclaimed water treatment technology still faces the challenges of high energy consumption, high cost and poor stability, and it is urgent to develop high-efficiency, low-carbon and sustainable advanced treatment technology.

Membrane separation technology is widely used in advanced sewage treatment and resource recovery because of its high pollutant removal capacity, modular design and good coupling with biological treatment processes. Gradient interception from particulate matter to soluble salt can be realized through microfiltration, ultrafiltration, nanofiltration and reverse osmosis, and multi-stage barrier can be constructed by combining MBR (Membrane Bioreactor) or advanced oxidation process, and it has the potential to recover resources such as nitrogen, phosphorus and lithium ^[3-4]. However, traditional membrane materials are easy to be polluted and have high energy consumption.

In this study, a new membrane separation technology suitable for advanced treatment and recycling of municipal sewage was developed, focusing on solving the problems of poor pollution resistance, high energy consumption and low resource recovery efficiency of traditional membrane technology. Develop new membrane materials with low cost and high pollution resistance;

Optimize the operation parameters and energy recovery mechanism of membrane system; Construct an integrated process of "biological treatment-membrane separation-resource recovery" to realize efficient purification of sewage and resource utilization of nitrogen and phosphorus.

2. Research method

2.1 Experimental materials and equipment

2.1.1 Experimental materials

The experiment used polyvinylidene fluoride (PVDF) and polyether sulfone (PES) as base film materials, two-dimensional materials such as graphene oxide (GO), MXene, ZIF-8, and amphiphilic polymers as modification additives, NMP and PVP as solvents and pore forming agents to prepare modified membranes ^[5]; The experimental raw water is taken from the secondary effluent of a sewage treatment plant in a certain city, with water quality characteristics of COD_{Cr} 50-80 mg/L, NH₃-N 5-10 mg/L, TN 15-25 mg/L, TP 3-5 mg/L; Standard chemical reagents were used for water quality analysis, and membrane fouling simulation experiments were conducted using bovine serum albumin (BSA), sodium alginate (SA), and humic acid (HA).

2.1.2 Experimental installation

The experiment is equipped with a complete membrane preparation and characterization system, including a phase transformation method membrane device, scanning electron microscope (SEM), atomic force microscope (AFM), Fourier transform infrared spectrometer (FT-IR), contact angle measuring instrument, and Zeta potential analyzer, for membrane structure and surface performance analysis; Using a laboratory cross flow membrane filtration device (with an effective membrane area of approximately 100 cm²) combined with a constant flow pump, pressure sensor, and electronic balance to test the permeate flux and separation performance of the membrane; Water quality analysis relies on equipment such as total organic carbon (TOC) analyzer, ion chromatography (IC), GC-MS (gas chromatography-mass spectrometry), and ICP-OES (inductively coupled plasma emission spectrometer) to achieve accurate detection of organic matter, nutrients, trace pollutants, and metal elements; At the same time, a "anaerobic aerobic bioreactor (A/O) - MBR reverse osmosis (RO)/nanofiltration (NF)" coupled pilot platform with a processing capacity of 100-200 L/d was built, integrating online pH, DO and pressure monitoring and energy recovery devices to evaluate the integrated performance of membrane processes in actual sewage treatment and reuse.

2.2 Experimental scheme design

2.2.1 Preparation and characterization of new anti-pollution membrane materials

The technology combines non solvent induced phase separation (NIPS) with surface modification ^[6-7]. By blending modification, hydrophilic nanomaterials (GO, MOFs) were incorporated into the casting solution to prepare modified flat ultrafiltration (UF) membranes. By using interface aggregation technology, a nanocomposite polyamide desalination (NF/RO) separation layer doped with two-dimensional materials was constructed on a polysulfone ultrafiltration membrane. Observe the surface and cross-sectional morphology and roughness of the membrane through SEM/AFM; Analyze the surface chemical composition of the film through FT-IR and XPS; Evaluate hydrophilicity through contact angle ^[8]. Test the pure water flux and rejection rate of BSA/HA at a pressure of 0.1-0.2 MPa, and evaluate the initial performance of the membrane based on this.

2.2.2 Study on membrane fouling behavior and optimization of operation parameters

Using dead end filtration, a constant flow filtration experiment was conducted using simulated water distribution (BSA+SA+HA+NaCl₂) and actual secondary effluent ^[9]. Real time recording of transmembrane pressure (TMP) changes and drawing of contamination curves. After the filtration, the polluted membrane was cleaned and the components of the polluted layer (TOC, polysaccharide

and protein) were analyzed. The surface adhesion of the polluted membrane was analyzed by AFM, and the types of membrane pollution (reversible/irreversible pollution) and main contributing factors were quantitatively analyzed. Orthogonal experiments were designed to investigate the effects of operating pressure (0.05-0.25 MPa), cross-flow velocity (0.5-1.5 m/s), operation period (intermittent operation/continuous operation) and physical/chemical cleaning strategies (backwashing intensity, period and concentration of chemical cleaning agent) on membrane flux maintenance and energy consumption, and to determine the optimal operation window ^[10].

2.2.3 Construction and verification of integrated process of "biology-membrane separation-resource recovery"

Coupling the prepared high-performance NF membrane unit with an A/O biological treatment unit. The A/O unit is responsible for denitrification, phosphorus removal, and degradation of most organic matter, and its effluent directly enters the NF unit. The nitrogen and phosphorus nutrients enriched on the NF concentrated water side (mainly in the form of NH_4^+ and PO_4^{3-}) are guided to the crystallization reactor and recovered by adding magnesium source to generate guano stone (MAP). Long-term monitoring of the effluent quality of the integrated system, focusing on the removal rate of COD, turbidity, TN, TP and specific emerging pollutants. Calculate the recovery rate of water resources. The struvite precipitate was collected, and its phase and morphology were identified by XRD and SEM to evaluate its resource potential.

2.2.4 Energy recovery mechanism and whole process life cycle assessment (LCA)

In NF/RO unit, the pressure exchanger model is introduced to simulate the energy recovery process of high-pressure concentrated water, and the net energy consumption (kWh/m^3) of the system is calculated, and compared with the traditional system. Based on the experimental data, the material and energy balance model of the whole process from "water inflow" to "product water and resources" is constructed ^[11]. Using LCA method, the performance of the integrated technology in energy consumption, carbon footprint and economic cost is preliminarily evaluated, and compared with the traditional advanced treatment process, so as to demonstrate its technical economy and environmental sustainability.

3. Experimental result

3.1 Properties and anti-pollution characteristics of new composite membrane materials

GO-doped PVDF ultrafiltration membrane (GO-PVDF) was successfully prepared by solvent-free phase separation. The results of characterization and performance test show that the performance of the modified membrane is significantly improved. The hydrophilicity of the modified membrane is greatly improved (Figure 1). The pollution experiment shows (Table 1) that GO-PVDF membrane shows higher initial and stable flux, and the flux decline rate is significantly reduced, and the flux recovery rate is higher after simple hydraulic cleaning. This shows that the introduction of GO can effectively reduce membrane pollution and enhance membrane anti-pollution and long-term operation stability.

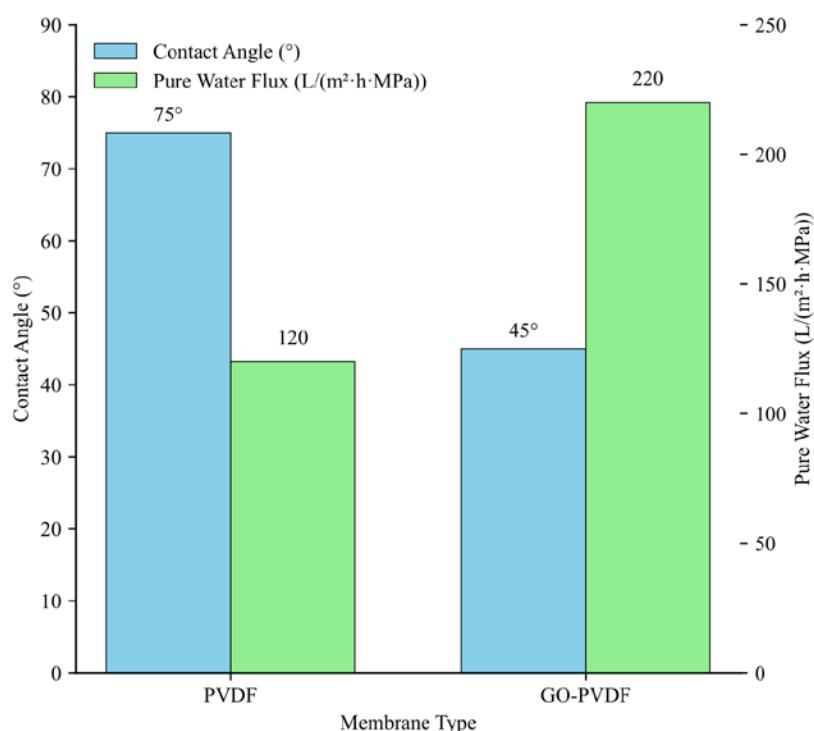


Figure 1 Comparison of membrane surface contact angle and pure water flux

Table 1 Experimental data of simulated sewage pollution by membrane filtration (operating pressure: 0.15 MPa, time: 2 h)

Membrane type	Initial flux (L/(m ² ·h))	Stable flux(L/(m ² ·h))	Flux decline rate	Recovery rate of hydraulic cleaning
Primary PVDF	180	65	63.9%	81.5%
GO-PVDF	330	195	40.9%	94.2%

3.2 Advanced treatment efficiency and resource recovery of integrated process

The "A/O-NF" coupling system was operated to continuously treat the actual secondary biochemical effluent, and the system showed excellent and stable advanced treatment effect. It can be seen from Table 2 that the water quality of the integrated process is excellent, and COD, NH₃-N, TP and turbidity are deeply removed, which completely meets the requirements of reuse of urban recycling water-water quality standard for industrial uses (GB/T 19923-2005) and other standards. NF unit has a low rejection rate for monovalent salt ions, but a high rejection rate for divalent ions and organic matter, which ensures the quality of produced water and effectively enriches nutrients in concentrated water.

Table 2 Comparison of key indexes of inlet and outlet water quality of integrated process

Water quality index	Water inflow (secondary effluent)	NF water production	Removal rate
COD _{Cr} (mg/L)	68.5 ± 10.2	8.2 ± 1.5	>88%
NH ₃ -N (mg/L)	8.2 ± 1.8	0.5 ± 0.2	>93%
TN (mg/L)	19.8 ± 3.5	4.1 ± 0.8	>79%
TP (mg/L)	3.5 ± 0.6	0.15 ± 0.05	>95%
Turbidity (NTU)	5.8 ± 1.2	<0.1	>98%

High-purity struvite ($\text{MgNH}_4\text{PO}_4 \cdot 6\text{H}_2\text{O}$) crystals were successfully recovered from NF concentrated water, realizing the resource utilization of nitrogen and phosphorus. By introducing the energy recovery device to simulate, the net energy consumption of the integrated process system is reduced by about 43% compared with the traditional high-pressure desalination process (Figure 2), showing significant energy-saving advantages.

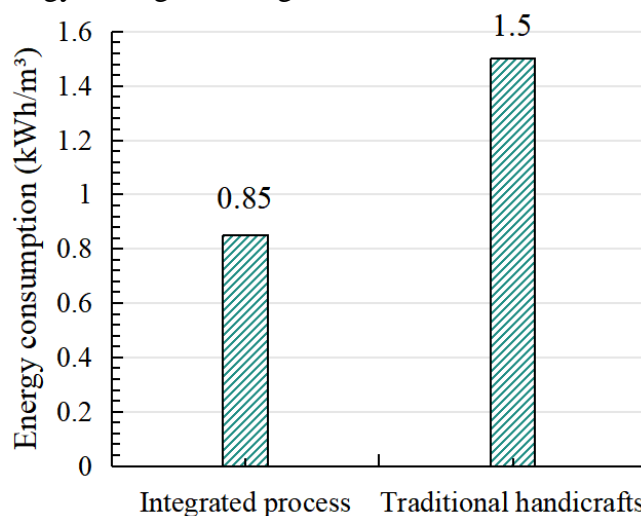


Figure 2 System energy consumption comparison

The experimental results show that the new GO-PVDF membrane developed in this study has excellent anti-pollution performance, high flux and easy cleaning. The "A/O-NF" integrated process based on this membrane can not only produce high-quality reclaimed water, but also recover nitrogen and phosphorus resources from NF concentrated water. At the same time, the overall energy consumption of the system is effectively reduced through the energy recovery mechanism, and the low-carbon goal of "purification-reuse-resource utilization" of sewage is realized.

4. Conclusion

Develop a new membrane separation technology suitable for advanced treatment and recycling of municipal sewage. GO-doped PVDF ultrafiltration membrane (GO-PVDF) was successfully prepared by combining non-solvent induced phase separation (NIPS) with surface modification, which significantly improved the hydrophilicity and anti-pollution of membrane materials. The experimental results show that the modified membrane shows higher initial and stable flux, the flux decline rate is significantly reduced, and the flux recovery rate is higher after simple hydraulic cleaning, which effectively reduces membrane pollution and enhances membrane anti-pollution and long-term operation stability.

The constructed "A/O-NF" coupling system shows excellent advanced treatment effect when treating the actual secondary biochemical effluent. The key indexes such as COD, $\text{NH}_3\text{-N}$, TP and turbidity all meet or exceed the standard requirements of reuse of urban recycling water-water quality standard for industrial uses (GB/T 19923-2005). NF unit has high rejection rate of divalent ions and organic matter, which ensures the quality of produced water and effectively enriches nutrients in concentrated water. High-purity struvite ($\text{MgNH}_4\text{PO}_4 \cdot 6\text{H}_2\text{O}$) is successfully recovered from NF concentrated water, realizing the resource utilization of nitrogen and phosphorus.

After the introduction of energy recovery device simulation, the net energy consumption of the integrated process system is reduced by about 43% compared with the traditional high-pressure desalination process, showing significant energy-saving advantages. The preliminary evaluation based on LCA method shows that the integration technology has excellent performance in energy consumption, carbon footprint and economic cost, and has technical economy and environmental sustainability. The new GO-PVDF membrane developed in this study and its "A/O-NF" integrated process can not only effectively purify sewage, but also realize resource recovery and significantly reduce energy consumption, providing an efficient, low-carbon and sustainable solution for

advanced treatment and recycling of municipal sewage.

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