RESEARCH ARTICLE

Nanofiltration membrane wastewater treatment technology for pharmaceutical enterprise wastewater

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With the rapid development of the pharmaceutical industry, the combined pollutants of microplastic particles and pharmaceutical and personal care waste in discharged wastewater have a significant impact on water ecology and human health. Therefore, developing effective wastewater treatment technologies has become an important issue for environmental protection and sustainable development. In this context, taking the molecular weight of pollutants as the starting point, a nanofiltration membrane method was proposed to purify the composite pollutants and improve the universality of such composite pollutants. The results were validated by optimizing the solid-phase extraction method for pollutant detection and showed that the recovery rate of composite contaminants in pharmaceutical and personal care products (PPCPs) increased first as the flow rate increased with 37.5-100% at 4 mL/min to 40-110% at 5 mL/min, and then, dropped back to 20-95% at 8 mL/min. According to the analysis of PPCPs' concentration in the eluent, the optimal eluent concentration had the best elution effect at 10 mL. The filtration membrane had high purification efficiency for composite wastewater. Compared to traditional sewage treatment schemes, this method could achieve a performance improvement of about 60%. It could basically control the concentration of various pollutants below 80 ng/L. The results indicated that the filter membrane had good durability and reusability, which could make significant contributions to environmental protection and sustainable development.

Keywords: nanofilm; sewage treatment; micro/nano plastic; PPCPs; solid phase extraction.

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Introduction

Pharmaceutical and personal care products (PPCPs), as a new type of pollutant, are widely present in various water bodies [1]. Their presence has had a serious impact on the environment and ecosystems, especially when micro/nano plastics combine to form a more toxic composite pollutant, which can cause serious damage to the genes and reproductive capabilities of organisms [2]. Dealing with the sewage treatment problem of PPCPs has become an important issue that needs to be solved urgently. Traditional sewage treatment solutions show good treatment performance when targeting one or several PPCPs pollutants [3]. However, due to the wide variety of PPCPs, when micro/nano plastics are combined into composite pollutants, not only the toxicity is enhanced, but also the removal difficulty is greatly increased. Therefore, for the treatment of wastewater from pharmaceutical companies, it is necessary to find an efficient and feasible method to solve this problem. As an emerging separation technology, nanofiltration membrane has the advantages of small pore size, strong selectivity, and high flux. It is widely used in the field of wastewater treatment and has great application potential [4, 5]. Through the size limitation and surface charge of nanopores, trace organic matter, heavy metal ions, microorganisms, and other pollutants in wastewater can be effectively removed, thereby improving the purification effect of wastewater. As an emerging filtration technology, nanomembranes have attracted the attention of many researchers. Zhang *et al*. used a thiol-disulfide exchange reaction to synthesize macroscopic 2D ultrathin protein membranes when developing robust 2D biological ultrathin membranes with adjustable structure and function. The results showed that this material adhered to various substrates and released a series of molecules without significantly affecting its activity [6]. Yu *et al*. applied ultra-sensitive side flow analysis (LFIA) based on multi-layer fluorescent nanofilm (GO/DQD) guided signal amplification to identify foodborne bacteria quickly and sensitively in complex samples. The results showed excellent stability and accuracy, and greatly improved sensitivity [7]. Xu *et al*. used a surface plasmon coupled emission method when studying a universal unlabeled fluorescent nanofilm sensor and found that unlabeled fluorescent sensors could be easily prepared through spin coating technology, which could be widely applied in *in situ* research [8]. Wang *et al*. employed a multiple lateral flow assay (LFA) when studying the side flow immunoassay biosensor. The results indicated that the proposed detection method had great potential in early diagnosis of respiratory virus infections [9]. Lan *et al*. used Mayadas and Shatzkes (MS) theories to explain the physical properties of polycrystalline gold nanofilms with thicknesses between 40.5 nm and 115.8 nm when studying their conductivity and thermal conductivity. The results indicated that grain boundary and substrate scattering could affect electron and phonon transport in polycrystalline metal systems [10].

Wastewater treatment, as one of the key issues in environmental protection, has received extensive attention on the treatment methods. Li *et al*. investigated whether the coastal wetlands in the Yellow River Delta became secondary pollution sources. The surface mount technology (SMT) and diffusive gradients in thin films (DGT) were combined to study the vertical distribution of different forms of phosphorus in sediment. The reception and purification capabilities of wetlands in the region were elaborated and the results showed that wetland sediments in this area had strong phosphorus retention ability, which could control the migration and transformation of endogenous phosphorus. Therefore, wetlands were not secondary pollution sources [11]. Xiao *et al*. used a multifunctional water treatment device when studying the treatment for water-soluble pollutants and surface oil slicks. It could generate clean water vapor after sewage treatment under sunlight. Bismuth oxybromide catalyst degraded water-soluble pollutants under light irradiation. The results demonstrated that SH sponge remained stable and easy to operate during wastewater treatment process [12]. Natrayan *et al*. used the sunlight purification method to purify wastewater when studying sewage treatment methods. According to the results, it had a better ability to prevent and treat coronavirus in sewage treatment [13]. Mukhortova *et al*. investigated the potential application of activated carbon in the treatment of aromatic nitrophenols and nitrite sulfonic acid wastewater. The main parameter of the adsorption process that provided maximum purification, the amount of activated carbon, and the pH of the mother liquor were determined. The research concluded a regeneration method for extracting activated carbon and determined the optimal extraction conditions [14]. Song *et al*. investigated the purification effect of different aquatic plants on the tail water of sewage treatment plants under low temperature conditions. By monitoring the pH value, the chemical oxygen demand (COD), NH_4^+- N, total nitrogen (TN), total phosphorus (TP), and plant growth conditions, the survival conditions of aquatic plants were obtained [15].

With the development of technology, the types of pollutants in industrial wastewater are becoming increasingly complex. Plastic pollution, as a common pollutant, widely occurs in various water bodies. They have a large number and variety. The physical and chemical properties are also different from the main water pollutants. Micro/nano plastics in the environment can be roughly divided into two categories, namely primary plastics, and secondary plastics [16, 17]. Primitive plastics mainly refer to plastics used for certain types of applications. Secondary plastics refer to plastic debris formed by the degradation of large plastics in the natural environment through a series of physical, chemical, and biological reactions. Among them, new plastics mainly include cleaning agents and detergents containing plastic microspheres, medical supplies, polishing materials used in industrial production and processing and accidental leakage of raw materials for plastic product production. The other type is secondary plastic, which mainly includes wear and tear of car tires, damage and cracking of agricultural plastic film, wear and release of synthetic fiber textiles, damage to daily discarded plastic supplies, and wear and tear of other plastic products. Pharmaceutical companies are one of the main sources of plastic pollution. There are many types of plastic pollutants in the industrial wastewater. The pharmaceutical industry produces a significant number of organic pollutants. The mixture of two pollutants has led to more serious environmental pollution and ecological damage. The destructive effects of a single micro/nano plastic on ecology can be mainly divided into two categories including physical and chemical damages. Physical damage mainly refers to the damage caused by plastic particles to biological health through entanglement with organisms or blockage of the esophagus. Chemical damage refers to the chemical toxins from plastics and their additives during sedimentation, causing damage to biological genes and cells [18]. In plastic production, to cope with different usage purposes, various types of plastic products often add different chemical reagents to improve the ductility, high temperature resistance, and other properties of plastics. Therefore, in different solution environments, the toxins released may vary due to differences in liquid pH, temperature, and lighting conditions. Some additives also have differences in their hydrophilic and hydrophobic properties [19]. The types and distribution of PPCPs pollutants, which are very common in the wastewater discharged by pharmaceutical enterprises, are statistically analyzed [20]. Although PPCPs have a fast degradation rate, the concentration in water remains high due to their large usage. The combination of such pollutants with plastic particles increases their toxicity, posing a greater threat to ecology and human health. This is due to the small size of plastic particles and their unique physical properties, which make them more likely to become carriers of toxic pollutants in wastewater. The composite pollution of micro/nano plastics and organic pollutants is mainly concentrated in pollutants of heavy metals, polycyclic aromatic hydrocarbons, and PPCPs, which has stronger toxicity and becomes more difficult to metabolize, making it easier to accumulate inside of the body causing serious problems to endocrine system, energy metabolism, molecular level, and cellular genetic information of organisms. Therefore, in the wastewater purification of pharmaceutical enterprises, not only do the plastic components need to be treated, but also the complex organic pollutants need to be considered.

From past studies, it has been found that sewage treatment has high research value and attracts many researchers in this field. However, few studies adopted the method of nanofiltration membranes to solve the problem of wastewater treatment. This study applied nanomembranes as the purification method based on the molecular weight of PPCPs composite pollutants and adopted optimized solid-phase extraction technology to detect changes in pollutant concentration, enhance the accuracy of data, and improve the efficiency of pharmaceutical wastewater purification work.

Table 1. Types and concentrations of PPCPs in simulated pharmaceutical wastewater solution.

Drug	Molecular formula	Molecular weight	LogP	PKa
Mycophenolic acid (MPA)	$C_{17}H_{2}0O_{6}$	320.34	2.62	4.47
Gliclazide (GLI)	$C_{12}H_{21}N_{3}O_{3}S$	323.41	3.24	3.5
Sulfamethoxazole (SMX)	$C_{10}H_{11}N_{3}O_{3}S$	253.27	0.91	4.3
Propafenone (PRM)	$C_{12}H_{14}N_{2}O_{2}$	218.25	0.93	12.9
Carbamazepine (CBZ)	$C_{15}H_{12}N_{2}O$	236.27	2.51	13.7
Methylbenzamide (DEET)	$C_{12}H_{17}NO$	191.27	2.56	-0.94
Ciprofloxacin (CIP)	$C_{17}H_{18}FN_{3}O_{3}$	331.34	2.01	6.15
Trimethoprim (TMP)	$C_{14}H_{18}N_{4}O_{3}$	290.32	0.93	7.22
Diclofenac (DIA)	$C_{14}H_{11}C_{l2}NO_{2}$	296.00	4.53	4.01
Amoxicillin (AMX)	$C_{16}H_{19}N_{3}O_{5}S{\cdot}3H_{2}O$	419.46	0.91	7.31
Atenolol (ATL)	$C_{14}H_{22}N_{2}O_{3}$	226.33	0.37	9.51

Materials and methods

Simulation of pharmaceutical wastewater composition

The traditional purification approach can only target a few types of PPCPs compounds with limited purification effects. PPCPs, as an emerging organic micro pollutant, have been detected with high frequency in surface water, groundwater, reservoirs, sewage plants, and oceans. As a major producer of PPCPs in China, 70% of prescription drugs are antibiotics, which makes the content of PPCPs compounds in the environment maintain in a relatively dangerous range. To simulate the composition of wastewater from actual pharmaceutical factories, 11 types of PPCPs were mixed and configured. The drug concentration of the mixture was above 98%. The specific components of the simulation solution were shown in Table 1. To prepare the organic mixture, 10 mg of various PPCPs standards were dissolved in methanol to prepare the stock solutions of 100 mg/L of each before mixing them together to get a mixture of PPCPs. The mixture was then mixed with plastic particles to roughly simulate the wastewater nature discharged by pharmaceutical companies.

The traditional sewage removal process of PPCPs relies more on advanced oxidation technology and physical means of activated carbon adsorption. However, oxidation technology has poor removal efficiency for non-nitrogen organic pollutants. The purification cost of activated carbon is also too high. This study applied a PIP nanofiltration membrane (PIP-NF) a nanofiltration membrane that has a better treatment effect on organic matter with a relative molecular weight between 150 and 1,000 to purify pharmaceutical factory wastewater with the aim of improving the purification and treatment efficiency. The filtration efficiency of nanofiltration membranes is influenced by factors including the physical properties of the membrane, solute properties, solution properties, and operating environment. Graphene oxide (GO) nanosheets were used to improve the polyamide (PA) membrane, enhancing the dialysis performance, and hydrophilicity.

Composite situation and concentration calculation method of microplastics and PPCPs in pharmaceutical wastewater

The adsorption capacity of the adsorbent material to the target pollutant was calculated below.

$$Q_t = \frac{\left(C_0 - C_t\right)\times V}{m} \tag{1}$$

where Q_t was the adsorption capacity at a specific time. C_0 was the initial concentration of the pollutant. Studying adsorption kinetics is beneficial for studying the adsorption mechanism in sewage purification. The pseudo first level dynamic model was shown in equation (2).

$$\begin{cases} \ln(Q_e - Q_t) = \ln Q_e - K_1 t \\ Q_t = Q_e(1 - \exp(-K_1 t)) \end{cases} \quad (2)$$

where Q_e was the equilibrium adsorption capacity of the adsorbent for the target pollutant at equilibrium. K_1 was the rate constant of the pseudo first-order kinetic model. After constructing the first level dynamic model, a second level dynamic model was built as follows.

$$\frac{t}{Q_t} = \frac{1}{K_2 \times Q_e^2} + \frac{t}{Q_e} \quad (3)$$

where K_2 was the rate constant of the second-order kinetic model. t was the adsorption time. Subsequently, the particle diffusion model in the solution was analyzed to simulate the diffusion of pollutants at the micro/nano level. The specific simulation was shown in equation (4).

$$Q_t = K_{id} \times t^{0.5} + C \quad (4)$$

where C was a constant for the number of thickness boundary layers. K_{id} was the particle diffusion constant, which reflected the diffusion ability of the particle. A large number indicated strong diffusion ability. In addition, temperature could also affect the activity of particles. An isothermal adsorption model for molecular motion was then constructed as below.

$$\frac{C_e}{Q_e} = \frac{1}{K_L \times Q_m} + \frac{C_e}{Q_m} \quad (5)$$

where Q_m was the theoretical saturated adsorption capacity of the adsorbent for pollutants. K_L was the adsorption constant of the model. By simulating the coarse filtration of molecular motion, the single molecular layer adsorption could be analyzed. To improve the model, the adsorption heat of each point should be expressed as:

$$Q_e = K_F \times C_e^{\frac{1}{n}} \quad (6)$$

where C_e was the pollutant concentration in the solution during adsorption equilibrium. K_L was the adsorption constant, which was related to the properties of the adsorbent itself, the amount used, and the environmental temperature during the reaction. The essence of adsorption is the adsorbate filling the adsorbent channel. The filling model could be represented as:

$$\begin{cases} \ln Q_e = \ln Q_m - \beta\varepsilon^2 \\ \varepsilon = RT\ln(1 + \frac{1}{C_e}) \\ E = \frac{1}{(2\beta)^{0.5}} \end{cases} \quad (7)$$

where β was a constant related to free energy. ε was the adsorption equilibrium constant. After obtaining the adsorption model, the pollutant concentration change curve was introduced with the expression of equation (8).

$$Q_e = K_p \times C_e \quad (8)$$

where K_p was the adsorption distribution coefficient. The model construction of the entire pollutant adsorption process was then completed. This method could be used to simulate the concentration changes during the filtration membrane purification. The size and morphology of micro/nano plastics were also analyzed. The plastic particles were simulated with the specific model represented below.

$$\begin{cases} D_W = \sum_{i=1}^{k} n_i D_i^4 / \sum_{i=1}^{k} n_i D_i^3 \\ D_N = \sum_{i=1}^{k} n_i D_i / \sum_{i=1}^{k} n_i \\ U = D_W / D_N \end{cases} \quad (9)$$

where N was the number of micro/nano particles. D_W was the average diameter of weight.

Table 2. Additive categories in micro/nano plastics.

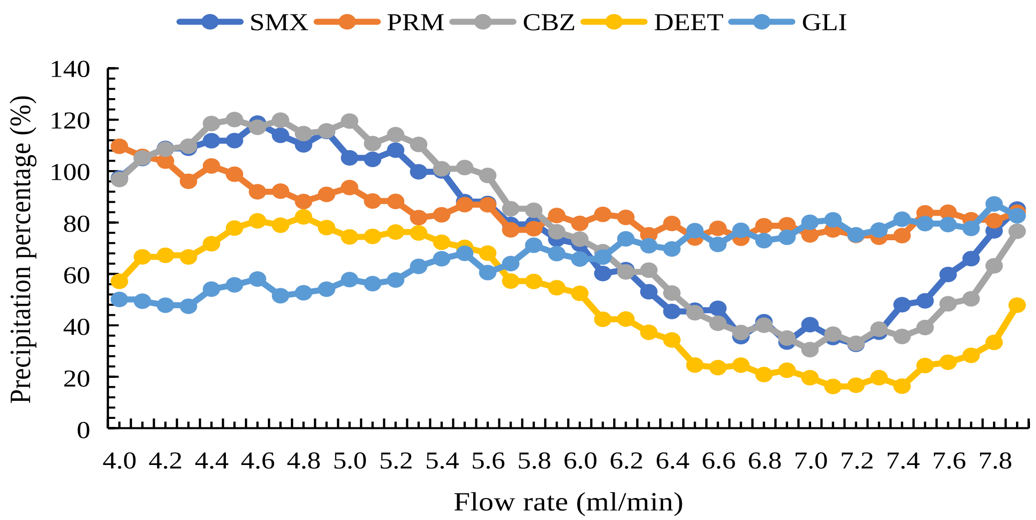
Chemical substances	Concentration (μg/g)	Polymer	Influence factor
Diisobutyl phthalate,	0.07	PE bag	Chemical additives
Di-n-butyl phthalate	0.13		
Hexabromodiphenylpropylamine	4,300	Expanded PS	Water parameters, temperature, UV irradiation
Dimethyl phthalate	0.01	PVC cable	Sunlight, bacterial exposure
Diethyl phthalate	0.08		
Pb	120	PVC pipe	PH value and temperature of water, Total dissolved solids, UV irradiation
Sn	85		
Ba	5		
Ca	1,650		
Cd	10		
Phthalate esters	8. 9	Packaging plastics	Polymer type, temperature
Alkyl phenol	15		
Bisphenol A,	0.55		
Di-2-ethylhexyl adipate	1.5		
Nonylphenol	0.12	PVC	Type and temperature of leaching solvent
	0.24	HDPE bottle	

D_N was the average diameter of the number.

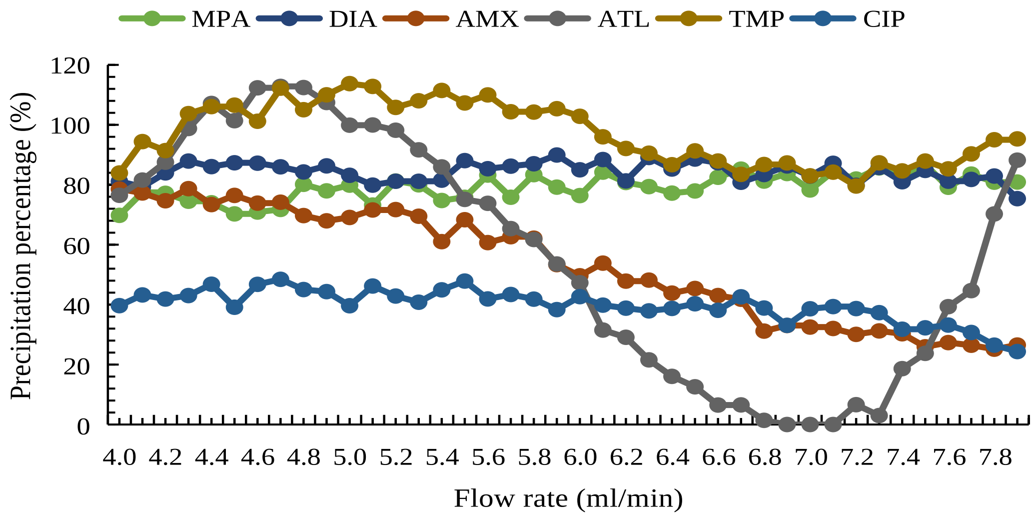
Results and discussion

The removal effect of nanomembrane on pollutants

To improve the purification efficiency of wastewater containing plastic particles, the main types of plastic additives in pharmaceutical enterprise wastewater were analyzed (Table 2). Additives in plastics can interfere with the nervous system of organisms, affect their reproductive ability, and induce genetic abnormalities. In addition, micro/nano plastics have impacts on human endocrine and reproductive development functions. Pharmaceutical companies, as one of the sources of nascent plastics, also contain a large amount of nascent plastics in their industrial wastewater. To verify the pollutant removal performance of the nanofilm, the pollutant measurement method in the solution was first optimized. To ensure the accuracy of experimental data and enhance the persuasiveness of the experiment, solid-phase extraction was chosen to test the concentration of composite pollutants in the liquid. To improve the extraction performance, the relevant parameters for the mobile phase during the extraction process was optimized to enhance the extraction effect. The hydrophilicity and hydrophobicity of PPCPs composite pollutants varied. To determine the mobile phase that should be used for the nanomembrane filtration, the solubility of different composite pollutants was analyzed. The separation efficiency of methanol/water and acetonitrile/water as flow relative to pollutants was compared. The results showed that, when acetonitrile/water was used as the flow term, the target substance showed a premature peak. The separation effect of various pollutants was poor. The chromatographic band was greatly disturbed due to the high-water phase ratio and high baseline. Therefore, methanol/water was chosen as the circulating phase. Compared to acetonitrile, methanol has lower cost and toxicity, making it more suitable for the separation of composite pollutants. The peak time of different composite pollutants varied (Table 3). Each composite pollutant was basically distinguished on the chromatographic band. The separation effect of only three composite pollutants (AMX, ATL, and TMP) was poor.



(a) Changes in precipitation rate of the first five groups



(b) Changes in precipitation rate of the last six groups

Figure 1. The efficiency of flow rate on the precipitation of composite pollutants.

However, due to the significant difference in separation times of 4.93, 8.27, and 14.37 min, the three pollutants could still be successfully separated. In solid-phase extraction, flow rate was one of the most important influencing factors in the pollutant separation step. At different flow rates, the separation ratio of pollutants also changed accordingly. By adjusting the flow rate during the purification process, different composite pollutants could be distinguished. The recovery ratios of hydrophilic-lipophilic balance (HLB) solid-phase extraction columns for different composite pollutants were determined at different flow rates (Figure 1). The results showed that, as the flow rate increased, the recovery rate of PPCPs composite pollutants first increased from 37.5 - 100% at 4 mL/min to 40 - 110% at 5 mL/min, and then decreased to 20 - 95% at 8 mL/min, which might be the reason that the rapid flow rate led to a decrease in the contact effect between the water sample and the micro/nanofiltration membrane, resulting in a decrease in filtration efficiency. Elution treatment was then carried out on the recovered pollutants using methanol with high recovery rate and relatively stable properties. The eluent

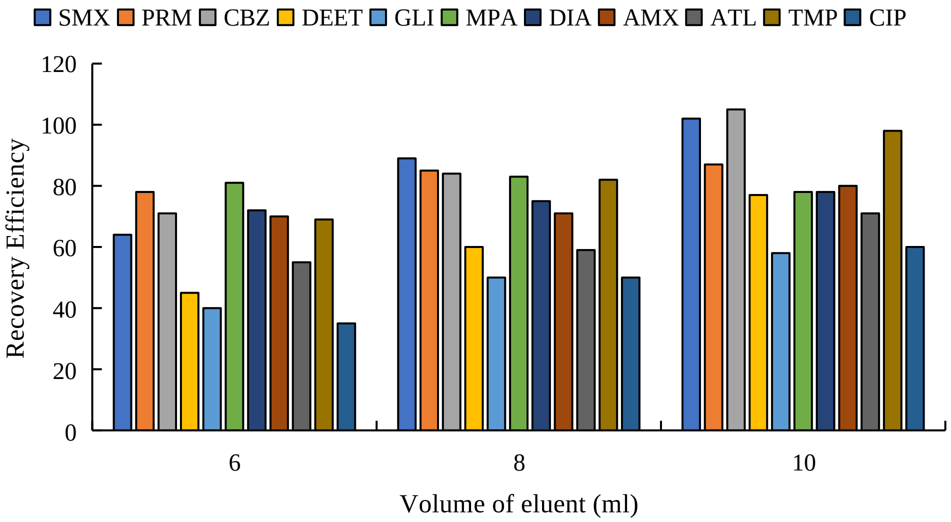


Figure 2. Elution efficiency of different volumes of eluent.

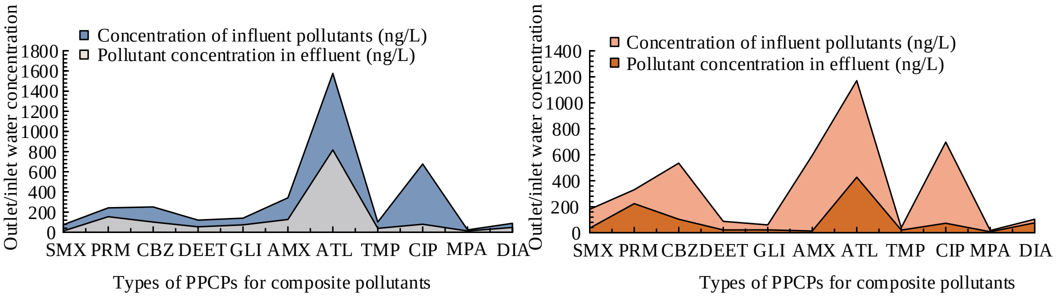
volume affected the final pollutant treatment efficiency. Therefore, 6, 8, and 10 mL of methanol were selected as eluents to calculate the specific recovery efficiency of composite pollutants. The optimal eluent concentration was 10 mL. The overall recovery efficiency of the eluent showed an upward trend with the more volume of eluent, the better the recovery efficiency (Figure 2).

Table 3. Chromatographic separation of composite pollutants in methanol/water mobile phase.

Composite pollutants	Wavelength (nm)	Time (min)
SMX	270	4.21
PRM	215	6.50
CBZ	277	15.61
DEET	215	17.80
GLI	229	19.40
MPA	254	22.54
DIA	254	24.10
AMX	235	4.93
ATL	235	8.27
TMP	235	14.37
CIP	285	17.10

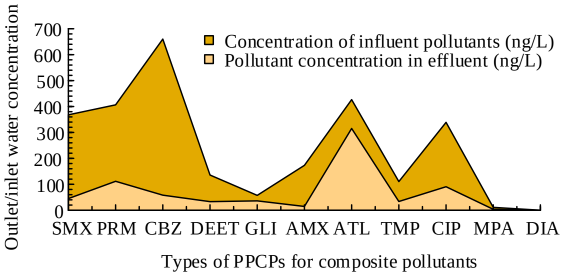
The efficiency of nano membrane technology for composite wastewater purification

Based on the concentration changes of composite pollutants in inlet and outlet water, different traditional methods for rough treatment of pollutants were analyzed. The specific changes of the pollutant concentration in the effluent and inflow were shown in Figure 3. The results showed that the purification performance of traditional composite pollutants fluctuated greatly. The purification effects of composite pollutants formed by PPCPs such as CBZ and CIP were poor with the purification performance less than 10%. Although the ATL showed the highest purification efficiency, there were also significant fluctuations in the purification efficiency of different methods with the maximum purification efficiency also less than 70%. Therefore, in the general sewage purification of pharmaceutical enterprises, advanced treatment should be carried out on the sewage after secondary treatment. The pollutant removal rates of the advanced treatment method and the secondary treatment method were shown in Figure 4. The purification effect of advanced treatment was generally better than the pollutant purification rate of the secondary treatment scheme. The removal rate of a certain PPCPs composite pollutant could reach 99.99% while the overall removal rate remained around 80%. The overall purification performance showed good removal performance for specific pollutants and the overall removal rate was still low. To verify the superiority of nano membrane



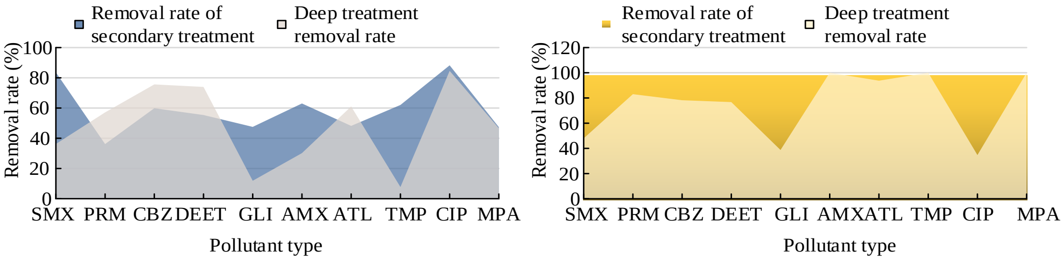
(a) Anaerobic tank&DE oxidation ditch

(b) Anaerobic tank&Carrousel oxidation ditch



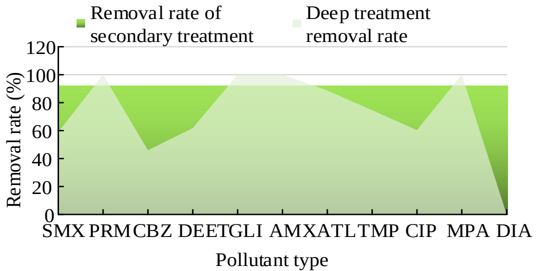
(c) Anaerobic tank&three ditch oxidation ditch

Figure 3. Changes in effluent and inflow of three traditional composite sewage treatment methods.



(a) Denitrifying biological filter+nitrifying biological filter+activated sand filter

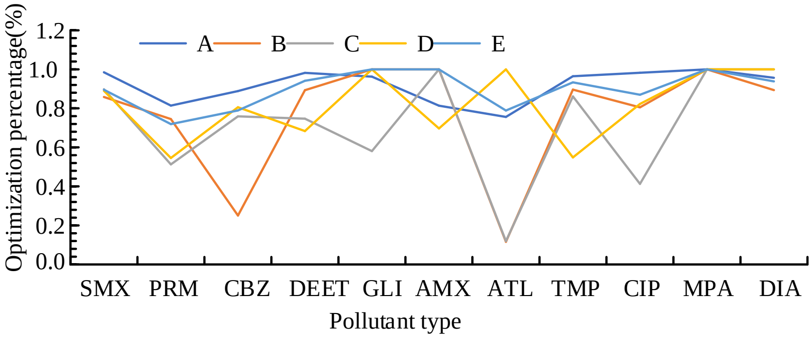
(b) Aerated biological filter+denitrification biological filter+activated sand filter



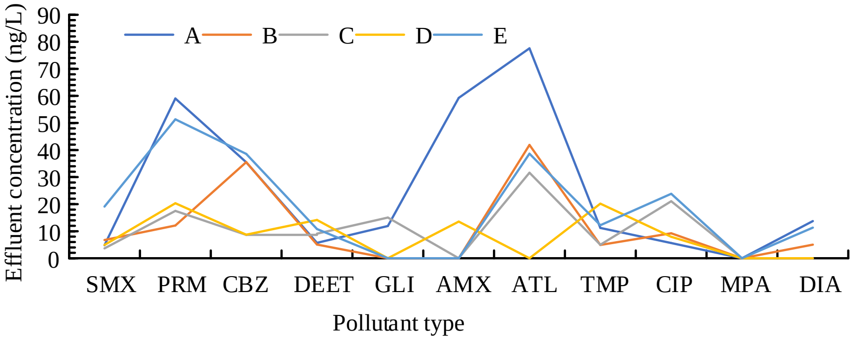
(c) Ecological wetland

Figure 4. Comparison of removal rates between secondary treatment schemes and deep treatment.

pollutant purification technology, a comparison was made between the nano membrane purification method and traditional deep treatment methods. The results showed that the filter membrane had a higher efficiency in purifying composite wastewater. Compared to traditional deep sewage treatment schemes, the filter membrane method could achieve a



(a) Optimization effect of effluent standards compared to various pharmaceutical enterprises



(b) Pollutant effluent concentration of A-E five pharmaceutical companies

Figure 5. Performance comparison between nanofiltration method and other advanced treatment methods.

performance improvement of about 60%. In the determination of composite pollutants in effluent, compared to the traditional method with a pollutant concentration of 100 ng/L, the membrane method basically controlled the concentration of various pollutants below 80 ng/L. Compared to traditional methods, the nanofiltration had great advantages (Figure 5).

Conclusion

Nowadays, with the acceleration of industrialization and urbanization, water pollution is becoming increasingly prominent, posing a serious threat to the environment and human health. Finding efficient, economical, and environmental water treatment technologies has become an urgent issue, especially in industrial and urban wastewater treatment. This study explored the application effect of nano membrane technology in wastewater treatment by improving the treatment effect of the technology, which was expected to provide a comprehensive solution for water treatment and water quality monitoring, contributing to the sustainability of water resources and environmental protection. Traditional pollutant treatment methods have poor universality for complex types of PPCPs. The combined pollutant purification ability of some PPCPs and plastic particles is also poor. Therefore, this study proposed a nano membrane purification technology for combined pollutant purification. Compared to the average removal rate of about 80% in traditional deep treatment purification method, the filter membrane method could basically achieve a performance improvement of about 60%. The various composite pollutants were controlled below 80 ng/L, demonstrating excellent purification performance of composite pollutants. The removal rate of a certain PPCPs composite pollutant approached 99.99%. The overall removal rate remained around 80%. When the volume of eluent was 10 mL, the optimal elution effect was achieved. However, the economic cost of membrane purification technology is high, which may prevent it for the secondary treatment stage of pharmaceutical factory sewage purification. Therefore, deep sewage treatment may serve as a direction for future method improvement.

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