Facile Fabrication of Positively Charged Nanofiltration Membrane for the Effective Separation of Cationic Dyes and Salt Mixtures

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Abstract-Textile wastewater often contains a significant number of cationic dyes, posing substantial environmental risks. To address this challenge, a simple fabrication procedure for synthesizing a positively charged membrane with high permeance and selectivity is needed to make this realistic. In this study, we developed a stable positively charged NF membrane by initially reacting P84 with Polyethyleneimine (PEI) and then further treating it with Trimesoyl Chloride (TMC). This involved casting a homogeneous P84 solution onto a non-woven support and immersing it into a PEI solution to create a P84/PEI membrane. The crosslinking reaction between PEI and the imide ring of P84 led to the formation of a stable P84/PEI structure with a hydrophilic surface and a positive charge. Subsequently, TMC was used in the IP process to create dense separation layer. The optimal membrane, P84/PEI_{0.6}/TMC_{0.05}, exhibited an excellent water flux of 26.2 L m⁻² h⁻¹ bar⁻¹ and high rejection for selected cationic dyes, including Rhodamine B (RhB), Janus Green B (JGB), Crystal Violet (CV), and Methylene Blue (MB), with rejection of 96.2%, 99.8%, 99.8%, and 94.7%, respectively. This facile fabrication process could accelerate the development of positively charged NF membranes to remove cationic dyes from industrial wastewater.

Keywords—positively charged nanofiltration membrane, dye/salt separation, textile treatment, crosslinking reaction

I. INTRODUCTION

Textile wastewater contains some cationic dyes, which present severe environmental hazards. Due to the significant advantages of Nanofiltration (NF) membranes, researchers pay more attention to using NF membranes for textile wastewater treatment [1, 2]. Many commercially available NF membranes are negatively charged due to the residue of the unreacted carboxylic acid groups during the Interfacial Polymerization (IP) process [3]. However, negatively charged NF membranes tend to have low rejection for cationic dyes due to strong electrostatic attraction. Positive dyes are attracted to the negatively charged membrane surface, forming a cake layer, and causing severe membrane fouling [4]. Hence, there is an urgent demand for the development of positively charged NF membranes with enhanced cation rejection efficiency.

Recently, more research has focused on applying cross-linked polyimide membranes with PEI for NF applications [5, 6]. The PEI offers great potential to create a positively charged membrane by introducing additional amine groups. This positively charged layer demonstrates notable effectiveness in the rejection of cations. For example, Feng *et al.* [7] employed PEI and TMC on a polyethersulfone

substrate to fabricate NF membranes using the IP process. The membranes with layer-by-layer structures were created by undergoing repeated cycles of sequential reactant deposition and reactions, leading to improved salt rejection. However, most reported operational procedures are complex. PEI is highly soluble in water, leading to potential concerns of weak binding between PEI and the substrate, thereby reducing the membrane stability.

In this chapter, a stable NF membrane with a positive charge was developed through a straightforward fabrication process. Initially, the homogeneous P84 solution was cast onto the non-woven support and then immersed in the PEI solution to create the P84/PEI membrane via phase inversion and cross-linking reaction process. Secondly, TMC reacted with the P84/PEI substrate via the IP process to generate a dense separation layer (P84/PEI/TMC). Utilizing these two simple steps, P84/PEI/TMC membranes were constructed by a covalent bond, achieving the formation of a stable structure. Furthermore, the membrane's chemical composition, cross-sectional morphology, and surface characteristics were analyzed. The separation efficiency of membranes in cationic dyes/salts mixtures was assessed. This innovative membrane exhibits significant potential for the recovery of valuable cationic molecules.

II. MATERIALS AND METHODS

A. Materials and Chemicals

The P84 polymer powders, characterized by a molecular weight of 153,000 g/mol, were sourced from HP Polymer GmbH in Austria. PEI with an average molecular weight of around 25,000 was obtained from Acros Organics. TMC (98%), n-hexane (95%), 1,4-Dionane (99.5%), *N*-dimethylformamide (DMF, 99.8%), Polyethylene glycol (PEG), and sodium sulfate (Na₂SO₄, 99%) were supplied by Acros Organics in Belgium. RhB (95%), JGB (65%), MB (95%), and CV (90%) were provided by Sigma-Aldrich, Belgium. Deionized (DI) water was used in all aqueous solutions.

B. Preparation of P84/PEI/TMC Membranes

The P84 casting solution was prepared by dissolving 22 wt% P84 polymer powder in a mixed solution of 1,4-dioxane and DMF, with a mass ratio of 1:4. Following degasification, the homogeneous solution was applied onto the non-woven support using a casting knife with a thickness of 200 μ m. Subsequently, it was immersed in varying concentrations of

PEI solution (0.1, 0.3, 0.6, and 0.9 wt%) for 1 hour to create the P84/PEI membrane. Various mass fractions of TMC (0.025, 0.05, and 0.1 wt%) in n-hexane were prepared and applied to the surface of the P84/PEI membrane for 30 seconds to initiate the IP process. The resulting membranes denoted as P84/PEIx/TMCy, where x represents the PEI concentration and y represents the TMC concentration. The resultant membrane were then thoroughly rinsed with DI water and stored at 4 °C in DI water.

C. Membrane Characterization

The chemical composition of the membrane surface was assessed using Fourier-transform infrared spectroscopy (FTIR, PerkinElmer Spectrum 100, Germany). For imaging the top surface and cross-sections of the samples, a field emission scanning electron microscope (FE-SEM, Hitachi S-4800) was utilized. Atomic force microscopy (AFM, Bruker MultiMode 8, Germany) was employed to characterize the surface roughness of the membranes. The zeta potential of the membrane surface was determined using an electrokinetic analyzer (SurPASSTM 2, Anton Paar) with 1.0 mmol/L KCl aqueous solutions. pH levels were adjusted using standard NaOH and HCl aqueous solutions in the range of 3.0 to 10.0. The dynamic water contact angle was measured using a goniometer (LSA200-T, Germany), with DI water used as the test liquid. Each set of samples underwent testing three times at different positions.

D. Membrane Rejection

The water permeance (*WP*, $L \cdot m^{-2} \cdot h^{-1} \cdot bar^{-1}$) was calculated as following Eq. (1) [8]:

$$WP = \frac{V}{A\,\Delta t\,\Delta P}\tag{1}$$

where V (L) is the volume of permeate collected during the interval filtration time Δt (h), A (m²) is the effective membrane area, and ΔP (bar) is the applied pressure.

The rejection R of dyes and salts was evaluated as the following Eq. (2) [9]:

$$R(\%) = \left(1 - \frac{c_p}{c_f}\right) \times 100 \tag{2}$$

where C_f and C_p represent the antibiotics/salts concentration of the feed and permeate solution, respectively.

The separation performance of the P84/PEI/TMC membranes was evaluated using a laboratory-scale crossflow setup with an effective area of 7.1 cm². The membranes were preconditioned by circulating DI water at 6 bar for 30 minutes. After achieving a stable state in water flux, the pressure was then decreased to 2 bar, and the water flux was measured. A mixture solution of a single dye type (200 ppm) and Na₂SO₄ (1000 ppm) was conducted to assess the separation efficiency of the tested membranes. Filtration was carried out for 10 minutes, after which the permeate was collected and analysed. The solute rejection (R, %) was determined.

The concentrations of the tested dyes were analyzed using a UV-1601 double-beam spectrophotometer from Shimadzu, Japan. The concentration of salts (Na₂SO₄) was determined using a conductivity meter (PW-1722, BYK LC2, Germany). In the case of mixed-salt or dye/salt bicomponent solutions, the concentrations of salt ions were determined using inductively coupled plasma-mass spectrometry, specifically the Perkin Elmer Nexion 5000 instrument.

III. RESULT AND DISCUSSION

A. Fabrication of Positively Charged P84/PEI/TMC Membrane

The chemical structure of the P84 polymer ((Fig. 1(a)), features two imide groups within each repeat unit along its backbone. To create positively charged membranes, a widely adopted method involves employing hyperbranched PEI, as shown in Fig. 1(b). The P84 substrate membrane is immersed in PEI solution, resulting in the formation of P84/PEI membrane (Fig. 1(c)). During this process, the imide rings within the P84 polymer are opened, and these open sites are hydrolyzed to carbonyl groups. These carbonyl groups then react with nucleophilic amino groups from the PEI, leading to the formation of amide bonds [5]. This reaction enables the amines in the PEI to cross-link the backbone chain of P84 polymers, forming a stable structure. In the subsequent IP step, TMC is used to react with the P84/PEI substrate, resulting in a dense P84/PEI/TMC separation layer (Fig. 1(d))



Fig. 1. Chemical structure of (a) P84 polymer, (b) PEI molecular, (c) P84/PEI membrane, and (d) P84/PEI/TMC membrane.

B. Optimization of Membrane Separation Performance



Fig. 2. Water permeance and RhB rejection of the P84/PEI/TMC membrane as a function of (a) the PEI concentration (TMC in n-hexane was fixed at 0.05 wt%) and (b) TMC concentration (PEI in DI water was fixed at 0.6 wt%), RhB of 200 ppm, 25 $^{\circ}$ C, 2.0 bar.

The separation performance of the novel developed positively charged P84/PEI/TMC membranes was assessed by evaluating their water permeance and RhB rejection. These assessments were performed on membranes prepared with varying concentrations of PEI and TMC to determine the optimal membrane fabrication parameters. As depicted in Fig. 2(a), at a PEI concentration of 0.1 wt%, the membrane displayed high water flux and low RhB rejection (~87.6%). This observation suggests a less dense separation layer with fewer positive charges due to an inadequate quantity of PEI participating in the crosslinking and IP reaction. Continuing to increase the PEI concentration resulted in a significant decrease in water permeance. For instance, elevating the PEI

concentration to 0.9 wt% resulted in an improved RhB rejection (~96.8%), but a substantial reduction in water flux (~24.2 $L \cdot m^{-2} \cdot h^{-1} \cdot bar^{-1}$). As the reaction involves more monomers, the resulting membrane becomes denser and carries a higher positive charge, resulting in decreased water permeance and improved RhB rejection. Consequently, a PEI concentration of 0.6 wt% was selected for further investigation due to its favorable characteristics, including a water permeance of approximately 26.2 L·m⁻²·h⁻¹·bar⁻¹ and a high RhB rejection of 96.2%. This specific PEI concentration was then used to examine the influence of TMC concentration on membrane performance. As illustrated in Fig.2(b), elevating the TMC concentration resulted in a decrease in water flux and a gradual rise in RhB rejection, mirroring the trend observed with increasing PEI concentrations. Upon reaching a TMC concentration of 0.05 wt%, the membrane achieved a favorable balance between water permeance and RhB rejection. Therefore, the optimal condition for the P84/PEI/TMC membranes is a PEI concentration of 0.6 wt% combined with a TMC concentration of 0.05 wt%, designated as P84/PEI_{0.6}/TMC_{0.05}. This specific membrane was selected for further investigation.

C. Physicochemical Characterization of Membranes



Fig. 3. (a) FTIR-spectra and (b) Dynamic water contact angle of the P84 $P84/PEI_{0.6}$, and $P84/PEI_{0.6}/TMC_{0.05}$ membranes.

Surface chemistry modifications in the pristine P84 membrane, P84/PEI_{0.6} membrane, and P84/PEI_{0.6}/TMC_{0.05} membrane was investigated using FTIR, with the spectroscopic findings illustrated in Fig. 3(a). Initially, the P84 membrane exhibited two prominent imide bands at 1,782 cm⁻¹ and 1,723 cm⁻¹, corresponding to the asymmetric and symmetric stretching modes of C=O (imide I), respectively. Additionally, the band at 1362 cm⁻¹ was assigned to the C-N-C stretching mode (imide II) [10]. Upon cross-linking with PEI, the imide I bands in the P84 membrane weakened, giving rise to two strong characteristic amide bands. The absorption peak at 1650 cm⁻¹ indicated the C=O stretching (amide I), while 1546 cm⁻¹ represented the C-N stretching (amide II) of the amide groups (-CONH-), confirming the successful cross-linking reaction between PEI and the P84 membrane. These amide bands were also observed in the $P84/PEI_{0.6}/TMC_{0.05}$ membrane [11]. Additionally, the P84/PEI_{0.6}/TMC_{0.05} membrane exhibited an intensified absorption band at 1,782 \mbox{cm}^{-1} attributed to the C=O stretching of carboxylic acids (-COOH). This result was indicative of the successful IP process and was associated with the hydrolysis of acyl chloride (-COCl), introducing carboxylic acid groups [12].

Dynamic contact angle measurements were employed to assess the hydrophilicity of the membrane surface. As depicted in Fig. 3(b), when cross-linked with PEI, the water contact angle decreased from 63° to 44° compared to the

pristine P84 membrane. This reduction is attributed to the increased presence of anchored PEI on the P84 substrate, leading to a significant enhancement in the hydrophilicity of the P84/PEI_{0.6} membrane surface [6]. Following the IP process, the introduction of hydrophobic TMC leads to a water contact angle of around 70° on the P84/PEI_{0.6}/TMC_{0.05} membrane, resembling the typical contact angle observed on general PA membranes. Additionally, the hydrolysis of unreacted acyl chloride groups from TMC and the presence of unreacted amine groups within the Polyamide (PA) layer maintain the membrane's hydrophilic properties.



Fig. 4. Top surface SEM images of (a) P84, (b) $P84/PEI_{0.6}$, and (c) P84/PEI_{0.6}/TMC_{0.05} membranes. Cross-sectional SEM images of (d) P84, (e) P84/PEI_{0.6}, and (f) P84/PEI_{0.6}/TMC_{0.05} membranes. AFM images of (g) P84, (h) P84/PEI_{0.6}, and (i) P84/PEI_{0.6}/TMC_{0.05} membranes.

The membrane surface morphologies analysis of the P84, P84/PEI_{0.6}, and P84/PEI_{0.6}/TMC_{0.05} membranes was conducted using SEM. The pristine P84 membrane exhibited a smooth surface, as illustrated in Fig. 4(a). In contrast, the P84/PEI_{0.6} membrane displayed scattered small particles (Fig. 4(b)), attributed to the formation of nodules through an in-situ amination reaction. This formation results from PEI molecules adsorbing and accumulating on the P84 membrane surface [6]. The P84/PEI_{0.6}/TMC_{0.05} membrane displayed a rougher surface morphology (see Fig. 4(c)), indicating the successful formation of the PA selective layer. Consistent with prior research findings, aqueous phase monomers such as PEI, which possess a higher concentration of amine groups, tend to move into the organic phase, fostering the formation of a ridge and valley structure, resulting in an uneven surface on the P84/PEI_{0.6}/TMC_{0.05} membrane [13].

The cross-sectional morphology of the P84 membrane, as depicted in Fig. 4(d), displays a distinctive asymmetrical structure characterized by a dense upper skin layer and a porous sublayer. Following the cross-linking reaction with PEI, there is a noticeable increase in the thickness of the layer covering the porous sublayer, with the dense layer thickening significantly to approximately 1.2 µm (Fig. 4(e)). This result indicates the successful cross-linking between PEI and P84 polymers during the phase inversion process. The P84/PEI_{0.6}/TMC_{0.05} membrane maintained a clearly visible thick layer, as illustrated in Fig. 4(f). Due to the typically several hundred nanometer thicknesses of the PA layer via the IP process, when combined with existing micrometer-level thicknesses, the PA layer becomes challenging to discern and still presents itself with an approximate thickness of ~1.2 μ m.

Surface roughness analysis of the membrane was conducted using AFM with a scanning area of $5 \times 5 \mu m$, as illustrated in Fig. 4(g)–(i). The pristine P84 membrane exhibited a smooth surface with a R_a value of 1.21 nm. During the phase inversion process, a uniformly PEI crosslinked P84 polymer was established, resulting in a slight increase in surface roughness to 1.44 nm for the P84/PEI_{0.6} membrane. Importantly, this well-formed polymer matrix in the P84/PEI_{0.6} substrate can further react with TMC via the IP process [3]. These factors contributed to the relatively rough surface, with a R_a value of 1.62 nm, observed on the P84/PEI_{0.6}/TMC_{0.05} membrane.

D. Separation Performance of the P84/PEI0.6/TMC0.05 Membranes



 $\begin{array}{l} \mbox{Fig. 5. (a) Rejection of Na_2SO_4 and various dyes RhB, JGB, CV and MB; (b) \\ \mbox{Zeta potentials; (c) Rejection of PEGs; (d) Pore size distribution of the } \\ \mbox{P84/PEI}_{0.6}/TMC_{0.05} \mbox{ membranes}. \end{array}$

The removal performance of the P84/PEI_{0.6}/TMC_{0.05} membrane for various dyes was investigated, as illustrated in Fig. 5(a). The rejection for RhB, JGB, CV, and MB were found to be 96.2%, 99.8%, 99.8%, and 94.7%, respectively. Additionally, the membrane exhibited low rejection for Na₂SO₄, approximately 25%, demonstrating its potential for effective dye/salt separation. The notable enhancement in dye rejection observed in the P84/PEI_{0.6}/TMC_{0.05} membrane can be primarily ascribed to the synergistic effects of electrostatic interactions and size exclusion mechanisms [14, 15]. In the context of the Donnan effect, the surface charge characteristics of P84/PEI_{0.6}/TMC_{0.05} membrane is an important factor in the removal performance of the selected pollutants. To gain a deeper understanding of this phenomenon, zeta potential measurements were performed on the P84/PEI_{0.6}/TMC_{0.05} membrane (Fig. 5(b)). Below a pH level of 9, the P84/PEI_{0.6}/TMC_{0.05} membrane exhibited a marked positive charge, while the P84 substrate had an isoelectric point at approximately pH 3.4. The substantial positive surface charge results from the protonation of amine groups and amide groups, leading to significant electrostatic repulsion with charged dye molecules and enhanced cation rejection. It is noteworthy that all four dye molecules have the same positive charge valency. However, the dye rejections by the $P84/PEI_{0.9}/TMC_{0.2}$ membrane followed the order: $JGB\approx CV> RhB > MB$. It is important to consider other contributing factors in this behaviour.

Additionally, the size sieving is also a crucial factor for consideration [16]. In the investigation of the separation mechanism, the measurements of Molecular Weight Cut-Off (MWCO) and pore size distribution of P84/PEI_{0.6}/TMC_{0.05} membrane were conducted, and the findings are depicted in Fig. 5(c) and (d). The MWCO of P84/PEI_{0.6}/TMC_{0.05} membrane was approximately 840 Da, corresponding to a pore diameter of approximately 0.91 nm. These findings also explain the relatively low rejection observed for MB, which has the lowest molecular weight (M_w) (319.9 Da). Furthermore, while the M_w of CV and JGB is lower than the MWCO of the resultant membrane, its rejection remains notably high. It can be attributed to the aggregation of dye molecules, which resul ts in enhanced effective sizes and reduced diffusivity [17]. It is noteworthy that this unusual phenomenon has also been observed by published works [18, 19]. To summarize, the positively charged surface of the P84/PEI_{0.6}/TMC_{0.05} membrane, dye aggregation, and the steric hindrance effect exerted by the dyes, all work synergistically to achieve the membrane's excellent dye rejection performance.

IV. CONCLUSION

In this chapter, the facile fabrication of positively charged NF membranes was successfully performed. Initially, covalent grafting of PEI with the P84 polymer was performed to create a more stable P84/PEI substrate. Further reaction with TMC resulted in the formation of a dense separation layer. By optimizing the membrane conditions, a high-water flux of 26.2 $L \cdot m^{-2} \cdot h^{-1} \cdot bar^{-1}$ for the positively charged P84/PEI_{0.6}/TMC_{0.05} membrane was developed. Due to its charge repulsion and size sieving effects, the membrane exhibited an excellent selectivity for cationic dyes: the rejection of RhB, JGB, CV, and MB was 96.2%, 99.8%, 99.8%, and 94.7%, respectively, with a low rejection of approximately 25% for Na₂SO₄, indicating its potential for effective dye-salt separation.

CONFLICT OF INTEREST

The authors declare no conflict of interest.

AUTHOR CONTRIBUTIONS

Yanyan Liu conducted the research, analyzed the data and wrote the paper. Songqing Hu and Bart Van der Bruggen had approved the final version.

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