

Acid-Resistant Thin-Film Composite Nanofiltration Membrane

Subjects: Engineering, Environmental

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Membrane filtration technology has attracted extensive attention in academia and industry due to its advantages of eco-friendliness related to environmental protection and high efficiency. Polyamide thin-film composite nanofiltration (PA TFC NF) membranes have been widely used due to their high separation performance. Non-acid-resistant PA TFC NF membranes face tremendous challenges in an acidic environment. Novel and relatively acid-resistant polysulfonamide-based and triazine-based TFC NF membranes have been developed, but these have a serious trade-off in terms of permeability and selectivity. Hence, how to improve acid resistance of TFC NF membranes and their separation performance in acidic environments is a pivotal issue for the design and preparation of these membranes.

Keywords: nanofiltration membrane ; acid-resistant nanofiltration membrane ; interface polymerization

1. Introduction

Membrane separation is a new separation technology that appeared in the early 20th century and developed rapidly after the 1960s [1][2]. Due to functions in separation [3], purification [4], concentration and refining [5], pivotal characteristics of high efficiency, energy saving, environmental protection, molecular level filtration, facile filtration, and easy control [6][7][8], membrane separation technology has attracted extensive attention and has been widely applied in the food industry, medicine [9], biology [10], environmental protection, the chemical industry [11], metallurgy [12], the energy industry, petroleum [13], water treatment [14], bionics [15] and other fields, contributing huge economic and social benefits and becoming the most important factor in separation science [16]. According to the pore size of the membrane, membranes used in separation can be divided into microfiltration membranes (MF), ultrafiltration membranes (UF), nanofiltration membranes (NF) and reverse osmosis membranes (RO) [16][17].

With the large-scale expansion of industrial production, many industrial processes, such as metal processing [18], mining [19][20], pulp manufacturing [21] and glass processing [22], not only consume a large amount of water (24% of water resource for the global water demand of industry by 2050) [23], but also produce a large amount of acidic wastewater [24]. For instance, it is estimated that at least 0.65 million tons of acidic waste solution are produced in the stainless-steel pickling process in China each year [25]. Acidic wastewater is a complex mixture containing various metal ions (Mn, Fe, Zn, Cu, Al, etc.) [26] and many kinds of organic compounds (such as dyes) [27], and often has high acidity (pH < 3) [28][29], which brings about tremendous pressure on sustainable and environmental development [25][30]. Current techniques for treating acidic wastewater mainly include precipitation [20][31], solvent extraction [20][31][32][33], adsorption [34][35], biological treatment [36], and membrane separation [37][38]. Precipitation is the most commonly used method for treating acidic wastewater. However, this process wastes acid resources due to the use of a large amount of alkaline reagents (such as CaO, Ca(OH)₂, NaOH, and Mg(OH)₂), and also creates a considerable amount of sludge-containing metal compounds, which are difficult to manage [31]. Solvent extraction is critical for recovering mineral acids, such as HCl and H₂SO₄, from wastewater created in hydrometallurgical processes [39]. The issue of low acid extraction and stripping, which is detrimental to recycling of the extractants, still remains [40]. Adsorption can, perhaps, remove metal ions from acidic wastewater [41], but it has an adsorption saturation and cycling limit that restricts its applicability [35][42]. Microbial treatment efficacy is limited and is susceptible to temperature and pH changes in the environment [43]. In conclusion, traditional treatment processes are unable to fully utilize acidic and metallic resources at an affordable cost; thus, new technologies should be developed.

Compared to conventional separation techniques, membrane technologies are very practicable for industrial molecular separations, including liquid–liquid separation and gas separation [34][44][45][46]. Nanofiltration (NF) membrane technology, as one of the membrane-based separation techniques, is a promising technology in the recyclability of valuable metals, the reusability of waste acid, and the reduction of sludge production [47][48][49][50], mainly attributable to the presence of

adjustable nanoscale pores in the membranes [51], and their unique surface charge that enables Donnan effects [52][53]. When the size of the electrically neutral components (neutral particles, organic small molecules) in the solution is larger than the pore size of the membrane, the neutral particles or the separation layer need to be modified to increase the probability of the neutral components passing through the nanofiltration membrane [54][55]. Moreover, due to the high deformation energy barrier [56][57] and the mass transfer resistance of the membrane pores [58][59], the nanofiltration membrane has strong retention capacity for neutral components that are larger or slightly smaller compared to the pore size of the membrane [60]. Due to the presence of ionizable functional groups in the chemical structure of the nanofiltration membrane [61], the membrane surface may be negatively or positively charged [62], which can result in electrostatic interactions between ions in the feed liquid and produce a strong repulsion effect between high valence state ions with the same charges present in high concentrations [52][63]. The resulting Donnan effect results in NF membranes having strong rejection of metal salt components [6][53][64]. With respect to their two primary retention mechanisms, nanofiltration membranes are well suited to two typical circumstances described below. When treating acidic wastewater containing metal ions, the higher valence state metal ions are retained for enrichment, while the lower valence state acidic ions and hydrogen ions are recycled by NF [65]. When treating acidic dye wastewater, the large hydrated volume of the dyes, and organic small molecules, are retained, while the acid is recycled by NF [66].

2. Challenge of NF Membranes in Acidic Environment

The degradation of the PA layer in acidic condition mainly depends on the stability of PA polymer network linked by amide bonds. The carbonyl group in amide bonds is susceptible to the delocalization of lone pairs of electrons, making the oxygen in the carbonyl group strongly nucleophilic [67].

The decline of membrane permeability during nanofiltration of acidic solution is often influenced by several factors, such as fluid viscosity and the compactness of the outer separation layer on the membrane. It is well known that the fluid viscosity and membrane permeability are inversely correlated [68], and the acidic feed liquid often showed higher viscosities than neutral fluids, leading to the lower permeate flux [69]. In addition, the hydrolyzed products, such as carboxylic acid and sulfonic acid from amide group and sulfonamide group on the separation layer, can form inter- and intramolecular hydrogen bonds with the remaining carboxyl groups, amine groups, and water on the membrane surface in an acidic aqueous solution, further impacting the separation layer structure and declining permeability of the membrane [70].

The decrease in selectivity of the NF membrane is often caused by membrane surface charge. NF membranes usually carry the negative charge in neutral and alkaline conditions and positive charge in acidic environment [71]. The type of the surface charge of the NF membrane is considered for selectivity of the membrane on the separation of different metal ions. In general, the salt rejection increases with increasing membrane surface charge, especially, and salt rejection is lowest at the isoelectric point (IEP) of the membrane. In an acidic solution, if pH is close to the IEP of the most negatively charged acid-resistant membrane, membrane surface charge will be significantly decreased while accompanied by a severe decrease in the Donnan effect [72].

3. Preparation and Regulation of Acid-Resistant NF Membrane Applied in Extreme Acid Environment

To completely solve the problem of hydrolysis of NF membranes dominated by polyamide-based materials in the extremely acidic environment, researchers have developed a series of acid-resistant membranes, such as polysulfonamide, triazine polyamine, and polyurea membranes, and so on. Such acid-resistant membranes showed high stability in strong acidic environments and could treat acidic feed liquids as well. Unfortunately, the separation performance of acid-resistant membranes was often unsatisfied [73]. The majority of them still stay in laboratory stage, and further manipulation is needed to be carried out in order to improve the separation performance.

The aforementioned PSA-based TFC NF membranes are relatively stable compared to PA-based membranes. The separation layer formed by interfacial polymerization of amine monomers with sulfonyl chloride also has good acid resistance and thereby inhibits hydrolysis and keeps a high stability due to the stable sulfonamide groups. If rigid conjugated phenyl PSA chains are integrated into the structure of the separation layer, PSA-based TFC NF membranes will become more stable in the extreme acidic environment. In addition, hydrolysis of the sulfonamide group occurs only at extremely low pH and high temperatures. However, the present acid-resistant polysulfonamide membranes generally exhibited unsatisfied separation performance. The main reason is that the organic monomers containing sulfonyl chloride group tend to have relatively higher molecular weight, lower diffusion coefficient, and reactivity than that bearing acyl chloride groups [74], which are easy to form a relatively loose separation layer with larger pore size [75]. To improve the

selectivity of the membrane, the thickness of separation layer is increased. As a result, the permeability significantly decreased, exhibiting the typical trade-off effect. For example, Eslami et al. prepared polysulfonamide membranes by interfacial polymerization of PEI, PIP, MPD, and BDSC [76]. For such a membrane, the highest flux was only $4.4 \text{ L}\cdot\text{m}^{-2}\cdot\text{h}^{-1}\cdot\text{bar}^{-1}$, while the rejection for salt solution was almost zero. This means that the separation performance of the membrane is necessary to be improved by changing the structure and composition of the membrane.

Using amine-rich macromolecular polymers, such as polyallylamine (PAH) or polyethyleneimine (PEI) as the aqueous monomers, will be useful to improve membrane permselectivity. In fact, the interfacial polymerization is regulated by the diffusion of organic phase into the aqueous phase. The monomer with higher molecular weight would lead to greater steric hindrance and lower diffusion rate, resulting in the fact that the interfacial polymerization just occurs on the side closest to the aqueous phase [77]. The reaction on the side of aqueous phase usually results in an increased membrane thickness. Therefore, it is necessary to use an appropriate concentration of aqueous phase monomer to reduce the diffusion rate of aqueous phase and improve the dispersion uniformity of aqueous phase, while the diffusion process of the organic phase is used to modify defects and reduce thickness. Moreover, the rich amine groups in the polymer chain can improve the positively charge capacity of the membrane surface and increase the selectivity of the NF membrane for metal ions. Wang et al. prepared positively charged and acid-resistant PSA TFC NF membrane by interfacial polymerization of macromolecular PAH and BDSC on the PES support layer [74]. By properly adjusting the concentration, the prepared membrane had more positive charges and a denser structure compared to the conventional PSA membranes. The water flux of the membrane was $6.82 \text{ L}\cdot\text{m}^{-2}\cdot\text{h}^{-1}\cdot\text{bar}^{-1}$, and the rejection rate was 88.3% for MgCl_2 after being soaked in 20% (wt/v) H_2SO_4 aqueous solution for 30 days.

To improve the crosslinking degree of interfacial polymerization reaction, 1,3,6-naphthalenetrisulphonyl chloride (NTSC) was used to replace BDSC. NTSC containing more sulfonyl chloride groups showed higher reactivity, which could increase the crosslinking degree of the separation layer and make it much dense. The dense interfacial layer can also reduce thickness and improve the separation performance of the acid-resistant membrane. Liu et al. synthesized the NTSC monomer by the reaction of sodium 1,3,6-naphthalene trisulfonate and thionyl chloride in N,N-dimethylformamide (DMF) [78]. The interfacial polymerization reaction of NTSC and PIP on PSf support layer afforded an acid-resistant PIP-NTSC TFC NF membrane [78]. Such a NF membrane showed a water flux of $5.8 \text{ L}\cdot\text{m}^{-2}\cdot\text{h}^{-1}\cdot\text{bar}^{-1}$, with more than 86.5% of rejection for Na_2SO_4 . Note that the separation performance did not show any significant decline after being soaked in 20% (wt/v) H_2SO_4 aqueous solution for two months or consecutively run in 4.9% (wt/v) H_2SO_4 aqueous solution for 60 days, indicating a good permselectivity and long-term stability, implying a promising application in treating acidic effluents from metal industries.

Furthermore, manipulation on interface polymerization process can also be used to improve the membrane separation performance. For example, cross-linking agents or surfactants were pre-coated on the support membrane or added into the aqueous phase [79][80]. This could not only improve the distribution of monomers on the support layer, but also regulate the diffusion process of monomers, thereby improving the separation performance [78][81]. Pre-preparing porous interlayer polymers, such as covalent organic frameworks (COF) and sulfonated poly(ether-ether-ketone) (SPEEK) on the surface of the support layer, can improve monomer distribution and provide water flow channels, thereby improving permeability and selectivity [82][83]. Adding COF or other nanoporous materials to the separation layer can increase the membrane flux and the surface charge [84][85][86], thereby significantly improving the separation performance [87]. It is also possible to use a technique, which is similar to the LBL assembly method [88] or multilayer interfacial polymerization, to reduce the separation layer defects and increase the membrane density, leading to the significantly improved permeability [89][90].

Cyanuric chloride is regarded as a good monomer with high acid stability. It reacted with amine monomer to form triazine polyamine membrane, which showed a high acid-resistant ability. The reasons are attributed to (1) the absence of carbonyl group in amine group of the separation layer structure that is prone to protonation and nucleophilic attack compared to the amide group [91] and (2) the single and double bonds of the carbon and nitrogen atoms of the triazine ring are arranged alternately to form a stable acid-resistant conjugated structure [92], which makes the separation layer have good acid resistance. However, the separation performance of the triazine polyamine-based membrane is also not well satisfied, mainly because the three chlorines from cyanuric chloride of the triazine ring have different reactivities [93]. The first chlorine has a high reactivity [94], while the substitution of the second and third chlorine in CC gradually becomes difficult. Such stage reactivity of chlorine substitution makes it less possible for diamine monomers with smaller molecules, such as PIP and MPD, to react with CC to form continuous and compact polymers with higher molecular weight. Other monomers, such as diethylene triamine (DETA), tetraethylene pentamine (TEPA), or PEI, which contain three or more amine groups, were used to react with CC via interfacial polymerization to obtain the membrane with good selectivity [95]. Note that the reaction of CC and aqueous monomer often occurs under a high concentration, high temperature, and long time, thereby usually leading to a relatively thick separation layer. Because PEI with high concentration is prone to

permeate into the pore of the support layer, this results in a serious pore blockage or reduction of pore diameter, thus reducing the permeability of membrane. The interfacial polymerization of PEI and CC on the hydrophilic PES UF membranes afforded acid-resistant polyamine membranes with the NaCl rejection of about 65% and a pure water flux of only $2.7 \text{ L}\cdot\text{m}^{-2}\cdot\text{h}^{-1}\cdot\text{bar}^{-1}$ [91]. Therefore, it is necessary to use some strategies to prevent the aqueous phase monomer from penetrating too deeply into the support layer, which include fine-tuning the concentration of reactants, regulating the reaction time, or introducing interlayer layer.

By finely regulating the concentration of the organic phase or decreasing temperature to alleviate the reaction rate, the separation performance of the polyamine membrane can be improved [96]. Yu et al. prepared a Janus acid-resistant nanofiltration membrane with opposite surface charge separation layer by using interfacial polymerization of PEI and low concentration of CC on PES UF substrate, and curing treatment was used to enhance crosslinking degree [97]. This approach, named the low concentration and curing treatment (LC-CT) method, resulted in a looser selective layer with more unreacted amine groups on the flip side of the membrane, as well as the formation of a less defective layer. The resultant TFC NF membrane showed the flux of $5.2 \text{ L}\cdot\text{m}^{-2}\cdot\text{h}^{-1}\cdot\text{bar}^{-1}$, along with 92.3% of rejection for MgCl_2 and 71.7% for Na_2SO_4 , suggesting that such a Janus NF membrane prepared via the LC-CT method can effectively improve the separation performance and acid stability compared to the commercial PA membrane NFM-1 and the commercial acid-resistant membranes NFM-1 and NFM-2.

Ionic liquids (ILs) have been used in the membrane post-treatment process to improve membrane performance [98][99]. The regulation of the interfacial diffusion process is performed mainly by utilizing the orderly directional arrangement of ILs at the water–oil interface, controlling the orderly diffusion of water-phase monomers to the interface and effectively manipulating the interfacial polymerization process. This approach can efficiently construct a highly ordered separation layer and improve the permeance of the acid-resistance membrane. Bai et al. [100] employed ILs, 1-aminopropyl-3-methylimidazolium chloride, and 1-aminopropyl-3-methylimidazolium bis((trifluoromethyl)sulfonylimide) to regulate the interfacial polymerization reaction of PEI and CC on the PSf UF support membrane for preparing a polyamine acid-resistant NF membrane with a highly ordered separation layer. Such a membrane showed a high selectivity, with 98.2% of rejection for Y^{3+} and good acid stability. Meanwhile, the permeability was significantly improved, reaching $71.2 \text{ L}\cdot\text{m}^{-2}\cdot\text{h}^{-1}\cdot\text{bar}^{-1}$.

Yu et al. prepared a dual-charge polyamine acid resistant NF membrane (ARNFM) by interfacial polymerization of PEI with low concentration CC on the surface of PES UF membrane, followed by the post-treatment of citric acid (CA) to improve the permeation performance and acid resistance [101]. Treatment of dense nanofiltration membranes with weakly acidic or alkaline reagents, such as CA, removed small oligomers and readjusted the membrane pores by reacting with surface active groups, thereby significantly improving permeation performance while maintaining selectivity [102][103]. For example, the ARNFM flux after CA treatment was enhanced 7.1 times to $2.3 \text{ L}\cdot\text{m}^{-2}\cdot\text{h}^{-1}\cdot\text{bar}^{-1}$, and the rejection was 97.6% for MgCl_2 and 90.8% for MgSO_4 [101].

Moreover, the separation performance of the membrane can also be improved by controlling the interfacial polymerization process on the support layer. For example, sodium dodecyl sulfate (SDS) adsorbed onto the surface of PES support layer can effectively change the electrostatic interaction between the aqueous phase and the support layer, further reduce the migration rate of the monomer, improve the uniformity of the distribution of the monomer, reduce the interfacial tension between the water and oil phases [104], reduce pore plugging and the defects of the separation layer defects, and thereby promote the separation performance [105]. This result also confirms that it is better to use SDS solution to activate the surface of PES support than adding it into the aqueous solution [105][106]. In addition, hydrophilic additives added to the aqueous phase can also improve the permeability by increasing the surface roughness and hydrophilicity of the separation layer [107][108].

Polyurea (PU) is an acid-resistant and mechanical strength material, which is often obtained through interfacial polymerization of amine monomers and isocyanate monomers. PU bonds with resonance structures have a higher degree of hydrogen bonding [109], which increases the energy barrier of C–N breakage in an acid environment. Hence, the urea linkages, being resistant to hydrolysis, bestow polyurea with excellent resistance to alkali and acid [110]. Polyurea membranes were first prepared as reverse osmosis membranes with a high density and poor permeability [111]. As a result, PU-based NF/UF membranes were almost neglected, especially in the field of NF separation of acid solution. Herein, a sample is showed. Zhang et al. used a series of isocyanate-based organic compound monomers, such as 1,4-phenylene diisocyanate (PDI), toluene diisocyanate (TDI), diphenylmethane diisocyanate (MDI), and hexamethylene diisocyanate (HDI), to react with PEI, PIP to form polyurea NF membranes via the interfacial polymerization on the PES support layer [112]. It indicated that PDI as the organic monomer exhibited a high reactivity. By optimizing the type and content of PDI monomer, the obtained PEI_{-0.1}/PDI_{-0.2} TFC UF membrane indicated a rejection of 97.1% for MgSO_4 and a

water flux of $0.65 \text{ L m}^{-2} \text{ h}^{-1} \cdot \text{bar}^{-1}$. Hydrogen bonds existing in urea groups in the PEI/PDI functional layer are beneficial for improving the permeation performance of the membranes. The results of static acid/alkali soaking tests corroborated that the increase in PDI content can not only enhance the degree of crosslinking, but also introduce more rigid and stable benzene rings in the PU layer, thereby promoting the stability of the TFC membrane in extreme pH conditions. Especially, after exposure to 20% (wt/v) HCl, 20% (wt/v) H_2SO_4 , and 20% (wt/v) NaOH aqueous solution for one year, the PEI_{0.4}/PDI_{0.25} membrane still preserved more than 90% of rejection for MgSO_4 , revealing excellent acid/alkali-resistant ability and high stability for treating acidic and alkaline industrial wastewater.

Additionally, the integration of nanostructured materials is also used as an effective strategy to promote the permeation flux of NF because some two-dimensional layered nanofillers with ultra-high specific surface area and unique interlamellar spacing can provide more water channels [113][114][115]. Feng et al. dispersed molybdenum disulfide (MoS_2) nanosheets into aqueous solution of polyethyleneimine (PEI) by virtue of the adhesion effect of tannic acid (TA). Additionally, the positively charged polyurea NF membranes (TSPU) were prepared by interfacial polymerization of aqueous phase PEI/TA- MoS_2 and TDI organic phase on the polyvinylidene fluoride (PVDF) UF [116]. The optimized membrane TSPU-0.015 showed a better hydrophilicity performance and a retention performance of more than 99% for high-valence heavy metal ions, such as Fe^{3+} and Cu^{2+} , and the permeation flux of TSPU-0.015 is $5.75 \text{ L} \cdot \text{m}^{-2} \cdot \text{h}^{-1} \cdot \text{bar}^{-1}$ for MgSO_4 aqueous solution, 2.88 times higher than that of undoped MoS_2 (TSPU-0), while a considerable the rejection rate of 93.18% for MgSO_4 is reached. Probably, such nanostructured material-integrated NF membranes should be further investigated in acid-stability and separation performance in acidic environments.

Hence, due to a high crosslinking degree and strong acid/alkali resistance, as well as high mechanical strength in separation layer, PU membranes need to be widely investigated and probably provide a promising candidate for the application in the separation of extreme acid or alkali wastewater industry.

In addition to polymeric nanofiltration membranes, which are typically manufactured via interfacial polymerization, another large class of nanofiltration membrane systems are ceramic membranes [117]. In terms of layer structure (with separation, transition, and support layers), ceramic membranes are similar to polymer membranes [118][119] and have similar separation mechanism (with size sieving and the Donnan effect) [120]. However, most ceramic membranes have a larger pore size and molecular weight cutoff (MWCO) [121]. Ceramic NF also showed good performance in the treatment of printing and dyeing wastewater (removal of dyes and separation of salts) [122][123][124]. Moreover, many studies have shown that ceramic NFs perform were worse than polymeric NFs in terms of salt rejection [121][125]. However, ceramic membranes offer better operational stability, high temperature stability, and cleaning stability [126]. In extreme application circumstances, ceramic membranes typically exhibit good solvent stability [127][128]. However, most ceramic membranes are limited by the substrate material ($\gamma\text{-Al}_2\text{O}_3$) and have a pH tolerance range of 2–11, showing slightly better acid resistance than PA membranes. Ceramic membrane materials with excellent acid resistance, such as TiO_2 [73] and SiOC [129], are generally more expensive [130]. Current research on the scaling up of ceramic membranes for applications is incomplete and needs to be further explored [131].

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