



New promising polymer for organic solvent nanofiltration: Oxidized poly (arylene sulfide sulfone)

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ABSTRACT

Polymeric membranes have attracted much interest in recent years for use in organic solvent nanofiltration. This article reports the development of a novel solvent resistant oxidized poly (arylene sulfide sulfone) (PASS) membrane and proposes a new strategy to enhance the solvent resistance of this PASS membrane via improving the rigidity, intermolecular interaction and packing density of polymer chains by a simple oxidation process. The PASS membranes were firstly fabricated by phase inversion, and then a facile green oxidation process was carried out to improve their solvent resistance. The oxidized PASS (O-PASS) membrane was found to exhibit an excellent stability in aggressive solvents, such as DMF and NMP, and endure 98% H₂SO₄ without sacrificing its filtration performance. In organic solvent nanofiltration (OSN), the O-PASS membranes exhibited a high rejection of dyes such as rose Bengal (RB), erythrosin B (EB), reactive orange 16 (RB16) and Sudan black B (SBB), and a stable filtration performance for 30 h in DMF. Compared to other reported OSN membranes, this membrane showed an exceptionally high solvent permeation. This extraordinary OSN filtration performance, easy fabrication process and the strong solvent resistance, make PASS a very promising polymer in the field of OSN.

1. Introduction

Solvent-resistant nanofiltration (SRNF) or organic solvent nanofiltration (OSN) is a promising pressure-driven membrane process for retaining solutes up to a size of 2 nm with a molecular weight cut-off of 200–1000 g/mol in a variety of organic solvents. Owing to its high efficiency, low energy consumption and operational stability, OSN membranes have been “tailor-made” for specific purposes, including catalyst and solvent recovery and pharmaceutical purification [1,2]. Compared to an ordinary nanofiltration membrane, OSN membranes should be chemically stable and robust to endure continuous contact with organic solvents. Therefore, the rational selection of membrane materials for OSN is of significant importance to realize its high and reproducible long-term performance.

Inorganic membranes have been considered as good candidates for OSN due to their intrinsic excellent chemical durability [3]. However, the complicated fabrication processes and high cost hindered their large-scale application. In this case, a variety of polymeric materials with superior advantages of easily scaling up and relatively low cost

have been extensively studied in the application of OSN. For example, polyimide (PI) [4], polydimethylsiloxane (PDMS) [5], polyacrylonitrile (PAN) [6], poly(ether ether ketone) (PEEK) [7] and polybenzimidazole (PBI) [8,9] have been developed to synthesize solvent stable membranes. Nevertheless, swelling and dissolution of these polymers in aggressive solvents, which inevitably results in a loss of membrane selectivity, are still a problem for practical applications. Thus, it is of great importance to explore novel high-performance polymers and advanced modification methods to improve the chemical stability of polymeric organic solvent nanofiltration membranes.

Poly(arylenesulfide sulfone) (PASS), a type of newly developed high-performance engineering plastics, is structurally modified from polyphenylene sulfide (PPS) [10]. As shown in the synthesis route and chemical structure of PASS in Fig. 1, the repeating unit of the PASS chain is composed of phenylene groups, a sulfone group and a thioether group. This unique chain structure endows PASS with a high glass transition temperature (T_g) of 218 °C, indicating a great tolerance to high temperatures. Besides, the monomer and catalyst used in the PASS synthesis are inexpensive, making it cheaper compared to traditional

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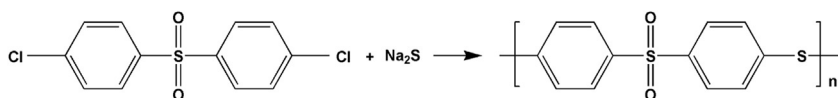


Fig. 1. Synthesis route of poly(arylenesulfide sulfone) (PASS).

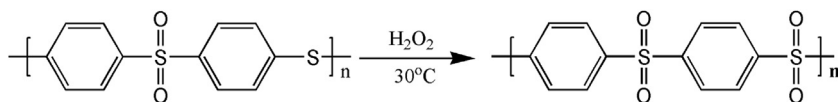


Fig. 2. Oxidation reaction of PASS.

high-performance polymers. Moreover, PASS also showed a good chemical stability in most commonly used solvents, such as n-hexane, n-heptane, ethyl acetate, methanol, ethanol, isopropyl alcohol, n-butanol, diethyl ether and toluene [11]. These attractive properties make it a promising polymer for fabricating of solvent resistant membranes. However, the solvent resistance of PASS in aggressive solvents like tetrahydrofuran, DMF and NMP is not strong enough, which would restrict its widely application in OSN. Therefore, it is of great significance to enhance the solvent resistance of PASS.

Many strategies have been implemented to improve the chemical durability of a membrane, including cross-linking, wet or dry annealing and incorporating inorganic nanoparticles [12]. However, less research has been done to enhance the membrane stability by modifying the polymer chains at the molecular level. A starting point for this is that the opposite of solvent resistance of a polymer is polymer dissolution. During polymer dissolution, the solvent first diffuses into the polymer chains; with the diffusion of the solvent into the polymer, the intrachain force is blocked by the small solvent molecule, leading to the disentanglement of polymer chains and dissolution of the polymer. Therefore, the rigidity, packing density and intermolecular interaction of polymer chains, acting as important parameters to influence the diffusion of the solvent into the polymer, are crucial to the dissolution of a polymer. A polymer with low rigidity, loose packing and weak intermolecular interaction is relatively easy for solvents to diffuse into the polymer chains, resulting in a poor solvent resistance [13]. On the contrary, the solvent resistance of a polymer would be greatly enhanced by improving the rigidity, intermolecular interaction and packing density of polymer chains. Some commonly used polymers are solvent resistant because of their special polymer chain structure. Polypropylene is widely used as nonwoven in the fabrication of organic solvent nanofiltration membranes. Its solvent resistance is derived from the highly ordered packed polymer chains in the crystalline region, which makes it difficult for the solvent to diffuse into the polymer chains. As to the intermolecular interaction, solvent resistant cellulose can be a very good example [14]. The intramolecular hydrogen bonding, from one unit to the next, creates rigid chain structures, which makes it difficult for solvents to dissolve cellulose. Much more examples can be found, including polytetrafluoroethylene, polyethylene and polyethylene terephthalate. A high intermolecular interaction, packing density and chain rigidity make a polymer difficult to dissolve; this is a general principle in polymer science [15]. However, to the best of our knowledge, these general principles have not been used intentionally in promoting the solvent resistance of polymer in the fabrication of organic solvent nanofiltration membrane.

PASS is built up at molecular level by thioether groups (-S-), sulfone (-SO₂-) and benzene rings. The C-S bond has a bond length of 180 pm and a bond angle of 90°, which is relatively large and gives more freedom for the PASS chain to move, while the -SO₂- units enable the PASS to have a more rigid chain because the polar groups would reinforce the intermolecular interaction of a polymer chains. It was reported that the -S- group can be oxidized to -SO₂- by using hydrogen peroxide (H₂O₂), concentrated nitric acid or their mixtures with other solvents [16–19]. This gives an opportunity to intentionally improve the solvent resistance of PASS by increasing the packing density and intermolecular interaction of PASS chain via the oxidation of -S- to

-SO₂-.

In this study, the strategy of improving polymer chain intermolecular interaction, packing density and chain rigidity at molecular level was intentionally employed to increase the PASS solvent resistance and to fabricate an OSN membrane via a simple oxidation process. Specifically, hydrogen peroxide (H₂O₂) was used as a green, versatile and economical oxidant to oxidize -S- to -SO₂- to modify the structure of PASS chains, and improve the solvent resistance of PASS. The chemical reaction during oxidation is shown in Fig. 2. A porous PASS membrane was first prepared via phase-inversion and then oxidized by H₂O₂ in acetic acid solution. Fourier transform infrared spectroscopy (FT-IR) analysis and X-ray photoelectron spectroscopy (XPS) were carried out to confirm the chemical structure changes during the oxidation process. The performance and stability of these pristine and oxidized PASS membranes were evaluated after three days of exposure to several solvents. Organic solvent nanofiltration was performed to investigate the performance of oxidized PASS membranes.

2. Experimental

2.1. Materials

Poly (arylene sulfide sulfone) (PASS, [η] = 0.56) was synthesized by aromatic nucleophilic substitution at atmospheric pressure [20]. N-methyl-2-pyrrolidone (NMP, 99%), dimethylformamide (DMF, 99.8%), tetrahydrofuran (THF, 99.5%), toluene (99.5%), isopropanol (99.5%), and n-heptane (99%) were purchased from Sigma-Aldrich and used as solvent. Deionized water was employed in the experiments as coagulant. Sulfuric acid (H₂SO₄, 97–98%), hydrogen peroxide (H₂O₂, 30%) and acetic acid (CH₃COOH, 99%) were obtained from Sigma-Aldrich and were used as the oxidant (in mixtures). Rose Bengal (RB, 90%), reactive orange 16 (RB16, 70%), and disperse orange 3 (DO3, 90%) were purchased from Sigma-Aldrich, and Sudan black B (SBB, high purity), erythrosin B (EB, pure), and methyl orange (EB, pure) were obtained from Acros Organics. The properties of these dyes were used as solutes and their properties are summarized in Table 1.

2.2. Preparation of membranes

2.2.1. Preparation of PASS membranes

Before the casting of the PASS solution (20 wt%, 24 wt%) on a glass plate, the solution was degassed at room temperature for 12 h. Subsequently, the glass plate with the casted polymer solution was immediately immersed in the pure water coagulation bath for phase separation. After the formation of PASS membranes, they were immersed in pure water to remove the residual NMP. The casting process was performed by using a controllable casting machine. The thickness of the casting knife was 150 μm. PASS membranes fabricated by using PASS solutions with a concentration of 20 wt% were employed to investigate the influence of oxidation on the filtration and solvent resistant performance, while PASS membranes applied in the organic solvent nanofiltration were fabricated by using the PASS solution with a concentration of 24 wt%.

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