



Novel silica-functionalized aminoisophthalic acid-based membranes for base recovery via diffusion dialysis

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ABSTRACT

The progress in diffusion dialysis (DD)-based separations provides an effective platform for the development of suitable materials for cation exchange membranes (CEM), as CEM-based DD processes aimed at alkali recovery have generated much interest due to their unique ion transport phenomena. Here, for the first time, we report the use of novel silica-functionalized aminoisophthalic acid (AIPA)-based CEMs with polyvinylalcohol (PVA) used as a binder for DD applications (base recovery). The synthesis of the active material AIPA involved a classic nucleophilic reaction between 5-aminoisophthalic acid and 3-glycidoxypropyltrimethoxysilane. By varying the amount of AIPA inside the membrane matrix, the properties of the prepared membranes can be altered. The prepared AIPA membranes were investigated systematically in terms of water uptake (W_R), ion exchange capacity (IEC) and thermomechanical measurements, such as DMA and TGA. The influence of AIPA on the base recovery behaviour of the membranes was investigated in detail. The prepared AIPA membranes displayed water uptake values (W_R) within 44.2–47.5%, ion exchange capacity (IEC) values between 0.48 and 0.93 mmol/g, initial decomposition temperatures (IDTs) of 193 to 219 °C, tensile strength (TS) values of 50.1 to 58.4 MPa and elongation at break (E_b) values of 6.6 to 224.9%. At 25 °C, the dialysis coefficient (U_{OH}) values were between 0.0068 and 0.0097 m/h, whereas the separation factors (S) ranged from 17.86 to 31.79. The membranes have great potential for base recovery via diffusion dialysis.

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1. Introduction

Rapid development in the chemical and biotechnology sector requires stable and dependable ion-exchange membranes (IEMs) for separation methods including diffusion dialysis (DD), electro-dialysis (ED), electromembrane reactors and Donnan dialysis [1–3]. During the last two decades, IEMs have drawn tremendous attention owing to their unique selectivity for specific ions [4,5]. Alternatively, IEMs also possess extraordinary capabilities for acid and base recovery from waste water [6,7]. Conventionally, ion-exchange membranes are classified into two broad classes: (i) cation-exchange membranes (CEMs) and (ii) anion-exchange membranes (AEMs), according to the type of functional groups found in the polymer backbone. The IEMs are well-known for their unique properties and are used in different industries for

numerous electrochemical applications based on separation techniques [8–11]. Different manufacturing industries, such as the paper, leather, dyeing and printing, and aluminium and tungsten ore smelting industries, produce large quantities of alkaline waste, which is considered to be a major problem from the environmental standpoint [12,13]. Therefore, alkali recovery from the waste waters is crucial for maintaining a pollution-free environment. Almost every nation in the world has already established a large number of processing plants and waste water related industries to resolve this issue [14,15]. The decisive goal behind this technique was to get rid of the polluted environment by recovering alkali from discarded solution alongside commercial aids to the industries [16,17].

To address the problem of alkaline wastes, different methods including concentration, burning and neutralization have been employed, but unfortunately their application is constrained due to massive acid or energy requirements. Ion-exchange resin-based adsorption methods have become a promising approach due to their mild and energy-efficient characteristics, but their irregular

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nature remains a major concern. Additionally, the resins require regeneration after a number of successive operations. Therefore, we must find a more efficient technique for alkaline waste treatment. The lack of reliable techniques for alkali recovery leads us to the DD process, which is recognized as the best among all other separation techniques due to its exceptional features [18,19]. Lower energy intake, low operational cost, no pollution, continuous operation and an environmentally friendly nature are some of the additional benefits of the DD process [20,21]. DD is therefore the most promising separation technique available to date. In DD, a concentration gradient is responsible for the passage of solutes through an IEM from a higher to a lower concentration region [22,23]. This process was efficiently used for base recovery from aqueous waste solutions and has been reported in numerous studies in the literature [6,13].

Experiments involving base recovery via DD with CEMs appeared to be inconsistent, as sufficient attention was not invested in the CEMs due to the lack of high performance CEM forming materials. A promising diffusion dialysis membrane must satisfy criteria, such as base resistance, base permeation and high selectivity, to demonstrate its suitability in DD-based methods [24]. Hence, the fabrication of the membranes was planned to meet the above criteria, so that these membranes could become good candidates for DD applications [18,19]. Several different polymer backbones including sulfonated poly(2,6-dimethyl-1,4-phenylene oxide) (SPPO), polyvinylidene fluoride (PVDF) and even polyvinyl alcohol (PVA) have been used for base recovery [6,13,25,26]. SPPO-based membranes showed high stability in terms of thermo-mechanical behaviour, but their low base permeation remained a challenging issue. Hence, the low permeability factor forced us to find a different polymer backbone in which the introduction of novel functionality could achieve improved outcomes over the existing polymers [27].

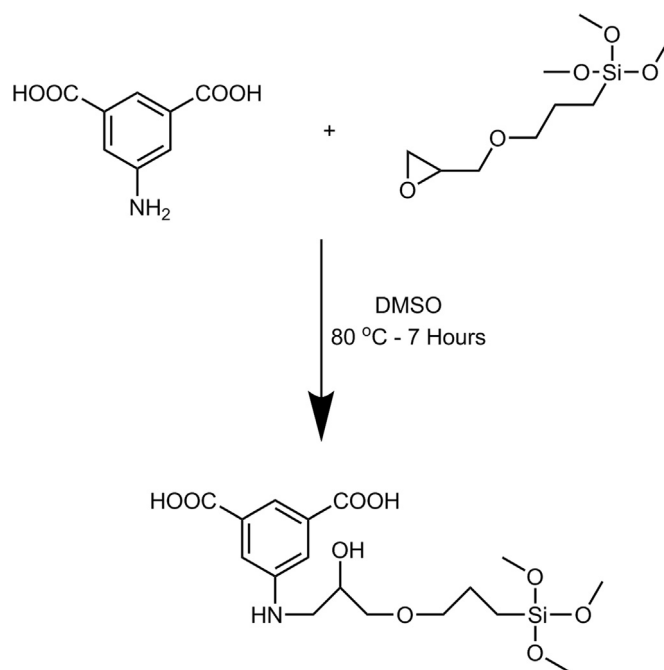
From this perspective, we focused predominantly on a more hydrophilic polymer (PVA) backbone. At the same time, the introduction of a different functionality, such as crosslinking agents inside the PVA polymer matrix, can provide better stability and results [28]. Additionally, the incorporation of different crosslinking agents can significantly reduce the high swelling of the PVA backbone [26]. Thus, PVA-based membranes proved to be a unique choice for DD applications.

With the aim of advancing the potential of CEM-based DD methods, a technique is presented in this manuscript for the fabrication of an organic-inorganic nanocomposite CEM based on PVA and AIPA (silica functionalized amino isophthalic acid). The prepared AIPA acted as a crosslinker, and sol-gel chemistry with PVA was used to develop a highly crosslinked polymer matrix. The AIPA synthesis was totally novel and is reported for the first time in the literature. The reaction proceeded smoothly under mild conditions to yield the anticipated product. The details of the membrane preparative process, e.g., the extent of AIPA on the membrane matrix and its influence on the membrane physicochemical properties, have been comprehensively studied. Moreover, a DD experiment was conducted with the developed membranes using NaOH/Na₂WO₄ as a model waste feed solution to evaluate the separation performance and base recovery.

2. Experimental

2.1. Materials

Polyvinyl alcohol (PVA, average degree of polymerization: 1750 ± 50) was purchased from Shanghai Yuanli Chemical Co. Ltd. (China). 5-aminoisophthalic acid and 3-glycidoxypropyltrimethoxysilane were purchased from Energy Chemical, China.



Scheme 1. Reaction scheme of the synthesized silica-functionalized 5-aminoisophthalic acid (AIPA).

Dimethyl sulfoxide, sodium hydroxide, hydrochloric acid, sodium tungstate dihydrate were of analytical grade and purchased from Sinopharm Chemical Reagent Co. Ltd. (Shanghai, China). All other solvents were of analytical grade and used without any further purification. Deionized water (DI water) was used throughout the experiments.

2.2. Synthesis of silica functionalized 5-aminoisophthalic acid (AIPA)

Silica-functionalized 5-aminoisophthalic acid (AIPA) was synthesized by the reaction of 5-aminoisophthalic acid and 3-glycidoxypropyltrimethoxysilane in the manner described below (Scheme 1). First, a 50-mL perfectly dried round bottom flask was loaded with an appropriate amount of 5-aminoisophthalic acid (1.0 g, 0.0055 mol). To dissolve the starting material completely, 12 g of dimethyl sulfoxide (DMSO) was slowly added to the flask while the temperature was maintained at 80 °C. The reaction mixture was then stirred for 1 hour, after which 3-glycidoxypropyltrimethoxysilane (1.31 g, 0.0055 mol) was added to the reaction mixture. Stirring was continued at 80 °C for 6 h to ensure the completion of the reaction. The product appeared as a dark brown viscous solution (14.47 wt%) in DMSO. The obtained product was characterized using NMR and FT-IR spectroscopy. ¹H NMR (DMSO-*d*₆, 400 MHz): δ 0.60 (2H), δ 1.56 (2H), δ 2.37 (1H), δ 3.17 (9H), δ 3.42 (6H), δ 3.93 (1H), δ 4.20 (1H), δ 7.35 (2H), δ 7.67 (1H) ppm.

2.3. Fabrication of the AIPA-based PVA membranes

The AIPA crosslinked hybrid PVA membranes were fabricated via a standard sol-gel process as summarized briefly in Scheme 2. First, the PVA (5 wt%) was completely dissolved in dimethylsulfoxide (DMSO) at 100 °C while a suitable amount of a DMSO solution of AIPA was also separately stirred in a glass vial for 30 minutes. Subsequently, the viscous AIPA solution was carefully added to the PVA solution and the resulting mixture was stirred overnight at 65 °C. The sol-gel process was accomplished by the hydrolysis of the silanes. Thus, the obtained gel was cast on top of

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