



# Influence of the ions distribution of anion-exchange membranes on electro dialysis



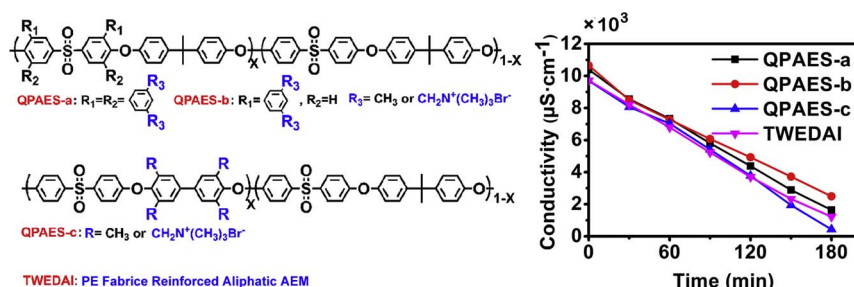
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## GRAPHICAL ABSTRACT



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## ABSTRACT

Anion exchange membranes (AEMs) are the cores of electro dialysis (ED) desalination devices, which have attracted wide attention in recent years. However, the influence of the ions distribution of AEMs on ED performances has not been well explored, which is strongly instructive to design the ED AEMs. Here we synthesized two kinds of side-chain-ionic AEMs QPAES-a and QPAES-b, and main-chain-ionic AEM QPAES-c. To uncover the relationship between the AEMs structures and properties, these AEMs were characterized with <sup>1</sup>H NMR and FTIR, and evaluated in terms of ion exchange capacity, water uptake, swelling ratio, λ, conductivity and transport number. Especially, we used NaCl aqueous solution to simulate seawater to investigate the ED performances of the synthesized AEMs by 180 min of ED processes. To distinguish our AEMs, the ED performance of commercial TWEDAI was similarly tested for comparison. The results confirmed that the ED performance with a lowest final conductivity of 430 μS·cm<sup>-1</sup> of the main-chain-ionic QPAES-c is much better than that of TWEDAI and the side-chain-ionic AEMs. The ED performance stability tests convinced that QPAES-c had ED lifetime comparable to that of TWEDAI. These results show that the main-chain-ionic AEMs have their potential as ED AEMs.

## 1. Introduction

The electro dialysis (ED) process has been proven to be a robust, efficient and versatile method for the selective removal of ionic species

from non-ionic species in aqueous media [1–8]. The base for the ED performance is the selective transport of ions of the ion-exchange membranes (IEMs) under the influence of an electrical field [9]. The advantages of ED over other separation processes includes versatility in

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term of feed streams with minimum pre-treatment requirements, low energy cost, easier and low cost of maintenance [10]. As is known to all, the requirement for fresh water has been going up owing to the increasing industrial and domestic water consumption. The availability of fresh water has become one of the most critical problems to be solved in social and economic fields [11]. Desalination technology, which offers fresh water from seawater, can effectively solve the problem of lack of freshwater resources [12,13]. Thermal desalination and reverse osmosis (RO) technology have been the commercial large-scale desalination technologies. Thermal desalination consumes substantial amounts of thermal and electric energy, which result in a large emission of greenhouse gases. The well-developed RO technology can desalinate seawater with much less energy than thermal desalination [12]. The ED process is more convenient than RO because higher brine concentration can be obtained as there are no osmotic pressure limitations [11]. However, the high cost of ion exchange membranes (IEMs) used in ED devices, and short service lifetimes of IEMs after undergoing high-density electrical field are the hurdles to the widely commercial applications [14]. As one of the potentially commercial desalination technologies, ED arranges the IEMs alternately under a direct current to separate ions from the solution, can produce both fresh water and salt conveniently [15]. To improve the performances of the ED devices and commercial feasibility, the upgrades in the comprehensive properties of IEMs and low cost are essential.

Traditionally, the IEMs are divided into two kinds of charged membranes anion-exchange membrane (AEM) and cation-exchange membrane (CEM) according to the fixed ionic groups. As the key component in ED devices, CEMs have been commercialized owing to the reliable manufacturing technique [16]. AEMs still face technique challenges in large-scale preparation and further improving their performance. AEM is a kind of IEMs having fixed positive charged ion groups on the polymer backbones. It can conduct anions and insulate cations. The backbones of AEMs polymers can be generally divided into two categories, aliphatic family and aromatic family. The aliphatic backbones include polyethylene [17,18], polystyrene [19,20], polyacrylate [21], etc. And polyphenyl [22], poly(phenylene oxides) [23,24], poly(ether sulfones) [25–27], poly(ether ketones) [22,28,29], polybenzimidazole (PBI) [30] have been reported as aromatic backbones. In addition, quaternary ammoniums [31], imidazolium [32,33], pyridinium [33], guanidinium [34], quaternary phosphonium [35], metal cationic groups [36] have been published as the positive charged ion groups located at polymer backbones. Among them, the quaternary ammonium and imidazolium are widely used due to the low cost and the excellent basic properties. The advanced AEMs of these studies have excellent properties and have shown good results when applied to fuel cells. To ED desalination process, high ions permselectivities and conductivities of the AEMs are especially important to improve the efficiencies of the ED processes [37,38]. Xu's works reported that BPPO-based AEMs quaternized by *N*-methylmorpholine (NMM) and dimethylethanolamine (DMEA) showed transport number values up to 0.96 [39] and 0.98 [10] respectively. Both of them had better ED desalination performance than a commercial membrane Neosepta AMX at the flow rate of 60 mL/min of the feed solution (0.1 M NaCl) in the dilute cell under a direct current of 28 mA/cm<sup>2</sup> [10,39]. Aziz ur Rehman group published another series of the quaternary phosphonium functionalized electro dialysis AEMs from BPPO, among which MDPP-43 with a top transport number value of 0.95 presented better ED performance over Neosepta AMX [40]. Shen's work disclosed the ED performances of an internal cross-linked bipyridinium AEM BPPO-20 (NaCl remove: 59.7%; energy consumption: 5.97 kWh/kg NaCl), which were slightly higher desalination efficiency and lower energy consumption than that of Neosepta AMX (NaCl remove: 58.3%; energy consumption: 6.51 kWh/kg NaCl) [41]. Vinod K. Shahi group reported VTMS-co-DMAEMA copolymer based hybrid AEMs fabricated by the combination of sol-gel, cross-linking and quaternization, the best candidate AEM-0.08 with a permselectivity of 0.9 gave highest current

efficiency of 78.46% and lowest energy consumption of 4.77 kWh/kg NaCl at 4.0 V applied potential (Feed of DC: 0.2 M NaCl solution) [42]. Up to now, the reported works have concentrated on the detailed membranes with special designed ionic groups or fine membrane structures and no general instructive results about the influence of ionic groups location have been published. The relationship between the ions distributions and the ED performance should be dug out to direct the design of the new ED candidates.

Our previous works reported that quaternary ammonium functionalized poly(arylene ethers) main-chain type AEMs and side-chain type ones had excellent thermal, chemical stability and conductivity [43,44]. Another work in our group disclosed that a main-chain type AEM based on quaternary ammonium poly(arylene ether ketone) exhibited a high separate factor (94.5 for S, H<sup>+</sup> to Fe<sup>2+</sup>) in diffusion dialysis [45]. These results indicate that quaternary ammonium poly(arylene ethers) AEMs has potential to be applied in ED devices. In this study, we designed and synthesized three different kinds of quaternary ammonium poly(arylene ethers) AEMs with varying ions distributions side-chain ions and main-chain ions to investigate the influence of the designed structures on the ED performances. The IEC, water uptake, swelling ratio, transport number and other parameters were assessed. Also, ED tests were performed to detect their desalination efficiency. To distinguish the AEMs from the current ones, a commercial ED AEM TWEDAI ran the ED test under the same conditions.

## 2. Experimental

### 2.1. Materials

4,4'-Difluorodiphenyl sulfone (FPS) was purchased from Tokyo Chemical Industry Co. Ltd., Tokyo of Japan. *N,N*-Dimethylacetamide (DMAc), azodiisobutyronitrile (AIBN), *N*-bromosuccinimide (NBS), Bisphenol A (BPA), and 1,1,2,2-tetrachloroethane were purchased from Aladdin Reagent, Shanghai of China. 2,2',6,6'-tetramethyl biphenol (TMBP) [46], 3,3',5,5'-tetra(3'',5''-dimethylphenyl)-4,4'-difluorodiphenyl sulfone (8CH<sub>3</sub>-FPS) [47] and 3,3'-di(3'',5''-dimethylphenyl)-4,4'-difluorodiphenyl sulfone (4CH<sub>3</sub>-FPS) [27] was synthesized according to the reported methods. The commercial anion exchange membrane TWEDAI was purchased from Shandong Tianwei Membrane Technology Co. Ltd., China. All the other solvents and reagents in the work were of analytical grade and used as received from the commercial chemical reagents companies.

### 2.2. Synthesis of bromomethylated poly(arylene ethers)

#### 2.2.1. Synthesis of BPAES-a

The N<sub>2</sub> inlet and outlet and the Dean-Stark manifold were installed in a 100 mL round bottom three-necked flask equipped a magnetic stirrer. 8CH<sub>3</sub>-FPS (1.8783 g, 2.8 mmol), FPS (2.1357 g, 8.4 mmol), BPA (2.5568, 11.2 mmol), K<sub>2</sub>CO<sub>3</sub> (2.3219 g, 16.8 mmol), DMAc (16.5 mL), toluene (16.5 mL) were added into the flask. Under nitrogen protection, the reaction was carried out at 150 °C for 4 h. Then the toluene was removed and the temperature was raised to 180 °C. The reactants system gradually became viscous with the prolonging reaction time. Gradually, added about 50 mL of DMAc to dilute the mixture during the polymerization. After reacted for 24 h and cooled down to 80 °C. Added about 400 mL of methanol, 4 mL of concentrated hydrochloric acid to a 500 mL beaker. The reaction mixture was slowly added dropwise to the methanol/hydrochloric acid mixture under vicious magnetic stirring to give a white flocculent precipitate. The product was washed three times with methanol and dried under vacuum at 80 °C for 24 h. The PAES-a product was obtained with a yield of 98.0%.

The bromination process of PAES-a is as follows. 50 mL three-neck round bottom flask was equipped with a spherical condenser, N<sub>2</sub> inlet and outlet, and a magnetic stirring. PAES-a (1.1 g, 2.0137 mmol) dissolved in 1,1,2,2-tetrachloroethane (15 mL), then NBS (0.7965 g, 4.4752 mmol), AIBN (0.03664 g, 0.2231 mmol) were added. The

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