



Performance evaluation of electric-responsive hydrogels as draw agent in forward osmosis desalination



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ABSTRACT

Forward osmosis (FO) has gained more attentions because it has the potential to be an emerging desalination technology. Stimuli-responsive hydrogels, as a novel class of FO draw agent, can completely avoid reverse solute diffusion and release water easily under external stimuli. In this study, 2-Acrylamido-2-methyl-1-propane-sulfonic acid (AMPS) and 2-(Dimethylamino)ethyl methacrylate (DMAEMA) monomers are used to prepare a series of electric-responsive AMPS/DMAEMA hydrogels by aqueous solution polymerization. These hydrogels are adopted as draw agent, by using 2000 ppm NaCl solution as the feed, A0.55D0.45 and A0.53D0.47 hydrogel show better initial water fluxes which are 2.09 and 1.63 LMH, separately. And A0.55D0.45 hydrogel produces 16.12 L·m⁻² total water flux and 0.67 average 24 h water flux. These hydrogels are dehydrated under 15 V electric stimuli. After the first regeneration, their corresponding water recovery rates are 67.45% and 39.36%, separately. After the second regeneration, their initial water fluxes are still recorded at 1.54 and 1.08 LMH, respectively. In contrast with other published literatures, the water flux produced by as-prepared hydrogels has increased significantly. Moreover, using an external electric field to achieve regeneration can simplify the operation of forward osmosis process.

1. Introduction

Rapid population growth and global warming have caused an inhomogeneous supply distribution for available water resource. And the shortage of fresh water has become an important global issue that seriously affects the survival of mankind. Membrane technology for desalination, such as reverse osmosis (RO) [1], nanofiltration (NF) [2] and membrane distillation (MD) [3] and forward osmosis (FO) [4], had a potential advantage to relieve the crisis of freshwater scarcity. FO depends on the osmotic pressure difference across the semi-permeable membrane as the driving force and operates at no or very low hydraulic pressure [5], which leads to decreasing energy consumption of drawing water [6]. Besides, it had higher water recovery, and may have lower fouling propensity or fouling that was more reversible than in RO processes [7,8]. And this membrane technology requires low influent water quality and simplifies the pretreatment process of wastewater, so it has attracted wide attentions around the world. At present, the most widely used draw solutes are inorganic salts like NaCl, which can obtain

higher water flux [9,10]. However, the reverse draw solute diffusion not only result in contaminating the feed solution and reducing the osmotic pressure difference, but also the separation and regeneration of water need high pressure RO process. Therefore, although the membrane develops quickly in FO desalination, preparing and selecting a suitable draw solute, which has some characteristics including high osmotic pressure, lower reverse solute diffusion, low-cost regeneration, non-toxicity and safety, is still the focus for FO technology.

Many researchers have done a lot of researches and attempts on the draw solute. McCutcheon et al. [11] firstly presented ammonium bicarbonate (NH₄HCO₃) as draw solute in FO desalination due to its high osmotic pressure and concluded that it produced higher water flux and needed thermal decomposition at 60 °C to regenerate. Although the moderate thermal regeneration consumed less energy, the reverse diffusion of NH₄HCO₃ was much higher than that of NaCl and alkaline NH₄HCO₃ would affect the service life of FO membrane. Magnetic nanoparticles as draw solute have attracted much attention due to their high osmotic pressures, no reverse solute osmosis and facile recovery,

Abbreviations: AMPS, 2-Acrylamido-2-methyl-1-propanesulfonic acid; DMAEMA, 2-(Dimethylamino)ethyl methacrylate; FT-IR, fourier transform infrared spectroscopy; SEM, scanning electron microscopy; DSC, differential scanning calorimetry; SR, swelling ratio; RO, reverse osmosis; NF, nanofiltration; MD, membrane distillation; FO, forward osmosis; FS, feed solution; DS, draw solution; NaCl, sodium chloride; PAA-Na, polyacrylic acid sodium salts; MBAAm, *N,N'*-methylenebisacrylamide; APS, ammonium persulfate; NaHSO₃, sodium bisulfate; *W*, water content; *R*, water recovery rate; GO, graphene oxide; NIPAm, *N*-isopropylacrylamide; PSA, poly(sodium acrylate); PVA, poly(vinyl alcohol)

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but the conglomerations of nanoparticles and nanoparticles residues in produced water would still limit the practical application [12]. Ge et al. [13] introduced polyelectrolytes of a series of polyacrylic acid sodium salts as draw solutes for FO process in 2012, and their reverse leakages were much lower. Nevertheless, in order to obtain a higher water flux, these polyelectrolytes needed increasing their concentration, which may exacerbate membrane fouling and concentration polarization. At the end of 2014, Guo et al. [14] proposed that 0.5 g/mL Na⁺-functionalized carbon quantum dots could produce the osmotic pressure of 153.6 atm, which was appropriate to become the draw solute for FO. But, it required membrane distillation to concentrate when it was diluted, so the problem for complex regeneration process and higher energy consumption still existed.

Hydrogel was a three dimensional network of hydrophilic polymer which was crosslinked by either physical or chemical bonds, and could contain a large amount of water when maintaining the structure [15]. In 2011, it was the first time for Li et al. [16] to report that temperature-responsive polymer hydrogels were explored as draw agent in FO desalination, followed by light-responsive hydrogels [17], pressure-responsive hydrogels [18] and other kinds of stimuli-responsive hydrogels as draw agent. These kinds of draw agent extracts water molecules from the feed solution via the osmotic pressure, then these swollen hydrogels were dehydrated under the light, heat or pressure to obtain the fresh water [19], so these hydrogels can completely avoid the reverse osmosis. It was reported that weakly crosslinked poly(acrylic acid)/poly(sodium acrylate) copolymer hydrogels with the volume fraction between 0.03 and 0.3 exhibited a swelling pressure ranging from 0.20 to 4.23 MPa [20]. Razmjou et al. [17] designed bifunctional polymer hydrogels layers as a water-absorptive layer and dewatering layer to become the FO draw agents in order to realize the continuous production of fresh water. However, compared to other stimuli-responsive polymer hydrogels, electric-responsive polymer hydrogels are more convenient to apply and control [21]. Hydrogels sensitive to electric current are usually made of polyelectrolytes. And electric-responsive hydrogels undergo shrinking or swelling in the presence of an applied electric field. In 1965, it was first reported that the deformation of polyelectrolyte hydrogels could be observed under the stimuli of an electric field [22]. Electric-responsive hydrogels have shown merits in some fields such as micro-mechanics, flexible actuators and artificial muscle tissues when comparing to other rigid materials. So, our research group have done research on preparing electric-responsive hydrogels as FO draw agent, and simplified the regeneration process by applying electric field which is easy to control and operate [23], but the dewatering rate under electric-stimuli is not carried on.

In this work, the monomer of 2-Acrylamido-2-methyl-1-propanesulfonic acid (AMPS) and 2-(Dimethylamino)ethyl methacrylate (DMAEMA), *N,N'*-methylenebisacrylamide as a cross-linker, ammonium persulfate and sodium bisulfite as redox initiator were employed to prepare AMPS/DMAEMA hydrogels with a range of different monomer ratios by aqueous solution polymerization. AMPS monomer has sulfonic group with high charge density, which is easy to dissociate in solution to become negatively sulfonate ion. And DMAEMA monomer has alkaline tertiary amine groups which can be protonated in acidic medium to become positive ion. Increasing number of ions can enhance the swelling pressure, and high hydrophilicity and conductivity can be helpful to improve the water flux and regeneration ratio under electric stimuli. More importantly, the research on the electric-responsive hydrogels for FO desalination is quite little.

2. Experimental section

2.1. Chemicals and materials

Both monomers 2-(Dimethylamino)ethyl methacrylate (DMAEMA, 99%) and 2-Acrylamido-2-methyl-1-propanesulfonic acid (AMPS, 98%)

were purchased from the Aladdin (Shanghai). *N,N'*-methylenebisacrylamide (MBAAm, ≥ 99.0%) was provided by the Tianjin Kernel Chemical Reagent Co. Ltd. (Tianjin). Sodium chloride (NaCl, ≥ 99.5%) and sodium bisulfate (NaHSO₃) were acquired from Tianjin DaMao Chemical Reagent Factory (Tianjin). Ammonium persulfate (APS) was obtained from the Tianjin GuangFu Technology Development Co. Ltd. (Tianjin). APS and NaHSO₃ were formulated as 8 wt% and 4 wt% aqueous solution before use. All the chemicals were used as received without further purification. The dialysis bag (MWCO = 14,000) used for swelling study was purchased from Sigma. CTA-ES membrane made from cellulose triacetate (CTA) with an embedded polyester screen mesh was obtained from Hydration Technologies Inc. (Albany, OR).

2.2. Preparation of xerogel particles draw agent

AMPS and DMAEMA monomer were polymerized via aqueous solution polymerization to produce the AMPS/DMAEMA hydrogels. First of all, a desired amount of AMPS, DMAEMA and MBAAm were put in a beaker with 30 mL deionized water added. And it was thoroughly dissolved by using a magnetic stirrer for several minutes at room temperature to form a colorless and transparent solution. Secondly, a certain amount of APS and NaHSO₃ solutions (molar ratio = 1:1) were added to the above solution with the molar ratio of monomers, cross-linker and initiator fixed at 100:1:1. The concentration of monomers was 1 mol/L. Subsequently, it was mixed completely by a magnetic stirrer. Finally, it was transferred to the sealed centrifuge tube with a cover, which was put in 50 °C water bath for 5 h to finish polymerization. After polymerization, the centrifuge tube was taken out and cooled to room temperature. Then, columnar hydrogel was removed and soaked into deionized water for three days which was changed every day to remove the unreacted monomers and linear polymer of low molecular weight. The molar ratio of AMPS and DMAEMA was 0.5/0.5, 0.53/0.47 and 0.55/0.45, and as-prepared hydrogels were denoted as A0.5D0.5, A0.53D0.47 and A0.55D0.45, separately. Swollen hydrogels were dried at 60 °C in a convection oven and samples of hydrogel particle size (1–2 mm) were made by grinding dried hydrogels and sieving them. Consequently, the xerogel particles were treated as draw agent in FO desalination.

Only the amount of cross-linker changed, so the molar ratio of monomers, cross-linker and initiator fixed at 100:2:1. According to the above preparation process, the hydrogels fabricated were labeled as A0.5M2, A0.53M2 and A0.55M2, separately. The photos for different hydrogels were shown in Fig. 1a. A0.5D0.5 is white and columnar hydrogel that is very soft and has good elasticity, while A0.53D0.47 and A0.55D0.45 become colorless, transparent and hard with the increasing of hydrophilic sulfonic acid groups. However, A0.5M2 columnar hydrogel is pale white, less soft and much harder than the A0.5D0.5. A0.53M2 and A0.55M2 hydrogel become colorless and transparent because of the more hydrophilic sulfonic acid groups. Overall, the intensity of the latter is better than that of the former, which is attributed to the increasing amount of cross-linker.

In the polymerization, ionic complex with two double bonds was formed by proton transferring reaction between AMPS and DMAEMA firstly. This complex bonded mutually because of the electrostatic force between anion and cation in order to produce a similar polymerized monomer of organic salt structure [24]. Then, ionically crosslinked AMPS/DMAEMA hydrogel was fabricated by free radical polymerization, and its protonated process and the structure of AMPS/DMAEMA hydrogel were exhibited in Fig. 1b.

2.3. Characterization

Swelling behaviors of different xerogels particles were investigated by the gravimetric method. The water uptake of different xerogels particles was determined as follows. 0.3 g different particles were put

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