



Hydrogel-polyurethane interpenetrating network material as an advanced draw agent for forward osmosis process



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ABSTRACT

Water desalination and purification are critical to address the global issue of the shortage of clean water. Forward osmosis (FO) desalination is an emerging low-cost technology for clean water production from saline water. The lack of a suitable draw agent is one of the hurdles for the commercialization of FO desalination technology. Recently, the thermoresponsive hydrogel has been demonstrated to be a potential draw agent for the FO process. However, the commonly used hydrogel powder shows a much lower flux than other kind of draw agent such as inorganic salts. In this work, a hydrogel-polyurethane interpenetrating network (HPIP-N) with monolith form was prepared by controlling the radical polymerization of the monomers (*N*-isopropylacrylamide and sodium acrylate) in the macropores (~400 μm) of commercial polyurethane foam (PUF). These HPIP-N composites show a flux as high as 17.9 LMH, which is nearly 8 times than that of hydrogel powders (2.2 LMH). The high flux is attributed to the 3-D continuous hydrogel-polyurethane interpenetrating network, which can effectively enhance the water transport inside the monolith.

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1. Introduction

Water desalination and purification are critical to address the global issue of the shortage of clean water (Patel-Predd, 2006; Subramani and Jacangelo, 2015; Akther et al., 2015). The applications of membrane-based technique such as nanofiltration (NF), reverse osmosis (RO), membrane distillation (MD), and forward osmosis (FO) are widely used to solve the scarcity of fresh water. Forward osmosis, using the osmotic pressure difference as the driving force to draw the clean water from saline water through a semi-permeable membrane, has been recognized in recent years as an attractive process for water desalination and treatment, because it has the potential to achieve reduced process costs by directly using other types of energy such as low-grade heat, reduced fouling propensity, and is easy to clean (Cath et al., 2006; McCutcheon et al., 2005; Gormly et al., 2011; Hoover et al., 2011; Luttmiah et al., 2011; Chung et al., 2012). Although the FO technology has attracted increasing attentions in water treatment, one of the main obstacles is the lack of high-performance draw solute (Chekli et al., 2012; Ge and Chung, 2013a; Li et al., 2013). It is very challenging to

develop an ideal draw agent with a high osmotic pressure, easy and fast regeneration without significant loss of draw agent, and non-toxicity. Much research has focused on new draw agents such as inorganic salts, functionalized magnetic nanoparticles, and thermoresponsive polyelectrolyte solutions (McCutcheon et al., 2005; Achilli et al., 2010; Ling et al., 2010; Xu et al., 2010; Yen et al., 2010; Bai et al., 2011; Ge et al., 2011, 2012; Ge and Chung, 2013b; Ling et al., 2011; Sarp et al., 2012; Alnaizy et al., 2013; Cai et al., 2013a,b; Ge et al., 2013).

Our group has recently reported for the first time, the use of a stimuli-responsive polymer hydrogel as the draw agent in FO desalination (Li et al., 2011a,b, 2013; Li and Wang, 2013; Ou et al., 2013; Razmjou et al., 2013a,b,c; Zeng et al., 2013; Wang et al., 2014). Hydrogels with three-dimensional networks of polymer chains and abundant hydrophilic groups can entrap large volumes of water due to their relative higher osmotic pressure, and are thus widely used as scaffolds for tissue engineering, as temporary supports for cells and as vehicles for drug delivery systems. Important is the fact that stimuli-responsive polymer hydrogels can undergo a reversible swelling change in response to external environmental stimuli. For example, poly(*N*-isopropylacrylamide) (PNIPAM)-based hydrogels show a lower critical solution temperature (LCST) at about 32 °C, which can change from hydrophilic to hydrophobic

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above their LCST and release the entrapped water from their network. This intrinsic property can be applied for the dewatering of hydrogel after FO desalination, thus exhibiting an energy-efficient method to regenerate the draw agent. Although the hydrogel has been proved to be a potential draw agent, there are still many issues to be solved, such as low flux, and low yield for clean water. To date the hydrogel has usually been used in the powder form, with the particle size ranging from 2 to 1000 μm . As the hydrogel particle size decreases, the contact area between hydrogel and FO membrane increases remarkably, resulting in an enhanced FO flux (Razmjou et al., 2013a,b,c; Hartanto et al., 2015; Zhang et al., 2015; Ali et al., 2015). At the same time, increasing the amount of draw agent will give an enhanced flux but this does not increase indefinitely with more hydrogel. This is because the water transport from the first layer of hydrogel particles contacted with FO membrane to the other layers becomes difficult. Due to this transport barrier, the hydrogel powder shows a lower flux compared to the common used draw agent such as inorganic salts. It is urgent to solve the transport problem for the hydrogel draw agent and further increase the flux, if this approach is to be further used.

Another drawback for the hydrogels is their weak mechanical toughness. After swelling, the hydrogel can be easily broken when handled during application. In order to improve their mechanical properties, researchers reported the synthesis of polymer hydrogel/polyurethane foam (PUF) composites (Liu et al., 2008; Teramoto et al., 2014). As the PUF shows 3-D macroporous structure; it is a good candidate to accommodate the hydrogel. At the same time, the plastic scaffold can also enhance the mechanical properties of hydrogel and make it easier to handle. However, the performance of foam-infiltrated hydrogels as the draw agent material in forward osmosis has never been yet investigated.

In this work, a hydrogel-polyurethane interpenetrating network (HPIP) with monolith form was prepared by controlling the radical polymerization of the monomers (*N*-isopropylacrylamide and sodium acrylate) in the macropores ($\sim 400 \mu\text{m}$) of commercial polyurethane foam (PUF). By tailoring the shape of PUF, the HPIP can be easily shaped. The swollen PUF/hydrogel can be easily handled during the application and shaped even by scissors, without destroying their monolithic shape. The content of hydrogel in the PUF/hydrogel composites can be easily adjusted from 50 to 89 wt% by increasing the concentration of monomers from 12.6 to 20 wt%. These PUF/hydrogel composites were used as draw agents for forward osmosis desalination, and we were able to demonstrate a high flux from 3.9 to 17.9 LMH as the content of hydrogel in the composites from 50 to 89 wt%. By comparison, the flux was only 2.2 LMH when hydrogel powders were used as the draw agent. The high flux is attributed to the 3-D continuous hydrogel-polyurethane interpenetrating network, which can effectively enhance the water transport inside the monolith.

2. Experimental section

2.1. Chemicals and materials

Sodium acrylate (SA, 99%), *N*, *N'*-methylenebisacrylamide (MBA, 99%), *N*-isopropylacrylamide (NIPAM, 96%) and ammonium persulfate (98%) were purchased from Sigma-Aldrich Australia. Forward osmosis (FO) membranes made from cellulose triacetate with an embedded polyester screen mesh were provided by Hydration Technologies Inc. (Albany, OR).

2.2. Preparation of hydrogel draw agent

The poly(*N*-isopropylacrylamide)-*co*-poly(sodium acrylate)

(PNIPAM-*co*-PSA) hydrogel powders were prepared via radical polymerization by using NIPAM and SA as monomers, MBA as a cross-linker, and ammonium persulfate (APS) as an initiator. Typically, 3.33 g of SA and 1.67 g of NIPAM were dissolved in 25 ml of deionized water at room temperature. Then, 0.12 g of MBA and 0.08 g of APS were added into the above solution. After complete dissolution, the polymerization was carried out at 90 °C for 2 h. To remove the unreacted monomers and low-molecular-weight polymer, the hydrogels were cut into small pieces and immersed into deionized water at room temperature for 3 days. The hydrogel was then dried at 80 °C in an oven and the powder form obtained by grinding dried hydrogel with a mortar and pestle.

To prepare PNIPAM-PSA-PU composites hydrogels, the polyurethane foam (PUF) of tailored macro-size was fully soaked in a clear solution of monomers, cross-linker and initiator. The PUF was squeezed using spatulas to exclude the air in the foam. The saturated PUF and residual precursor were heated to 90 °C to cause polymerization. The hydrogel outside of the PUF was carefully scraped before soaking in EtOH and water mixture (v/v, 1:1), to remove the unreacted monomers and low-molecular-weight polymer. The PNIPAM-PSA-PU composite hydrogel was obtained after being dried at 80 °C in an oven. The mass ratio of PUF and hydrogel was calculated by determining the mass of PUF before and after loading hydrogel. The mass ratio of gel respect to PUF (from 1:1 to 8:1) was adjusted by changing the concentration of monomers solution from 12.6 to 20 wt%.

2.3. Characterization

The Scanning Electron Microscopy (SEM) image of PUF and PNIPAM-PSA-PUF composites hydrogel was determined by field-emission scanning electronic microscopy (FEI Nova NanoSEM 450). The sample was sputter-coated with platinum before imaging.

FO measurements were carried out in a home-made setup, as previously reported (Zeng et al., 2013). The dried hydrogel monoliths with size of about $0.5 \times 0.5 \times 1 \text{ cm}$ as a draw agent were placed on the active side of the FO membrane with an effective area of 4.90 cm^2 , while DDI water or NaCl solution with different concentrations (0.2–3.5 wt %) were used as the feed solution on the upstream side of the FO membrane. The FO membrane was immersed in DDI water for at least 12 h before use. Water flux, F ($\text{L m}^{-2} \text{ h}^{-1}$, or LMH), was calculated by

$$F = \frac{V}{At}$$

where V (L) is the volume of water absorbed by the hydrogel, calculated by dividing the mass of the water (i.e., the mass increase of the hydrogel measured by a KERN ASL256-4A balance, Germany, accuracy: 0.1 mg) by its density, t (h) is the time and A (m^2) is the effective area of the FO membrane (4.90 cm^2).

The swelling experiment was carried out by soaking the monolith with the same size (about $1 \times 1 \times 1.5 \text{ cm}$) in 500 ml of DI water. The mass of sample was recorded at intervals after carefully wiping the water adsorbed on the surface of monolith with tissue. To visually investigate the water transport inside the dry hydrogel/PUF, copper nitrate solution was used. Typically, 0.6 g of $\text{Cu}(\text{NO}_3)_2 \cdot 3\text{H}_2\text{O}$ was dissolved in 50 mL of water. Then the solution was poured in a petri dish to obtain a solution of around 3 mm depth. The dry hydrogel/PUF monolith stood in the solution to contact with the solution adequately. The colour of dry hydrogel/PUF monolith was recorded via photos.

In the solar dewatering process, 0.76 g of dry hydrogel PNIPAM-PSA-PUF composites with different swelling ratios (100, 200,

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