



Highly permeable and mechanically durable forward osmosis membranes prepared using polyethylene lithium ion battery separators



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ABSTRACT

A porous polyethylene (PE) membrane commercialized as a lithium ion battery separator was utilized as a support for the fabrication of a highly permeable and mechanically durable thin film composite (TFC) forward osmosis (FO) membrane. The highly open and interconnected pore structure of the PE support combined with its thin thickness ($\sim 8 \mu\text{m}$) is beneficial for mitigating internal concentration polarization, thus enhancing FO water flux. The proper plasma treatment on the PE support and the use of a surfactant enabled the stable formation of a polyamide permselective layer on top of the support via a commercial interfacial polymerization process. The prepared PE-supported TFC (PE-TFC) membrane exhibited a remarkably high FO performance (~ 3.5 times higher water flux and $\sim 35\%$ lower specific salt flux than the commercial HTI-CTA membrane in FO mode) due to its significantly low structural parameter ($\sim 161 \mu\text{m}$) and high permselectivity. The PE-TFC membrane also had superior mechanical properties compare to the much thicker commercial FO membrane due to the exceptionally high mechanical integrity of the PE support, ensuring the mechanically stable membrane operation. The proposed strategy offers a new material platform for FO membranes with strong commercial potential and excellent performance and durability.

1. Introduction

Forward osmosis (FO) is an advanced technology that utilizes an osmotic driving force between a dilute feed solution (FS) and a concentrated draw solution (DS) across a semi-permeable membrane to selectively control water transport [1–3]. Recently, FO technology has received significant attention as a promising separation process applicable to wastewater treatment, seawater desalination, food processing and renewable energy production due to its potential benefits of low operating energy and low fouling propensity [4]. However, the commercialization of the FO process has been hampered by the lack of adequate membranes with high performance and durability [5–7].

Many FO membranes have been developed as the form of a thin film composite (TFC), consisting of an ultrathin polyamide (PA) selective layer fabricated via interfacial polymerization (IP) on top of a porous support layer, owing to its high permselectivity [7–9]. While the PA layer structure has been extensively optimized for achieving high permselectivity, TFC FO membranes still suffer from severe internal concentration polarization (ICP) in the support, which reduces the effective osmotic driving force across the membrane, resulting in the loss

of water flux. Thus, FO membranes should be designed with a support whose structure can minimize ICP. It has been generally recognized that a thin, hydrophilic, highly porous and low tortuous support structure is favorable for facilitating internal mass transport and thus suppressing ICP [10–14]. In addition to these structural requirements, the support requires high mechanical strength to ensure durable and stable membrane operation [15].

A variety of polymeric materials including polyacrylonitrile (PAN), polysulfone (PSf), polyethersulfone (PES), sulfonated PES, sulfonated polyphenylenesulfone, sulfonated poly (ether ketone) and cellulose ether have been explored as the support materials of TFC FO membranes. These polymers have typically been fabricated to the porous supports via a well-known phase inversion method, which are often backed by thick fabric layers for mechanical reinforcement [16–21]. Despite the simplicity of the process, the phase inversion approach generally produces support structures with relatively high tortuosity and thick overall thickness, which greatly increase the mass transfer resistance in the support [18]. Recent studies have focused on tailoring support structures to be more porous and less tortuous for mitigating ICP by utilizing advanced material-structuring techniques such as

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electro-spinning and nanoparticle incorporation [10,22–28]. These techniques have successfully led to the fabrication of high performance FO membranes by effectively reducing ICP but are commercially disadvantageous because of increased production costs, relatively low mechanical integrity and the lack of scalability. For example, nanofiber supports with high porosity and low tortuosity have been prepared via electro-spinning of PAN, cellulose acetate, PES, polyvinylidene fluoride, polyvinyl alcohol and nylon 6,6 [25–28]. Although these electrospun supports greatly enhance FO water flux, their relatively weak mechanical properties and time-consuming manufacturing process reduce their commercial viability [25–28]. These considerations necessitate the development of a high performance and mechanically durable TFC FO membrane using a new class of the support material that is commercially viable.

Porous polyethylene (PE) membranes have been used commercially as lithium ion battery separators (LIBS) owing to their relatively low cost, excellent mechanical and chemical robustness and low mass transport resistance resulting from their highly open and interconnected pore structure [29,30]. This PE membrane also possesses several positive attributes that would suggest their use as a support for TFC FO membrane: (1) the open, interconnected pore structure with high porosity and low tortuosity, which is favorable for reducing ICP (2) high mechanical strength, which can ensure durable and stable operation and (3) commercial availability at a reasonable cost, coupled with a well-established commercial IP process to produce the selective layer, which would facilitate the commercial production of TFC FO membranes.

In this study, we successfully fabricated a highly permeable and mechanically robust TFC FO membrane using a very thin ($\sim 8 \mu\text{m}$) LIBS PE membrane as a support. The PE support was pretreated with O_2 plasma to enhance its hydrophilicity prior to IP, where the impregnation of an amine aqueous solution and the subsequent formation of a PA layer were enabled. The prepared PE-supported TFC (PE-TFC) membrane exhibited superior FO performance and mechanical integrity compared to commercial FO membranes. To the best of our knowledge, this is the first report on the fabrication of highly performing and mechanically robust TFC FO membranes using commercially available LIBS PE membranes.

2. Experimental

2.1. Materials

Trimesoyl chloride (TMC, 98.0%, Tokyo Chemical Industry), *m*-phenylenediamine (MPD, 99.0% Tokyo Chemical Industry), sodium dodecyl sulfate (SDS, 99.0%, Sigma-Aldrich), isopropanol (99.9%, Daejung Chemical), *n*-hexane (95.0%, Daejung Chemical), dodecane (99.0%, Sigma-Aldrich) and sodium chloride (NaCl, 99.0%, Samchun Chemical) were used as purchased. Deionized (DI) water ($18.2 \text{ M}\Omega \text{ cm}$) was prepared using a Millipore Milli-Q purification system. Commercial LIBS PE membranes of $\sim 8 \mu\text{m}$ thickness were received from SK Innovation Co., Ltd (Korea). Two types of commercial FO membranes were obtained from Hydration Technology Innovation (HTI). HTI-CTA membranes consist of cellulose triacetate in which a mesh is embedded, while HTI-TFC membranes have a selective layer on top of a porous

polymer support backed by a non-woven fabric [2,5]. The overall thickness of the HTI-CTA and HTI-TFC membranes was estimated to be $\sim 100 \mu\text{m}$ and $\sim 150 \mu\text{m}$, respectively, and their detailed structures can be found in Supporting information S1.

2.2. Membrane fabrication

A schematic of the fabrication of the PE-supported TFC (PE-TFC) membrane is illustrated in Fig. 1. The strong hydrophobicity of the PE support (the measured water contact angle = $112.6 \pm 2.1^\circ$) prevents from wetting with a MPD aqueous solution, which is the first step of the IP process. In fact, the PA layer was not formed on the pristine PE support without plasma treatment. In addition, the intrinsically weak chemical interaction between the polyolefin (e.g., PE and polypropylene (PP)) support and the PA selective layer is unfavorable for the successful fabrication of a robust TFC membrane [31]. Thus, prior to IP, the PE support was pretreated with O_2 plasma not only to enhance its hydrophilicity and thus improve wetting with the MPD solution but also to increase the adhesion between the support and the PA layer, allowing for the stable formation of a TFC membrane [32]. This plasma pretreatment was conducted using a plasma process system (CUTE-MPR, UVFAB Systems) with a pressure of 0.09 kPa and plasma power of 50 W for 10 s. This plasma condition ensures that the hydrophilicity is reasonably improved without causing structural damage to the support, which was evidenced by no noticeable change in the surface pore structure and mechanical properties of the support after plasma treatment (see Supporting information S2 and Table S1). After the plasma treatment, the PE support became more hydrophilic, as evidenced by its decreased water contact angle ($91.3 \pm 1.4^\circ$). The PA selective layer was then assembled on top of the plasma-treated PE support via conventional IP. The monomer composition used for the IP process was optimized to achieve high flux and NaCl rejection of the membrane based on the preliminary experiment. A MPD (3.0 wt%) aqueous solution containing SDS (0.1 wt%) as a surfactant was poured onto the plasma-treated PE support mounted in a frame and drained off after 10 min. Together with plasma pretreatment on the support, the addition of SDS was found to be essential to the formation of a defect-free PA layer because it can further enhance the wettability of the support by reducing the support-solution interfacial tension, which enables a stable and uniform IP reaction [31,33]. In the absence of SDS, the MPD solution failed to completely wet the support and thus a uniform PA layer coating was unable to be achieved. Once the MPD solution had been drained, a rubber roller was used to remove the excess MPD solution from the support surface. Then, the MPD-impregnated PE support was exposed to a TMC (0.15 wt%) solution in *n*-hexane for 5 min, followed by rinsing with *n*-hexane to terminate the polymerization reaction and remove unreacted TMC, resulting in the formation of a PA layer. The prepared TFC membrane was then dried at an ambient temperature for 1 min and stored in DI water prior to use.

2.3. Membrane characterization

The porosity of the PE support was measured based on gravimetric analysis reported in the literature [12,19,22]. The PE support was immersed into a wetting solution (dodecane) overnight and then taken out

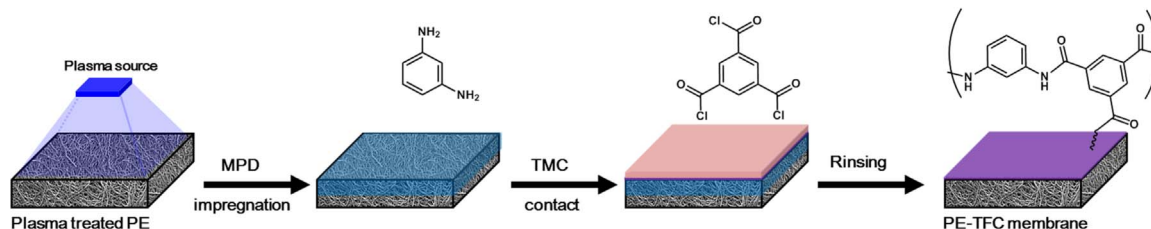


Fig. 1. Schematic illustration of the fabrication of the PE-TFC membrane.

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