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Industrial Scale Manufacturing Sub-10 nm Reverse Osmotic Desalination Membrane on Metallic Single-Walled Carbon Nanotubes Network

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Keywords: metallic single-walled carbon nanotubes network, reverse osmosis, desalination membranes, spiral wound elements, industrial scale manufacturing, aqueous-organic bilayer interfacial polymerization, uniform and smooth sub-10 nm polyamide (PA) nanofilm Reviewer Comment #1: The number and length of keywords seem to be excessive.

Different from extensively reported carbon nanotube-polyamide (PA) composite membranes, this article reports the scalable, low-cost manufacturing of metallic single-walled carbon nanotube network supported PA reverse osmotic desalination membranes. Metallic singlewalled carbon nanotubes (SWCNTs) separated from high pressure carbon monoxide conversion (HiPCO) SWCNTs with a diameter ranging from 0.6 nm to 1.2 nm were deposited on polyethersulfone (PES) films by replacing surfactants around SWCNTs dispersed in aqueous solution, for supporting uniform and smooth sub-10 nm PA nanofilms formed through aqueous-organic bilayer interfacial polymerization. The industrial scale manufactured PA/SWCNT/PES desalination membranes were rolled into spiral wound elements without damage through cutting, gluing, and rolling. The desalination tests on flat PA/SWCNT/PES membranes exhibited 30 liter meter⁻²hour⁻¹ (LMH) with 98.45% salt injection when fed with 1,000 ppm NaCl at pH = 9 under 17.2 bar. The normalized permeate flux of PA/SWCNT/PES spiral wound elements is about 17 LMH with 90% salt injection when fed with 2,000 ppm $MgSO_4$ at $pH = 9-11$ under 13.8 bar. The high-performance sub-10 nm smooth PA/SWCNT/PES desalination membranes are fabricated with materials saving methods and

can promote low operationcosts compared to conventional 50-100 nm crumpled PA films on supporting substrates.

1. Introduction

Among ultrafiltration, nanofiltration, reverse osmosis, and forward osmosis membranes, polyamide (PA) reverse osmosis (RO) spiral-wound membranes are critical for producing new sources of drinkable water and for other desalination needs in the water treatment ofseawater, wastewater, and surface water.1 PA RO membranes are utilized due to their low operating pressure in comparison to other RO membranes, leading to lower energy consumption and lower operating costs. The PA RO membranes used for desalination are manufactured via sequential layer-by-layer interfacial polymerization on a porous support, generating a crumpled PA layer with thickness from 50 nm to 200 nm between crest and trough.2 In the crumpled features resulting from the folding and stacking of the PA layer, the authentic separating PA film is smooth around 10 nm.3 Recently, Jiang et al. reported the fabrication of smooth sub-15 nm polyamide nanofilms with controllable thickness by aqueous–organic bilayer interfacial polymerization, which was transferred onto a variety of porous supports to provide high permeance nanofilm composite membranes for high permeance of 2.69 liter/(m²•hour•bar) desalination under 40 bar.²

PA RO membranes are easily bio-fouled and clogged due to the presence of microorganisms.¹ In order to control these microorganisms and prevent water-borne diseases, chlorine is typically added to disinfect upstream of the desalination sources. Unfortunately, chlorine rapidly degrades PA RO membranes. The incorporation of novel nanoscale materials, such as carbon nanotubes,⁴ graphene,⁵ and carbon nitride⁶ has been reported to improve the chlorine resistance of PA RO membranes. Notably, ~100 nm thick multi-walled carbon nanotube (MWCNT) PA composites fabricated via layer-by-layer interfacial polymerization were reported for high performance RO membranes for anti-scaling, $\frac{7}{7}$ antifouling, $\frac{8}{7}$ chlorine resistance,⁴ and higher flow flux (from 33.5 to 50 LMH under 50 bar).⁹ This improved performance was also reported for MWCNT polypropene nanocomposites¹⁰ and MWCNT polysulfone supports. 11

The enhanced water transport through the inner pores or inter-tubes of aligned MWCNTs was observed.12 Aligned single-walled carbon nanotubes (SWCNTs) with a diameter ranging from

0.67 nm to 1.27 nm embedded in a polysulfone matrix werereported to have 1000 times higher water flow than predicted by Hagen-Poiseuille flow.¹³ This result is similar to aligned double-walled carbon nanotubes encapsulated with chemical vapor deposited SiN_x films.¹⁴ Recent simulations and experimental data show that SWCNTs with 0.8 nm diameter are better water channels than protein aquaporins.¹⁵

In this report, the high-pressure carbon monoxide conversion (HiPCO) metallic single-walled carbon nanotube (SWCNT) network with an average length of \sim 1 μ m within a diameter from 0.6 nm to 1.2 nm was employed as the nanomaterial support for smooth sub-10 nm polyamide nanofilms fabricated via organic-aqueous bilayer interfacial polymerization on porous polyethersulfone (PES) substrates. The HiPCO metallic SWCNT network was deposited on PES substrates via surfactant displacement from a 2% sodium dodecyl sulfate dispersed aqueous solution. The HiPCO SWCNT network was characterized using a scanning electron microscope to show the metallic carbon nanotube network covering the pores as well as the carbon nanotubes inside the pores of the PES substrates. The measured contact angles of these membranes also indicated that the covered metallic SWCNTs reduce the roughness of the PES substrates. These mechanically strong, interconnected HiPCO metallic SWCNT networks could act as binders between PES and PA due to their π -conjugated surfaces that incline to bind the phenyl ring and amide of PA. These properties boost the performance of PA/SWCNT/PES desalination membranes, as demonstrated in desalination tests using both flat sheets and rolled spiral wound elements.

2. Experimental section

The high pressure carbon monoxide (HiPCO) metallic single-walled carbon nanotube (SWCNT) solution was stocked from previous semiconducting SWCNT work.16 HiPCO SWCNTs are single-walled carbon nanotubes produced catalytically by converting carbon monoxide in high pressure, high temperature reactors invented by Nobel Laureate Richard Smalley at Rice University, resulting in smaller diameters and fewer surface defects.¹⁷ Briefly, HiPCO SWCNT raw powder (100 mg) was added to 100 mL 2% sodium dodecyl sulfate (SDS, 99+% pure) aqueous solution and sonicated using an ultrasonic processor (Cole Parmer, 20 W) equipped with a 0.5-inch Ti flat tip for 20 hours under continuous water cooling. The residue catalyst, large nanotube bundles, and other impurities were removed via ultracentrifugation (45,000 rpm) using a Beckman TL-100 ultracentrifuge equipped with a

TLS-55 rotor. The top 90% of the supernatant was collected as the starting solution for gel chromatography18 and the near-infrared-visible (NIR-Vis) absorption spectrum was measured on an NS3 Nanospectralyzer. **Figure 1** shows the characteristic features of S₁₁ (830-1400) nm), S_{22} (600-800 nm), and M_{11} (400-645 nm) Van Hove singularities of small diameter SWCNTs. The supernatant HiPCO SWCNT solution was loaded onto an in-house packed column with allyl dextran-based gel beads. The eluted HiPCO SWCNTs were stocked and used in the fabrication of desalination membranes. The NIR-Vis absorption spectrum of these eluted HiPCO SWCNTs is dominated by $M₁₁$ (400-645 nm) Van Hove Singularities of metallic SWCNTs as shown in **Figure 1**. Based on these absorption peaks, the diameters of these HiPCO metallic SWCNTs are estimated to be in the range of 0.6 nm to 1.2 nm.^{19,20}

Figure 1. NIR-Vis absorption spectrum of 1 weight% raw HiPCO SWCNTs in 2% SDS aqueous solution (blue curve) and the normalized absorbance of HiPCO metallic SWCNTs eluted from a gel column using 2% SDS aqueous solution (red curve).

The industrial processes of manufacturing PA nanofilms on HiPCO metallic SWCNT network covered PES membranes are illustrated in **Figure 2**. These processes can be automated into a roll-to-roll system. After the white PES film was completely laid down in the tray (Step 1 &

Step 2), HiPCO metallic SWCNT solution was injected in the tray (Step 3) and then discharged after remaining insolution for 1 hour (Step 4). One layer of HiPCO metallic SWCNTs was attached to PES through *surfactant displacement to form grey PES films*, ascribed to the hydrophobic surface interaction and π -π interaction between the surface of SWCNTs and the phenyl rings of PES. These interactions are mechanically strong, evidenced by no apparent removal of metallic SWCNTs after vigorous scratching (Supporting **Figure S1**). After extensive washing with water, a 3 weight% m-phenylenediamine (MPD) colorless aqueous solution was injected into the tray (shown as pseudo green) (Step 5). Then, 0.15 weight% trimesoyl chloride (TMC) colorless hexane solution was carefully injected on the surface of the MPD aqueous solution (shown as pseudo pink) (Step 6). MPD and TMC underwent polymerization at the interface of hexene and water to form ~10 nm smooth PA nanofilms (Step 7). Although a layer of 10 nm PA can form in about 1 minute, the bilayer interfacial polymerization was allowed to proceed for 30 minutes to secure high quality PA nanolayers. The MPD aqueous solution was slowly discharged from the bottom of the tray, and PA nanofilms were pushed down onto the surface of HiPCO metallic SWCNT covered PES films following solution draining (Step 8). The TMC hexane solution was completely removed from the bottom of the tray to form PA/SWCNT/PES desalination films as elicited in the expanded drawings (Step 9). Both the MPD aqueous solution and TMC hexane solution were recycled for use. The hydrophobic surface interaction and π -π interaction between the surface of metallic SWCNTs and the phenyl rings of PA could help the binding of PA on SWCNTs/PES.

Figure 2. Illustrative drawings of industrial manufacturing of smooth sub-10 nm PA films on HiPCO metallic SWCNT covered PES substrates. Step 1, lay the PES substrate into the tray; Step 2, completely lay down the PES substrate in the tray; Step 3, inject HiPCO metallic SWCNT stock solution to fully cover the PES substrate in the tray; Step 4, discharge HiPCO metallic SWCNT aqueous solution from the tray; Step 5, inject 3% MPD aqueous solution to fully cover the PES substrate in the tray; Step 6, slowly inject 0.15% TMC hexane solution from the surface of 3% MPD aqueous solution for full coverage; Step 7, MPD and TMC undergo bilayer interfacial polymerization to form PA nanofilms; Step 8, slowly discharge 3% MPD aqueous solution from the bottom of the tray and push down the bilayer interfacial polymerized PA nanofilms ontoHiPCO metallic SWCNT covered PES substrate; Step 9, completely remove 0.15% TMC hexane solution from the bottom of tray. The last drawing shows the tri-layer structure of the PA/SWCNT/PES desalination membrane.

Samples were etched by a focused ion beam using a Nova 600 SEM/FIB System to expose a cross section and transfer it to a TEM grid for imaging. TEM was performed using an FEI Tecnai T12 transmission electron microscope.

Samples were gold sputtered, then SEM imaging was performed under 30 kV using a ZEISS Supra 40VP SEM.

Additional experimental details may be found in the SI section.

3. Results and Discussion

The PES substrates (0.03 Micron 200 mm \times 200 mm (PES0032005) and 508 mm \times 915 mm (PES1480010) supplied by Sterlitech) and HiPCO metallic SWCNT network covered PES membranes were imaged with scanning electron microscope (SEM) as shown in **Figure 3**. The SEM images of the PES show large pores ranging from 100 nm to 500 nm with certain tiny ones. Their surfaces are rather rough. The SEM images of metallic SWCNT thin film covered PES (SWCNT/PES) exhibit carbon nanotube networks on the surface of the PES as well asinside the pores of the PES. Around some larger pores of the PES, the metallic SWCNTs aggregated to SWCNT bundles on their edges due to coffee ring effects instead of covering the pore areas. The PA/SWCNT/PES desalination membranes were also imaged with SEM, presented in **Figure 4**. The gold sputtered SEM images are uniform and smooth. No pores were imaged on these PA/SWCNT/PES membranes. The PA/SWCNT/PES membranes were also directly imaged without metal sputtering. Many regular and smooth surfaces were observed. The SWCNTs underneath the PA nanofilm were also imaged, as shown in the inset of **Figure 4**. Different from PA carbon nanotube composites, which are highly dependent upon the loading of carbon nanotubes, $4,21$ the SWCNT network in PA/SWCNT/PES desalination membranes serve as a smooth support for sub-10 nm PA and the binding layer between PES and PA.

Figure 3. SEM images of PES films with sputtered gold nanoparticles (top) and HiPCO SWCNT covered PES films directly imaged (bottom).

The contact angles of PES, SWCNTs/PES and PA/SWCNTs/PES were measured and compared in **Figure 5**. All three contact angles are less than 90°, indicative of the hydrophilic surfaces of PES, SWCNTs/PES and PA/SWCNTs/PES. The smaller the contact angle is, the rougher the surface is.²² Thus, PES has a very rough surface with a contact angle of 27.14°. With a coating of HiPCO SWCNT thin film, the roughness of SWCNTs/PES is slightly reduced to 32.43°. With a smooth and uniform PA layer, the contact angle of PA/SWCNTs/PES increases to 74.82°.

Figure 4. SEM images of PA/SWCNT/PES desalination membranes directly imaged (left) and imaged with sputtered gold nanoparticles (right).

Figure 5. The measured contact angles of PES, SWCNTs/PES and PA/SWCNTs/PES.

The PA/SWCNT/PES desalination membrane was cut using an E-beam (**Figure 6A**) and tilted to 52 degrees for SEM imaging (**Figure 6B-D**). **Figure 6A** shows the PA layer on typical porous PES/SWCNTs. **Figure 6B** exhibits the cut hole on the PA/SWCNT/PES membrane with porous structures underneath the smooth surface, which is consistent with the

PES SEM images (**Figure 3**). With magnified SEM images from 1 micron to 400 nm (**Figure 6C**), then 50 nm (**Figure 6D**), the thickness of the PA nanofilm was measured to be between 7.1 nm and 8.5 nm in different locations, which falls in the normal range of thicknesses for organic-aqueous bilayer interfacial polymerization films.2,3

Figure 6. SEM images of deposited platinum bar on PA/SWCNT/PES desalination membrane with sputtered gold nanoparticles (**A**); E-beam cut PA/SWCNT/PES desalination membrane on the deposited Pt bar (**B**); magnified image of the cross-section of E-beam cut area (**C**); further magnified image of the cross-section of E-beam cut area with labeled PA thickness (**D**).

The desalination tests of PA nanofilms on flat PES/SWCNT composites $(200 \text{ mm} \times 200 \text{ mm})$ were conducted at both UCLA and Membrane Development Specialists (MDS) with consistent results. The results show around $30 \text{ lm}^{-2}h^{-1}$ (LMH) water flux and 98.45% salt rejection when fed with 2000 ppm NaCl at pH = 9 under 17.2 bar(**Figure 7**). No leakage was

detected during the test processes. This indicates that SWCNTs filled in rough commercial PES substrates play a significant role in supporting the sub-10 nm PA membrane. The lower pressure and high-water flux performance of smooth PA nanofilms on flat PES/SWCNT composites are consistent with other reports of PA/carbon nanotube composites. $4-10$, 22 This indicates that PA nanofilms on SWCNT/PES composites can withstandharsh conditions, have a long lifetime, and efficiently generate pure water at low operation costs.

Figure 7. The desalination test of 200 mm \times 200 mm PA/SWCNT/PES membranes(shown in the inset) fed with 2,000 ppm NaCl at $pH = 9$ under 17.2 bar, showing the stabilized flux of 30 LMH and 98.45% salt rejection.

The PA coating was also formed using conventional layer-by-layer interfacial polymerization (HiPCO metallic SWCNT covered PSE was immersed in 3% MPD aqueous solution, then placed in 0.5% TMC hexane solution). The formed membrane achieved 86.9% salt rejection when fed with 1000 ppm NaCl at pH = 8 under 17.2 bar(**Figure S2**). This may be due to SWCNTs intervening in the PA formation and leaving some openings, leading to lower salt rejection.4,21

The PA/SWCNT/PES/ desalination membrane scrolls (508 mm \times 915 mm) were rolled up by MDS to form spiral wound elements (Element 695447 and Element 695446) for the test (**Figure 8**). Element 695447 was rolled from a flat piece of membrane. Initially, about 75% rejection was achieved, but the rejection was reduced to 65% when pressure was increased. This means element 695447 had a mechanical leak within the membrane itself or on a glue line. On a good membrane and spiral wound element, if pressure increases, more water will permeate and allow the same amount of salt to be transmitted. Consequently, the permeate contains less salt, leading to an increased percentage of salt rejection. The spiral wound element 695446 was run at 27.6-34.5 bar with no pressure-related leaks and achieved 90% salt rejection (**Figure 8**). The normalized permeate of element 695446 is around 10 GFD (gallon feet⁻² day⁻¹) (17.01 LMH) when fed with 2,000 ppm MgSO₄ at pH = 9-11 and under 13.8 bar. These results show that the PA/SWCNT/PES/ desalination membranes can be handled without damaging the membrane during the spiral wound manufacturing processes of cutting, folding, and gluing the membrane into spiral wound elements, which is crucial for large scale industrial processing.

Figure 8. Photographic images of a 508 mm × 915 mm PA/SWCNT/PES desalination membrane, fabrication tray, and rolled spiral wound element (top). The detected salt rejection and normalized permeate at 13.8 bar and 25 °C from the desalination test on rolled spiral wound elements fed with 2,000 ppm MgSO₄ (bottom).

4. Conclusion

In summary, industrial scale manufacturing of smooth sub-10 nm PA nanofilms on HiPCO metallic SWCNT covered PES substrates (200 mm \times 200 mm and 508 mm \times 915 mm) through aqueous-organic bilayer interfacial polymerization was reported. The SEM and contact angle characterizations show the HiPCO metallic SWCNT network covered on the surface of PES, reducing both the pore area and surface roughness to provide high performance supports for flat PA nanofilms. The desalination test on flat $(200 \text{ mm} \times 200 \text{ mm})$ PA/SWCNT/PES membranes fed with 1,000 ppm NaCl at $pH = 9$ under 17.2 bar shows the water flux of 30 LMH with 98.45% salt rejection. The rolled spiral wound elements based on 508 mm × 915 mm PA/SWCNT/PES membranes were tested for desalination by feeding with 2,000 ppm MgSO₄ at $pH = 9-11$ under 13.8 bar. The normalized permeate flux of the spiral wound elements is around 10 GFD (17 LMH) with 90% salt rejection. The flat PA sub-10 nm nanofilm formed through aqueous-organic bilayer interfacial polymerization is materials saving in comparison to 50-100 nm crump PA film formed through layer-by-layer interfacial polymerization. Additionally, the sub-10 nm PA nanofilm will achieve higher permeation at lower pressure, consequently reducing operation costs. To further reduce the spiral wound element costs, the blend of HiPCO SWCNTs (especially acid treated SWCNTs with open ends) and PES or PA (other polymers) could be promising to be directly pasted on Tricot to eliminate two steps of membrane preparation.

Supporting Information

The following file is available free of charge.

Figure S1: Photographic images of SWCNTs/PES before and after vigorous scratches. Figure S2: The desalination test of 200 mm \times 200 mm PA/SWCNT/PES membranes (layerby-layer interfacial polymerized PA) fed with 1,000 ppm NaCl at $pH = 8$ under 17.2 bar showing the stabilized flux of 25 LMH and 76-87% salt rejection.

Acknowledgements

We would like to thank Professor David Jassby from the Department of Civil and Environmental Engineering at the University of California, Los Angeles for desalination membrane tests and Mr. Larry Lien from Membrane Development Specialists for rolling the spiral wound elements and performing the desalination membrane tests.

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Supporting Information

Industrial Scale Manufacturing Sub-10 nm Reverse Osmotic Desalination Membrane on Metallic Single-Walled Carbon Nanotubes Network

Huaping Li, Isabelle Winardi, Feirong Luo, Nadya Smolinski, Noah Smolinski, Michelle B. Li, and Qibing Pei*

Materials:

High pressure carbon monoxide conversion (HiPCO) single-walled carbon nanotubes were licensed and transferred from Rice University to Atom Inc. Sodium dodecyl sulfate (SDS), mphenylenediamine (MPD), trimesoyl chloride (TMC) were purchased from Sigma-Aldrich. Hexane and deionized water were supplied by FisherScientific. Polyethersulfone (PES) was purchased from Sterlitech.

Experiments:

Visible-Near-Infrared absorption was measured on an NS3 Nanospectralyzer.

Samples were etched by a focused ion beam using a Nova 600 SEM/FIB System to expose a cross section and transfer it to a TEM grid for imaging, performed by Dr. Noah Bodzin. TEM was performed using an FEI Tecnai T12 transmission electron microscope.

Samples were gold sputtered, then SEM imaging was performed under 30 kV using a ZEISS Supra 40VP SEM.

Bench Test Spiral Element and Flat Sheet P+I Diagram

Figure S1. Photographic images of SWCNTs/PES before and after vigorous scratches.

Figure S2. The desalination test of 200 mm \times 200 mm PA/SWCNT/PES membranes (layerby-layer interfacial polymerized PA) fed with 1,000 ppm NaCl at pH = 8 under 17.2 bar showing the stabilized flux of 25 LMH and 76-87% salt rejection.