Vapor Phase Infiltration of Metal Oxides into Microporous Polymers for Organic Solvent Separation Membranes

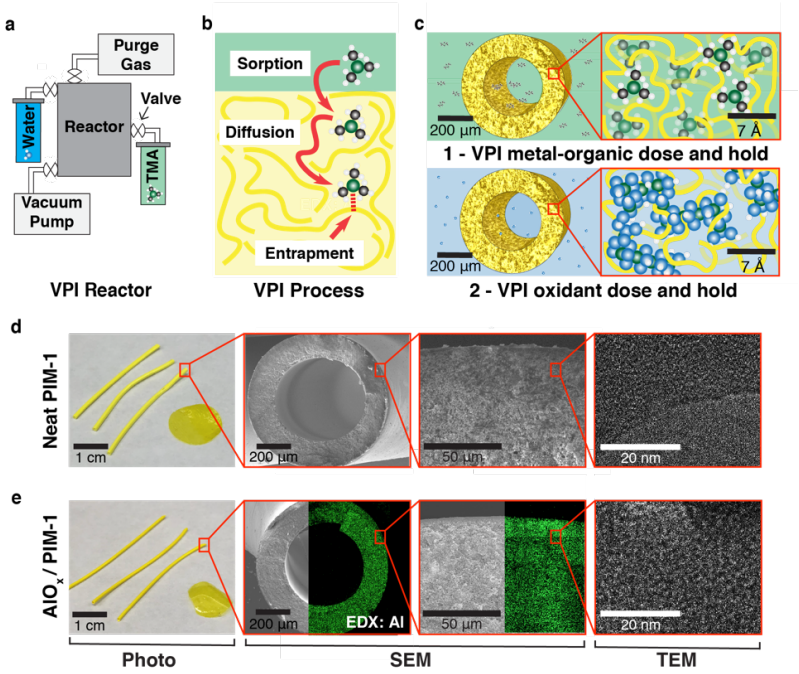

Figure 1. Schematic of vapor phase infiltration (VPI) and morphology characterization of pristine and hybrid PIM-1 membranes. (a) Diagram of the reactor used for VPI. Precursors are dosed into a static vapor environment. The chamber is pumped and purged with nitrogen between the precursor and co-reactant doses. (b) Schematic depiction of VPI process: precursor sorption, diffusion, and entrapment (coordination or chemical reaction). (c) Schematic depiction of (1) metal-organic precursors sorbing into the PIM-1 microporosity and becoming trapped and (2) water vapor sorbing into the structure and reacting with the metal-organic to form an interpenetrating metal oxide network. In this report, infiltration of precursors and co-reactants are often cycled twice to increase the inorganic loading. (d, e) Photographs, SEM images, and TEM images of (d) PIM-1 before infiltration and (e) PIM-1 after infiltration with trimethylaluminum and water (2 cycles). Green pixels in EDX map show aluminum distribution throughout the hybrid membrane.

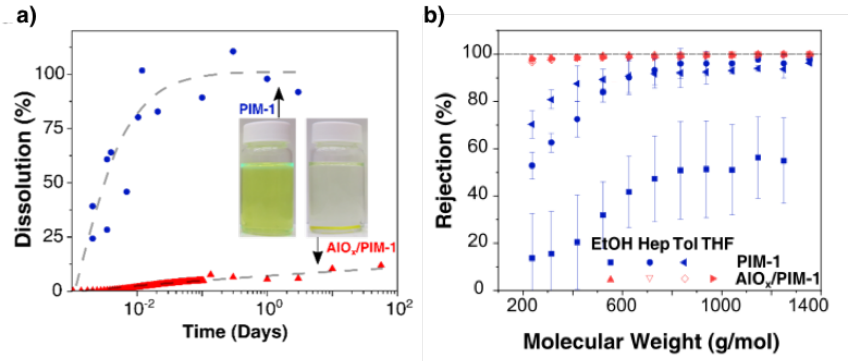

Figure 2. Dissolution, organic solvent nanofiltration (OSN), and organic solvent reverse osmosis (OSRO) performance of PIM-1 and AlO_x/PIM-1 hybrid thin film composite membranes (2 cycles of TMA and H_2O infiltration, with 10 minute TMA and water exposures at 90˚C). (a) Dissolution of PIM-1 and AlO_x/PIM-1 membranes in tetrahydrofuran (a strong solvent for PIM-1) determined via UV-Vis. The dashed lines are included to guide the eye. (b) Molecular weight cut-off curves (MWCO; the smallest molecular weight the membrane "successfully" rejects) of PIM-1 and AlO_x/PIM-1 thin film composites in different solvents using polystyrene oligomers as markers. Using 1,3,5-triisopropylbenzene, and 1,3-diisopropylbenzene as markers, the MWCO for PIM-1 and AlO_x/PIM-1 hybrid thin film composite membranes was found to be 204 g/mol.