Casting Polyvinylidene Difluoride on Graphene for Nanoscale Filtration Systems

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Supporting Information

Making PVDF Solution

PVDF Solution was made using a weight percent measurement of 88% of N-Methyl-2pyrrolidone (NMP) and 2% of Isopropyl Alcohol (IPA) were combined into a glass vile, and 10% of PVDF powder was added and mixed via shake plate. The solution was then baked at 40C for a minimum of 4 hours, then degassed for a minimum of 5 hours. Baking and degassing the solution eliminates any impurities in the solutions, as well as getting rid of any excess gas that may be trapped in the solution. Once degassed, the solution was sealed and stored at room temperature until use. Figure S1 shows the process of making PVDF solution below.



Figure S1. Creating PVDF solution.

Etching Copper Off the PVDF Layer

Once removed from the bath, the copper was cut out of the tape and allowed to dry. To etch away the copper, a crystallization dish was filled with 100 mL ammonium persulfate (APS) solution (0.2 mol/L APS concentration). Using a glass slide, the copper sample was placed copper side down into the solution and allowed to sit for 10 minutes then transferred to a glass dish filled with deionized water for 10 minutes. The sample was gently picked up with tweezers and dabbed across a paper tower before being placed back in the water for 10 minutes. This is called pre-etching, and its purpose is to prepare the copper and the under layer of graphene to be fully etched away, off of the graphene. This process was repeated twice, then the sample was placed in APS for at least 5 hours or until the copper is fully etched off. After 5 hours, the sample was removed from APS solution via glass slide, and transferred to a water bath for 10 minutes,

then transferred to a fresh, water bath, and allowed it to sit for 10 more minutes. Allowing the copper to sit in a water bath after being soaked in APS washes away any excess APS that may be lingering on the membrane. The sample was then placed in ethanol bath for a minute, then fully submerged in ethanol and allowed to dry overnight. Figure S2 below shows the phase inversion step of creating PVDF membranes, as well as the etching process.



Figure S2. Casting and etching PVDF.

Imaging

The casting was attached to a glass slide using carbon tape with the graphene side facing up. The slide was inserted into the Zeiss Merlin Scanning Electron Microscope (SEM) and images were collected, first looking at the top of the sample, then looking at the cross section, or edge. A 2 kV beam power and a 90-pA beam current was used to capture the images. The top of the sample shows the porosity of the sample, looking at both the number of pores and their size. The cross section shows the thickness of the membrane from a side view, in order to get a clear measurement.

Using ImageJ, the images' porosity and average pore size were determined. Porosity was found using the threshold tool, toggling the percentage cover until it met most of the pores. The average pore size was found by setting the scale of the image and drawing lines across the diameter of the pores. Several measurements were taken to accumulate an average. Each cross section was measured by setting the scale and drawing lines across the PVDF layer in the image. Figure S3 and S4 show a visual representation of how to use ImageJ.



Figure S3. Measuring Pore Diameter using ImageJ



Figure S4. Measuring Porosity using ImageJ

The top image shows how the threshold of each image was taken to determine the porosity. The thresholds of each image were then averaged to determine a final porosity coverage. The bottom image shows how the diameter of the pores was measured. Several pores were measured across each image, those were then averaged to determine the pore size.