Shear align graphene oxide nanosheets incorporated PVDF composite membrane for selective dye rejection with high water flux

Sk Safikul Islam[1], Theres Jose[1], Asiful Hossain Seikh[2], Mohammad Rezaul Karim[2], Ibrahim A. Alnaser[2,] and Suryasarathi Bose[1,] **

[1]Department of Materials Engineering, Indian Institute of Science, Bangalore, Karnataka, India-560012,

[2] Center of Excellence for Research in Engineering Materials (CEREM), King Saud University, P.O. Box 800, Al-Riyadh 11421, Saudi Arabia

*Corresponding author: sbose@iisc.ac.in; ianaser@ksu.edu.sa

Table of Contents

1. Characterization Tools 2

2. Pure water flux 2

3. Dye sieving performance 3

4. Desalination performance 3

5. XPS high-resolution spectra C 1S of SAGO@PVDF 4

6. Cross-sectional SEM images of the SAGO@PVDF membrane after 60 days of immersion in acidic, neutral, and alkaline solutions 4

7. Small-angle X-ray scattering (SAXS) (Iq2 vs q) plots of SAGO@PVDF membrane 5

Characterization:

To evaluate the formation of Goa and SAGO@PVDF membrane, FTIR spectra on the films were recorded on a PerkinElmer frontier by accumulating 16 scans over a range of 600−4000 cm^{-1} to obtain information about the different crystalline forms. X-ray diffraction (XRD) was carried out for further investigation of the crystalline phases on a PAN analytical X'pert pro using Cu Kα source operating at 40 kV and 30 mA in the 2θ range of 10-80° and a scan rate of 0.04° s^{-1}. The crystallographic texture was measured using X-ray texture goniometer based on Schulz reflection geometry using Co Kα radiation (D8 Discovery, Bruker). SAXS measurements were conducted utilizing a XEUSS SAXS SYSTEM equipped with a rotating anode copper source operating at 45 kV and 100 mA. To reduce air scattering, a vacuum environment was maintained throughout the entire X-ray beam path, covering the trajectory from the source to the sample and detector. XPS as performed on Axis Ultra (equipped with Al monochromatic source (1.486 keV)). The zeta potential of the membranes was investigated from the Surpass 3 instrument, Anton Paar having an adjustable cell set at a gap height of 102 μm and pH 7. Water contact angle measurements were carried out in a FAMAS water contact angle goniometer using sessile drop method. Thermogravimetric analysis was performed using TA Q500. UV-Vis spectra were generated using Perkin Elmer's UV/Vis/NIR Spectrometer, Lamda 1050+. SAGO@PVDF membrane surface uniformity and cross section was analysed by a scanning electron microscope (Ultra55 FE-SEM) from Karl Zeiss.

Pure water flux:

The pure water flux and sieving performance of the SAGO@PVDF membranes were evaluated by using hand made flux setup. Each membrane, with a diameter of 30 mm, was compressed at various pressures for a duration of 15 minutes. Subsequently, the measurements were taken three times across a pressure range of 10 psi to 100 psi to ensure data.

The pure water flux (Jw) was calculated using the following formula:

$$Jw = \Delta V / (A * \Delta t)\ (Lm^{-2}h^{-1}) \qquad (1)$$

Here, ΔV represents the change in volume of the feed over a given time interval (Δt), and A denotes the effective area of the membrane.

Dye sieving performance:

In order to assess the dye rejection performance of the SAGO@PVDF membranes, 20 ppm solutions of cationic and anionic dyes were employed as model foulants. A dead-end filtration setup was utilized, and the permeate was collected under a pressure of 58 psi. The concentration of the dye in the permeate was determined through UV Vis Spectrometry.

The dye rejection performance was calculated using the following equation:

$$\% \text{ Rejection} = [1 - (C_p / C_f)] * 100 \quad (2)$$

Where, C_p represents the concentration of the dye in the permeate (in ppm), and C_f corresponds to the concentration of the dye in the feed (in ppm).

Desalination performance:

The salt rejection performance of SAGO@PVDF membranes was investigated using Sterlitech dead-end setup under a pressure of 30 psi. In this study, a monovalent salt, NaCl, and divalent salt Na_2SO_4 at a concentration of 1000 ppm, were used as the feed solution.

For salt rejection experiment, the rejection can be calculated by using the following relation using the salt concentrations in the feed and permeate

$$\% R = [1 - (C_p / C_f)] *100 \quad (3)$$

where Cp and Cf represents in eq 3 are the concentration of the salt in the permeate (in ppm), and the concentration of the salt in the feed (in ppm).

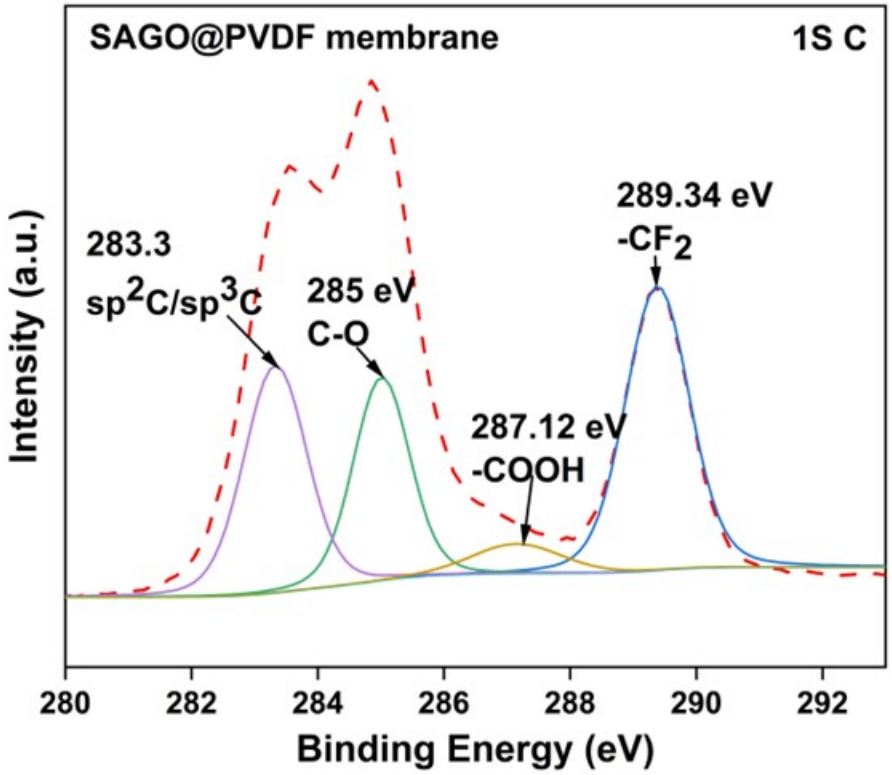

Figure S1. XPS high-resolution spectra C 1S of SAGO@PVDF

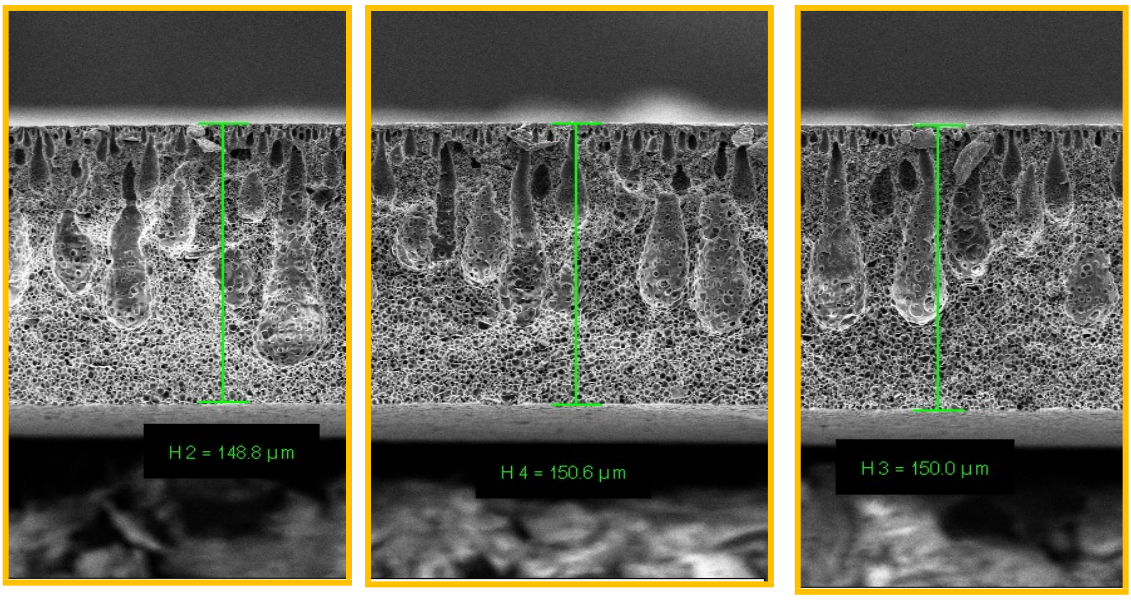

Figure S2. Cross-sectional SEM images of the SAGO@PVDF membrane after 60 days of immersion in acidic, neutral, and alkaline solutions (from left).

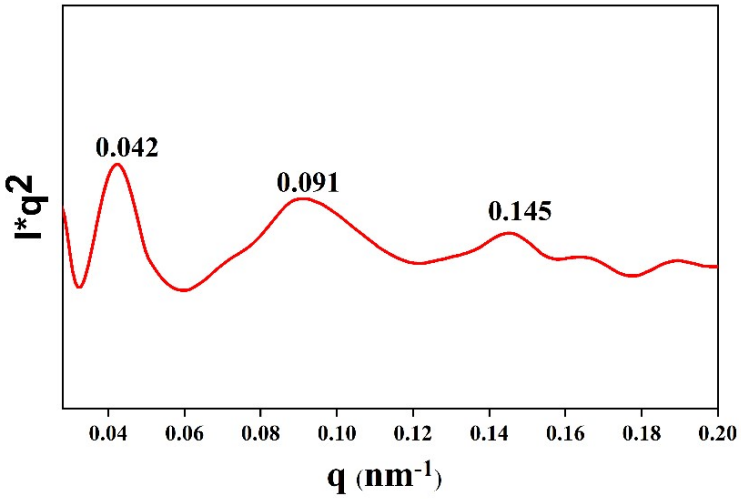

Figure S3. Small-angle X-ray scattering (SAXS) (Iq2 vs q) plots of SAGO@PVDF membrane