

## Electronic Supplementary Information

# **A novel strategy to fabricate inorganic nanofibrous membranes for water treatment: Use of functionalized graphene oxide as a cross linker**

Tong Zhang, Jincheng Liu\*, and Darren Delai Sun\*

*School of Civil and Environmental Engineering, Nanyang Technological University,  
Nanyang Avenue, 639798, Singapore*

E-mail: [JCLiu@ntu.edu.sg](mailto:JCLiu@ntu.edu.sg) (J. C. Liu); Fax: +65-6791-0676; Tel: +65-6790-9073;

E-mail: [DDSun@ntu.edu.sg](mailto:DDSun@ntu.edu.sg) (D. D. Sun); Fax: +65-6791-0676; Tel: +65-6790-6273;

## **Experimental Section**

### **Preparation of GO**

GO was synthesized according to the modification of Hummer's method from natural graphite,<sup>1</sup> and the process was described previously.<sup>2</sup>

### **Preparation of GO-SO<sub>3</sub>H**

To synthesize GO-SO<sub>3</sub>H, 100 mg of GO, 1.5 g of Sodium 2-chloroethanesulfonate hydrate and 800 mg of NaOH were added into 500 ml deionized water, and the suspension was subjected to ultrasonication for 3 h for reaction. Subsequently, 2 mL of concentrated HNO<sub>3</sub> was injected into the system. After stirring the mixture for 30 min, the resultant product was centrifuged and washed with ethanol for three times. Finally, the product was dispersed into 200 mL deionized water.

### **Preparation of K-OMS-2 nanowires**

K-OMS-2 nanowires were synthesized via a hydrothermal method.<sup>3</sup> In a typical procedure, 19.1 mmol of  $K_2SO_4$ , 38.2 mmol of  $K_2S_2O_8$ , and 19.1 mmol of  $MnSO_4 \cdot H_2O$  were dissolved in 80 ml of deionized water. The solution was then transferred to a 125 ml Teflon-lined stainless-steel autoclave. The autoclave was sealed and heated in an oven at 250 °C for 4 days. The resulting black precipitate was suspended in 1000 ml deionized water, and stirred vigorously for 12 h. After thorough washing with deionized water to remove remaining ions present in the product, the sample was dried at 105 °C for 24 h.

#### **Preparation of hierarchical K-OMS-2/GO-SO<sub>3</sub>H heterojunctions**

200 mg of the synthesized K-OMS-2 nanowires were dissolved in 50 ml the prepared GO-SO<sub>3</sub>H suspension. After homogenization under ultrasonic condition for 30 min, hierarchical K-OMS-2/GO-SO<sub>3</sub>H heterojunctions were formed.

#### **Assembly of K-OMS-2/GO-SO<sub>3</sub>H membrane**

The suspension of the synthesized hierarchical K-OMS-2/GO-SO<sub>3</sub>H heterojunctions was well dispersed under ultrasonic condition. Then, the suspension was filtered on a vacuum-filtration setup with a glass filter (ADVANTEC, GC-50, 0.45 μm), and the hierarchical K-OMS-2/GO-SO<sub>3</sub>H heterojunctions will form a compact cake layer on the glass filter. After drying at 105 °C for 1 day, a free-standing membrane was formed after removal of the glass filter.

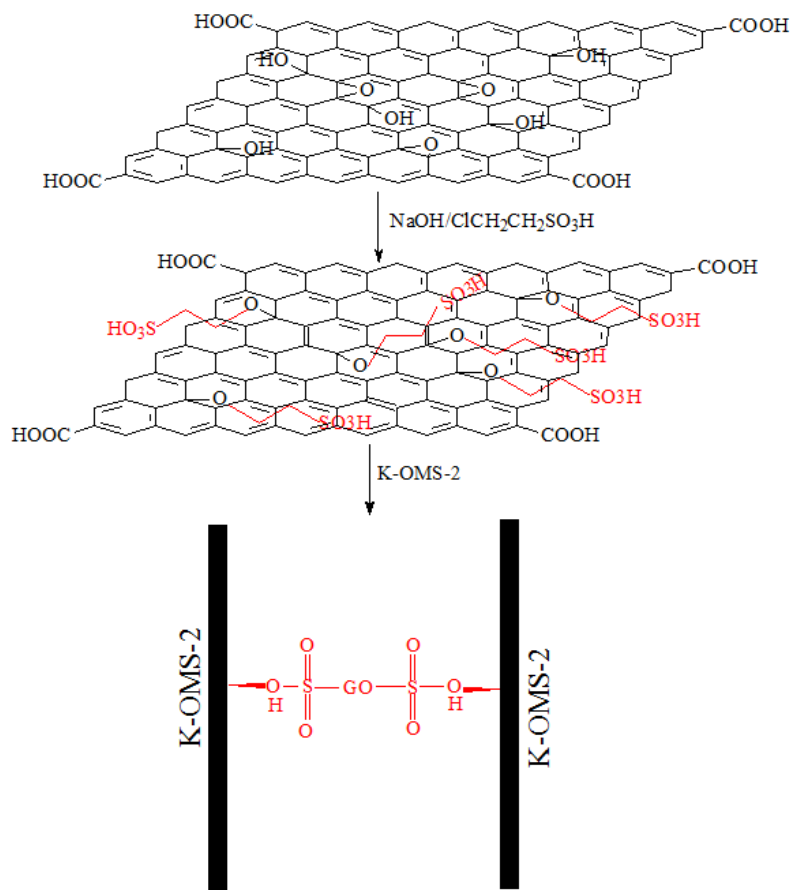
#### **Assembly of K-OMS-2 membrane**

K-OMS-2 membrane was also synthesized via a filtration process identical to that of aforementioned K-OMS-2/GO-SO<sub>3</sub>H membrane, except the using of K-OMS-2 nanowires instead of K-OMS-2/GO-SO<sub>3</sub>H heterjunctions.

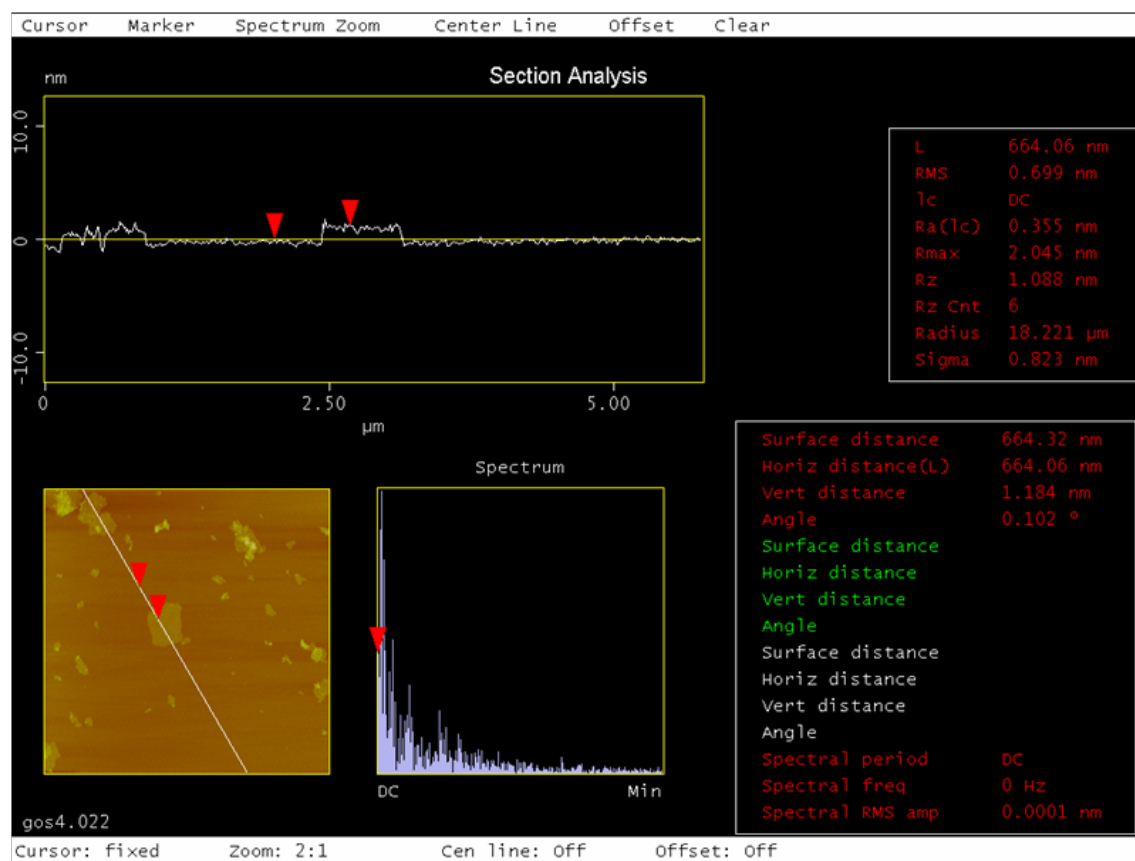
### **Characterization**

The XRD data was collected on a Bruker D8 Advance X-ray diffractometer (Cu K $\alpha$   $\lambda=1.5406$  Å). Morphology of the membrane was studied using a field-emission scanning electron microscope (FESEM) Jeol JSM-6340F (Japan) operated at 5 kV, and transmission electron microscopy (TEM) was carried out on a Jeol JEM-2100F (Japan) operated at 200 kV. Atomic force microscopy (AFM) was carried out using a non-contact mode on a PSIA XE-150 scanning probe microscope. The AFM sample was prepared by spin coating the dispersion water solution of GO onto a Si substrate covered with 300 nm thick SiO<sub>2</sub>. X-ray photoelectron spectroscopy (XPS) analysis was performed in an ultrahigh vacuum chamber, with a base pressure below  $2.66 \times 10^{-7}$  Pa at room temperature. Photoemission spectra were recorded using a Kratos Axis Ultra spectrometer equipped with a standard monochromatic Al K $\alpha$  excitation source ( $h\nu = 1486.71$  eV). Fourier-transform infrared spectroscopy (FTIR) analysis was performed using a Perkin Elmer GX with potassium bromide dye for the preparation of the sample pellet. The pore size of the synthesized membrane was measured using standard polystyrene microspheres.<sup>4</sup> The PS microspheres of diameters 0.05, 0.1, 0.2, 0.5, and 1  $\mu\text{m}$ , were purchased from Alfa Aesar. The concentration of PS microspheres solution was prepared to 0.033 wt %, and a lab-scale dead-end filtration setup was used for filtration.

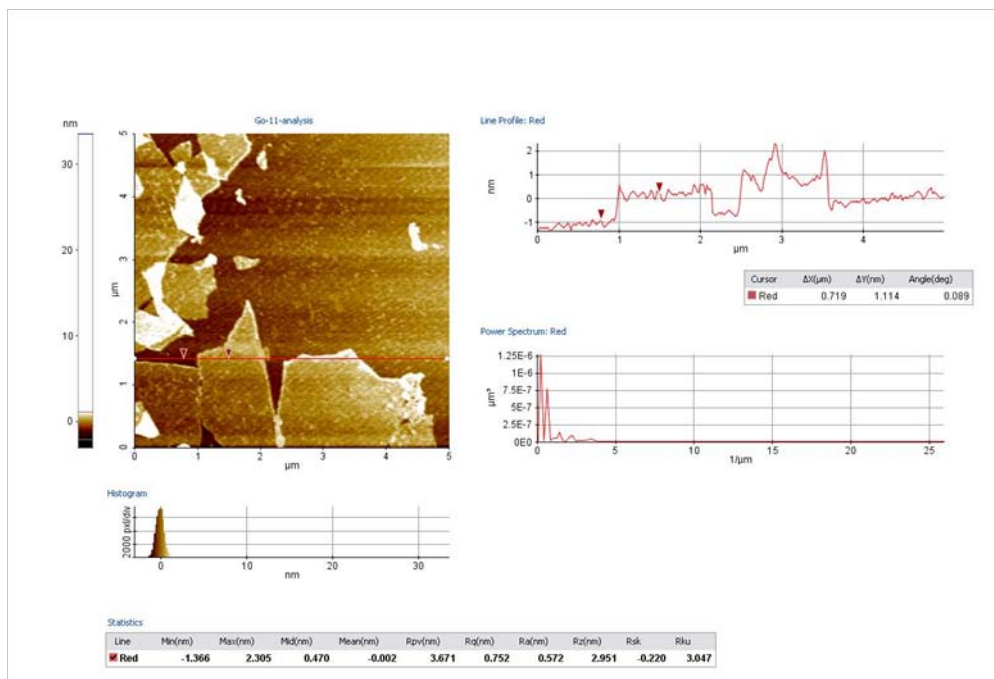
- 1 W. S. Hummers Jr and R. E. Offeman, *J. Am. Chem. Soc.*, 1958, **80**, 1339.
- 2 J. Liu, H. Jeong, K. Lee, J. Y. Park, Y. H. Ahn and S. Lee, *Carbon*, 2010, **48**, 2282-2289.
- 3 J. Yuan, K. Laubernds, J. Villegas, S. Gomez and S. L. Suib, *Adv. Mater.*, 2004, **16**, 1729-1732.
- 4 S. Nakao, *J. Membr. Sci.*, 1994, **96**, 131-165.



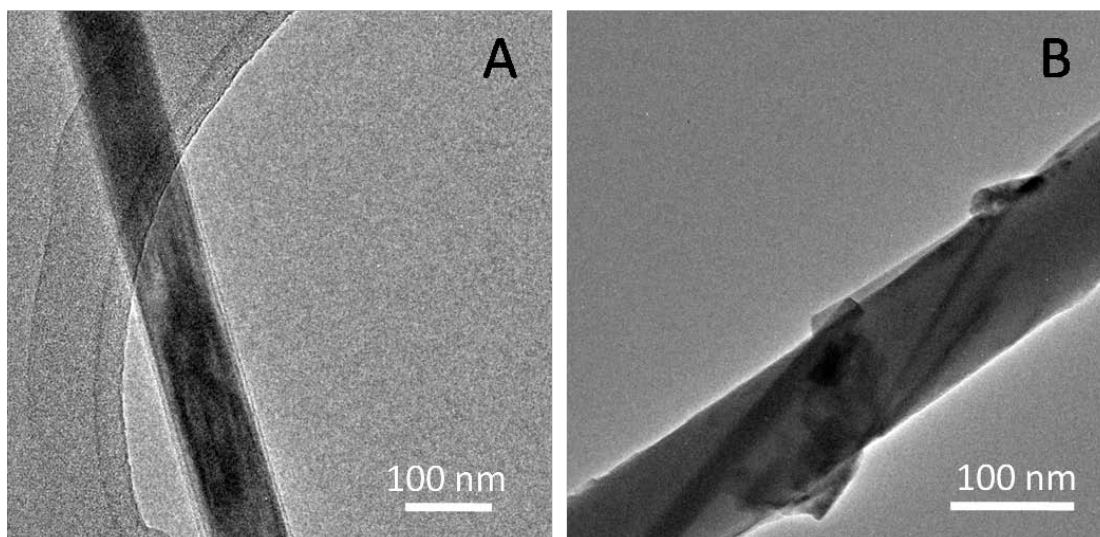
**Fig. S1** Scheme of the formation of K-OMS-2/GO-SO<sub>3</sub>H.



**Fig. S2** AFM image of single-sheet GO-SO<sub>3</sub>H.

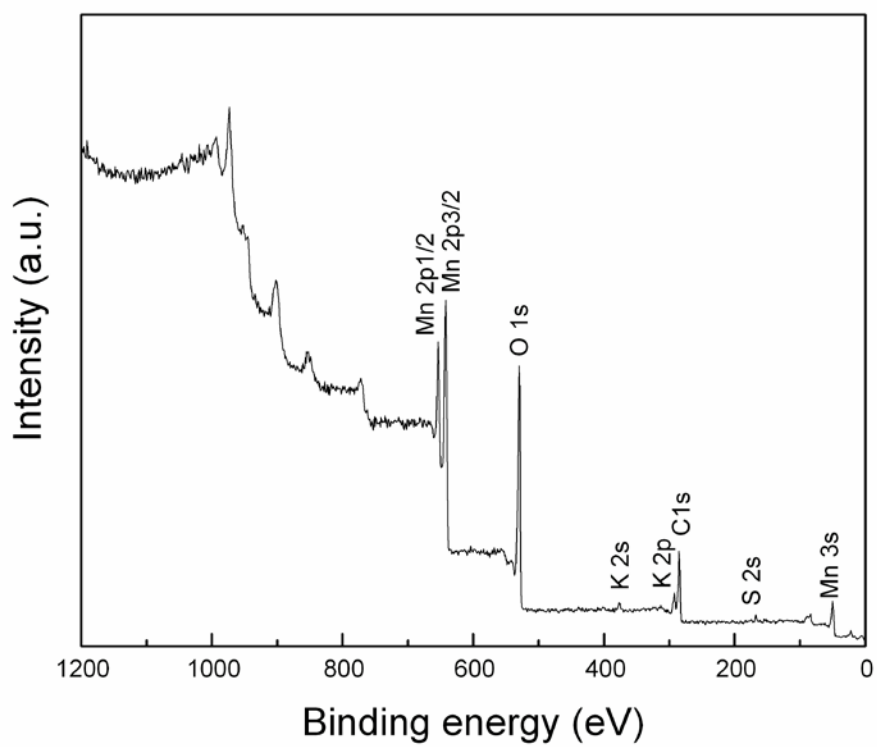


**Fig. S3** AFM image of single-sheet GO.

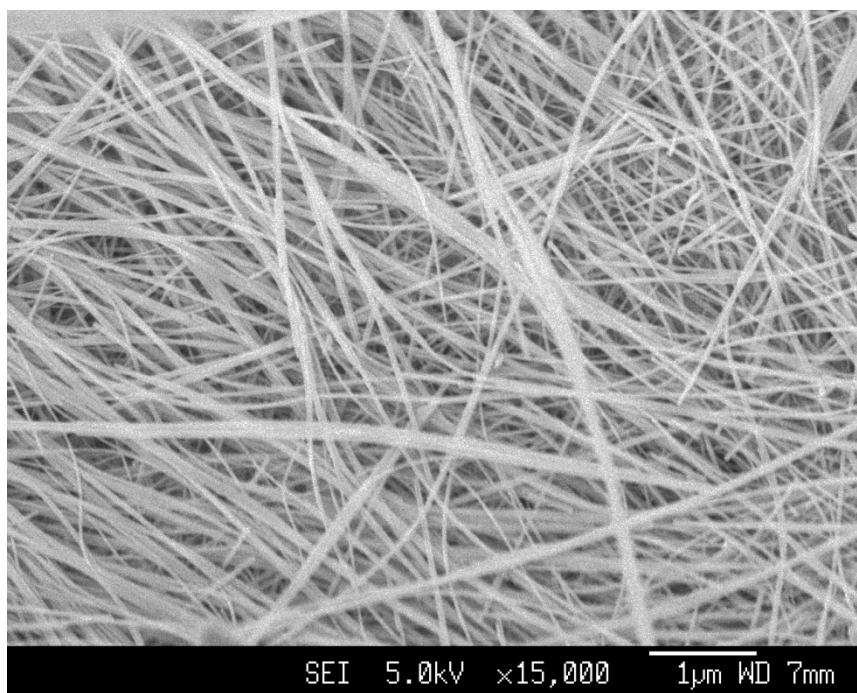


**Fig. S4** (A) TEM image of K-OMS-2/GO under pH 11; (B) TEM image of K-OMS-2/GO-SO<sub>3</sub>H under pH 11.

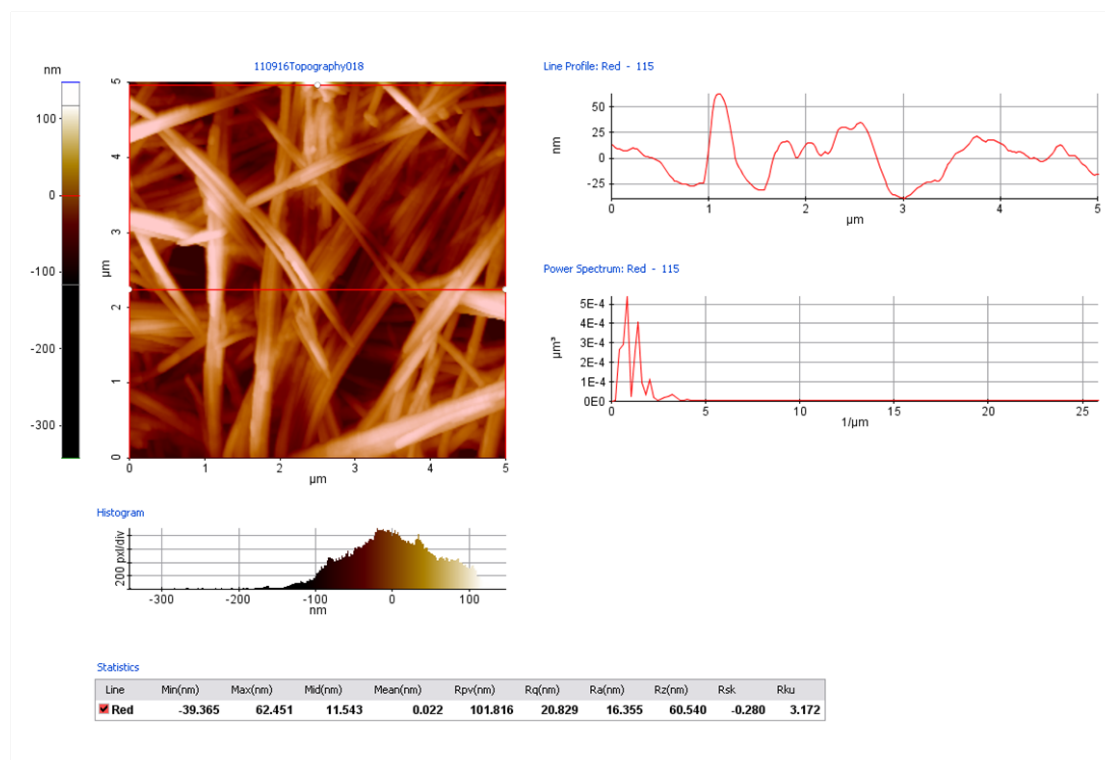




**Fig. S5** XPS survey spectra of the synthesized K-OMS-2/GO-SO<sub>3</sub>H.



**Fig. S6** Top view FESEM image of the K-OMS-2 nanowire membrane.



**Fig.S7** AFM image of the synthesized K-OMS-2/GO-SO<sub>3</sub>H nanofibrous membrane.