

Supporting information for

**Organic-inorganic hybridization for the synthesis of robust *in situ* hydrophobic polypropylsilsesquioxane aerogels with fast oil absorption properties**

Ze Wu, Lei Zhang, Ji Li, Xiaolu Zhao, and Chunhui Yang\*

MIIT Key Laboratory of Critical Materials Technology for New Energy Conversion and Storage, School of chemistry and chemical engineering, Harbin Institute of Technology, Harbin, 150001, China.

Email: kennywu@hit.edu.cn; yangchh@hit.edu.cn

## **1 Experimental methodology**

### **1.1 Fabrication process of SA**

TEOS (0.1 mol), ethanol (0.5 mol), water (0.3 mol), HCl (1M, 0.1 mL) and CTAB ( $5 \times 10^{-3}$  mol) were mixed and hydrolyzed at 90°C for 5 h with continuous stir. The sol (15 mL) was gelled by  $\text{NH}_3 \cdot \text{H}_2\text{O}$  (1 M, 0.5 mL) in an oven at 50°C. Gelation usually took place within 10 min. Obtained gel was aged by ethanol (15 mL) for 1 d and subsequently soaked in hexane (15 mL) for 6 h (three times) at 50°C. Finally, the alcogel was supercritical  $\text{CO}_2$  dried for 3 h (8 MPa, 40°C).

### **1.2 Fabrication process of PSA-7-T**

PTES (0.07 mol) and TEOS (0.03 mol) were pre-mixed under vigorous stirring in a glass bottle. After 5 min of stirring, ethanol (0.5 mol), water (0.3 mol), HCl (1M, 1.0 mL) and CTAB ( $5 \times 10^{-3}$  mol) were added to the mixture and then hydrolyzed at 90°C for 5 h with continuous stir. After hydrolyzation, the sol (15 mL) was transferred to a sealed glass vial and gelled by  $\text{NH}_3 \cdot \text{H}_2\text{O}$  (12.4 M, 0.5 mL) in an oven at 50°C. Gelation usually took place within 5-6 h. Afterwards, the obtained gel was aged by ethanol (15 mL) for 1 d and subsequently soaked in hexane (15 mL) for 6 h (three times) at 50°C. Then, TMCS (0.5 mL dissolved in 14.5 mL hexane) was added to modify the surface of PSA-7. After modification, the modified gel was washed by hexane (15 mL) for three times. Finally, the alcogel was ambient pressure dried (APD) in a gradient of 60, 80, 100 and 120°C and stay at each temperature for 2 h. For comparison, samples without the incorporation of PTES (SA) and TMCS modified PSA-7 (PSA-7-T) were synthesized through a similar process to that of PSA-7 system.

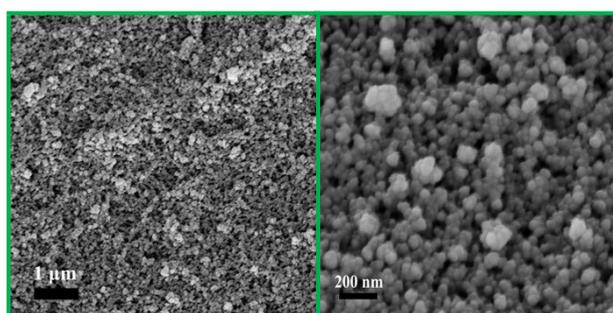
### 1.3 Digital photographs of phase separated precursors



**Figure S1.** Digital photographs of PSA-7 alcogel and phase separation samples with different amounts of PTES (70%, 80% and 90%, from left to right).

## 2 Results and discussion

### 2.1 SEM images of PSA-7-T



**Figure S2.** SEM images of TMCS modified PSA-7.

### 2.2 Compression-strain test of PSA-7



**Figure S3.** Photographs of PSA-7 after 70% compression. Few cracks were observed at the edge and surface (Left graph) of sample after compression. Bottom of the sample can remain smooth (Right graph).

### 2.3 Surface profile of PSA-7

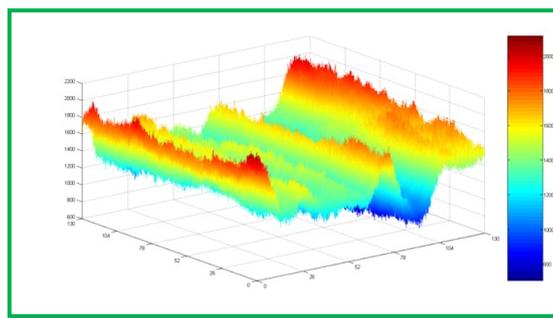


Figure S4. Surface profile of PSA-7 illustrated its rough surface endowed by the microstructure.

### 2.4 Absorption property of reused PSA-7 after 5 times

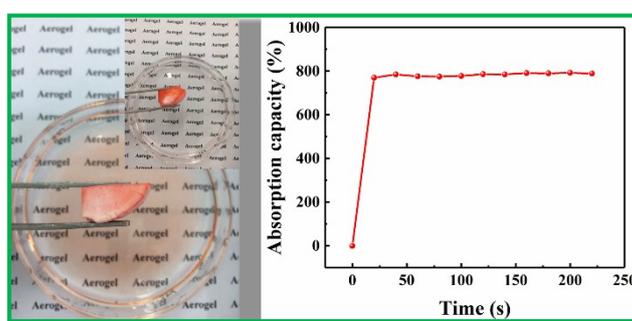


Figure S5. Absorption efficiency of PSA-7 for hexane and Absorption process of reused PSA-7 after 5 times for Sudan III dyed hexane and absorption efficiency for hexane.

### 2.5 $^{29}\text{Si}$ NMR and FTIR spectrums of PSA-7-H

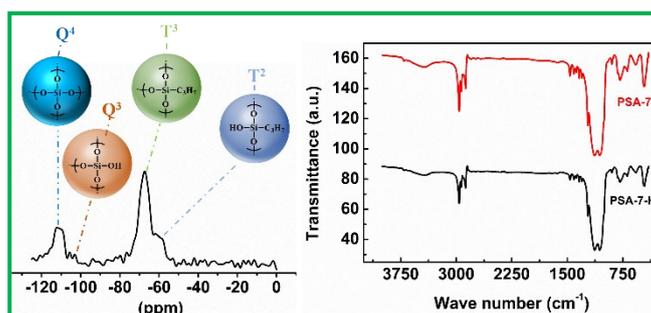


Fig. S6.  $^{29}\text{Si}$  NMR spectrum of PSA-7-H (Left) and FTIR spectrums of PSA-7 and PSA-7-H (Right).