ADVANCED MATERIALS

Supporting Information

for *Adv. Mater.*, DOI: 10.1002/adma. **201200795**

One-Step Interfacial Synthesis and Assembly of Ultrathin Luminescent AuNPs/Silica Membranes

*Shengyang Yang , Chen Zhou , Jinbin Liu , Mengxiao Yu , and Jie Zheng **

Supporting Information for

One-Step Interfacial Synthesis and Assembly of Ultrathin Luminescent AuNPs/Silica Membrane

By Shengyang Yang, Chen Zhou, Jinbin Liu, Mengxiao Yu and Jie Zheng*

Department of Chemistry, The University of Texas at Dallas, 800 W. Campbell Rd. Richardson, TX, 75080 (USA)

Experimental Section

Materials

Hydrogen tetrachloroaurate (III) trihydrate ($HAuCl_4 \cdot 3H_2O$, ACS reagent) and trypan blue were purchased from Acros Organics. (3-Mercaptopropyl)trimethoxysilane (MPTS, 95%) was purchased from Sigma-Aldrich. Toluene (99.9%) was purchased from fisher scientific. All reagents were used without further purification. Hygroscopic paper was purchased from Kimtech Science (Product code: 05511). High-purity water with the resistivity of ~18.2 MΩ*cm was used in the experiments.

Synthesis of Ultrathin Luminescent AuNPs/Silica Membrane

In a typical reaction, $HAuCl_4$ aqueous solution (2 mL, 10 mM) was diluted into 10 mL by adding DI water (8 mL) and added into a three-neck flask (100 mL, containing a stir bar). And then, toluene (10 mL) was added into the flask to form a liquid-liquid interface, followed by carefully adding MPTS (50 μL) into toluene phase. Subsequently, the flask with a water-cooled reflux condenser was put into pre-heated oil-bath at 90ºC for 5.5 h under stirring. After the reaction, the as-prepared ultrathin AuNPs/silica membrane at the toluene/water interface was stored in a closed vial at R.T. for further characterization. For the preparation of

superhydrophobic AuNPs/silica film, the water in the sample was removed and the AuNPs/silica nanocomposites was dispersed in toluene by sonication, followed by drop-casting on the water surface in a dish and allowing toluene to volatilize.

Characterization

Transmission electron microscopy (TEM) images were obtained from JEOL-2100 transmission electron microscope at an accelerating voltage of 200 kV. Fourier transform infrared (FTIR) spectra were recorded on a Nicolet Avatar 360 FT-IR spectrometer. Fluorescence and brightfield images were obtained by an IX-71 inverted fluorescence microscope (Olympus) system under Hg-lamp excitation and a Photon Max 512 CCD camera (Princeton Instrument). Scanning electron microscopy (SEM) images were recorded by a LEO 1530 VP, equipped with a field emission electrode. Steady-state luminescence spectra were acquired with a PTI Quanta Master Model QM-4 scanning spectrofluorometer. The excitation and emission spectra were corrected for the wavelength-dependent lamp intensity and detector response, respectively. Lifetime data were obtained using a xenon arc flash lamp and phosphorescence detector. Liquid nitrogen was used for low temperature PL studies. Atomic force microscopy (AFM) images of dried samples were performed in tapping mode with Park Scientific Instruments (PSI) Autoprobe M5 at R.T. in ambient air conditions using standard Si_3N_4 cantilevers. And, the AFM images were viewed and processed using WSxM v5.0 (free available SPM software, www.nanotec.es).[S1] Contact angle (CA) was detected on CA Goniometer (ramé-hart). Photographic images of the samples were captured by digital camera (S8100, Nikon).

$$Y(t) = \alpha \exp(-t/\tau) \qquad \text{(Eq.S1)}$$

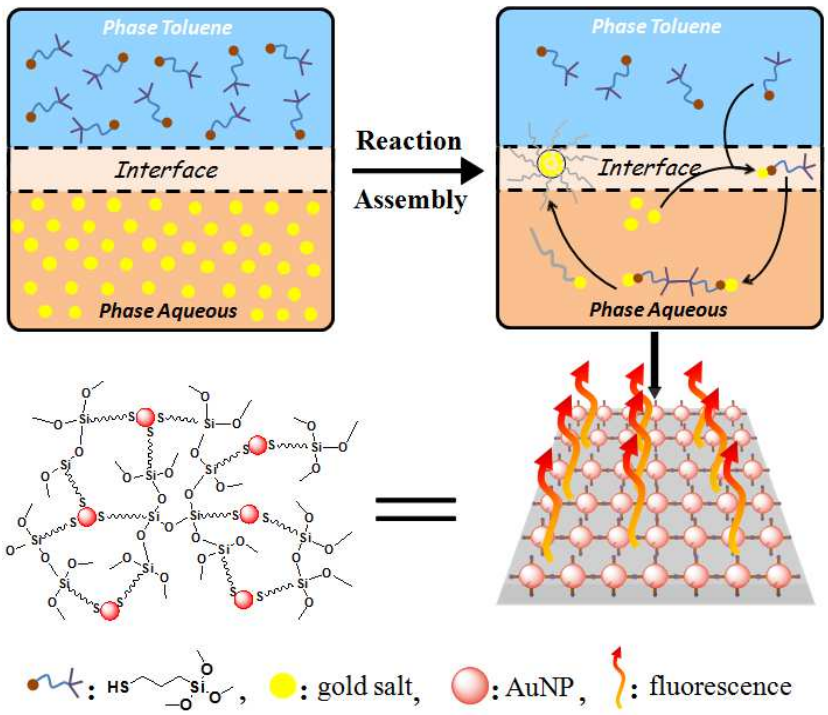

Figure S1. Schematic illustration of the synthesis and assembly of ultrathin luminescent AuNPs/silica membrane at the toluene/water interface.

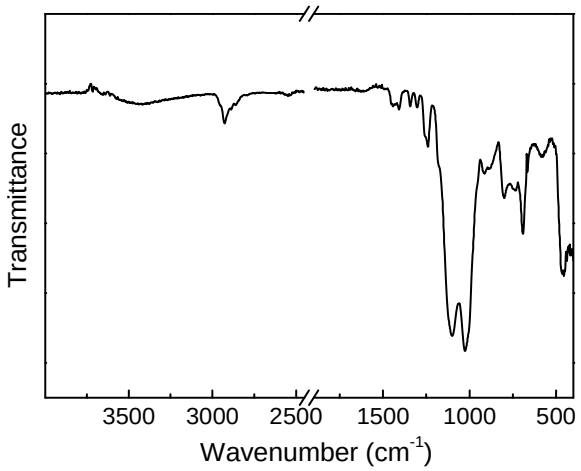

Figure S2. FT-IR spectrum of luminescent AuNPs/silica membrane.

Figure S3. Photograph of the dried AuNPs/silica nanocomposites transferred from toluene/water interface, taken under UV light (365 nm).

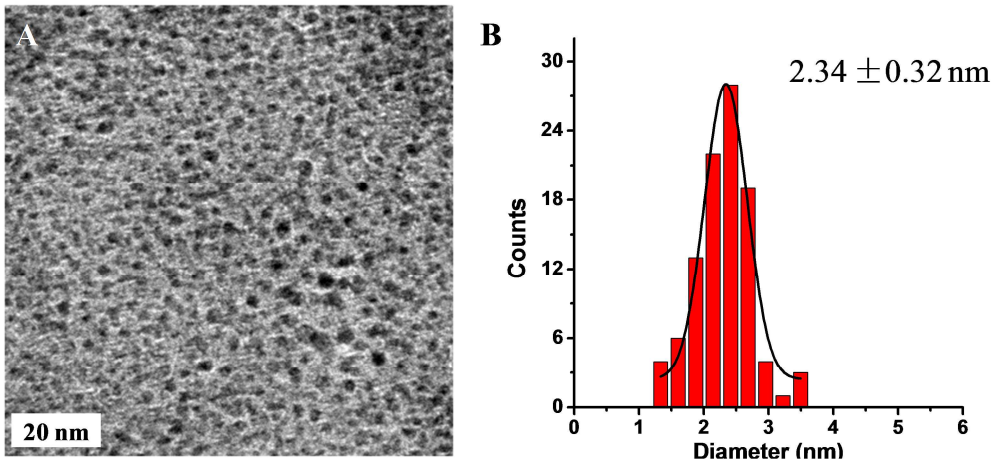

Figure S4. (A) TEM images of luminescent AuNPs in silica matrix formed at toluene/water interface. (B) Diameter distribution of the luminescent AuNPs determined by TEM image of Figure S3A. The data were obtained from software for diameter analysis on picture. (Department of Chemistry, Fudan University, version 1.1.0.33)

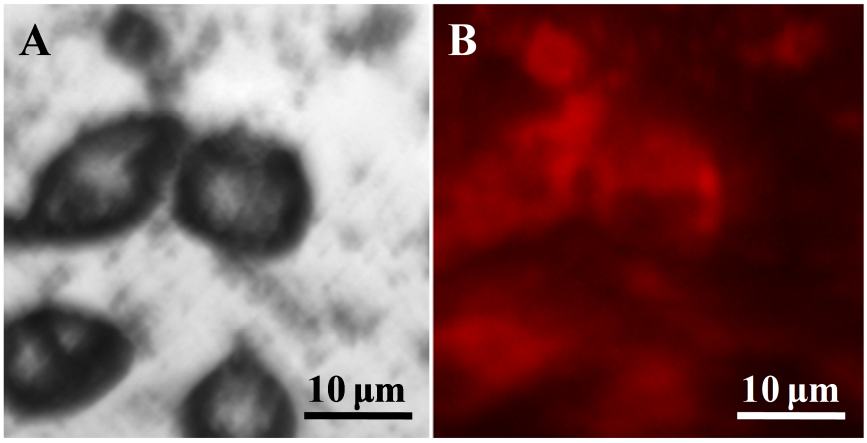

Figure S5. (A, B) Brightfield (A) and fluorescence (B) images of luminescent AuNPs/silica capsules formed on the toluene/water interface by shaking the corresponding membrane.

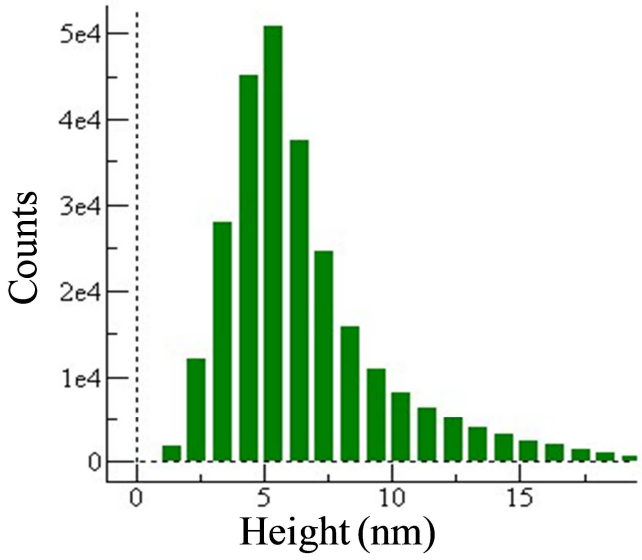

Figure S6. Height distribution of the whole membrane shown in Figure 3E. The analysis of height distribution via WSxM v5.0 software (Nanotec Electronica).[S1]

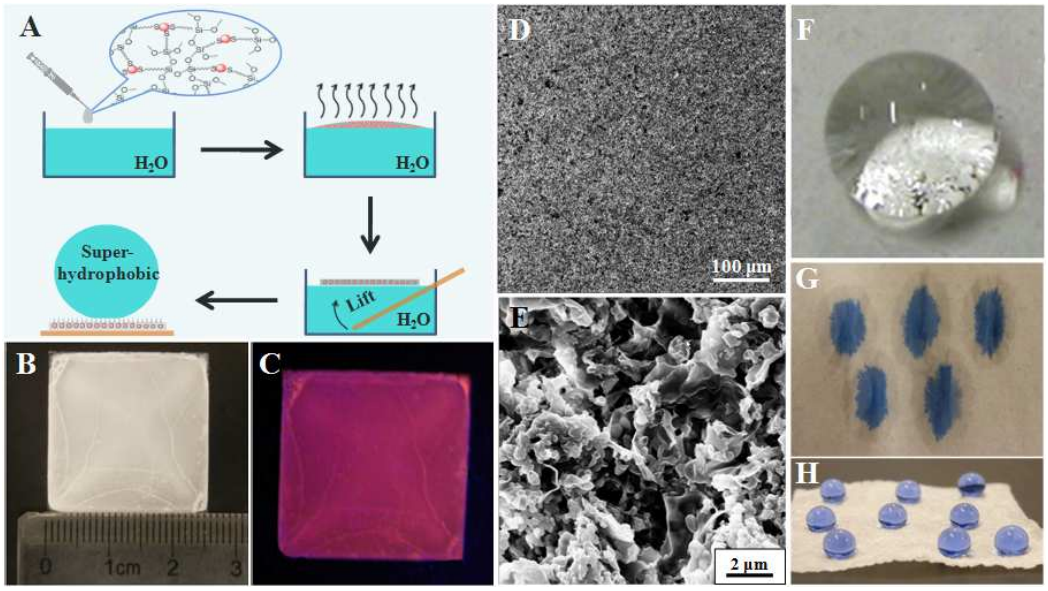

Figure S7. (A) Schematic representation of the procedure for fabricating the luminescent and superhydrophobic film composed of AuNPs/silicananocomposites. (B, C) Photographs of the assembled superhydrophobic AuNPs/silica film taken under daylight (B) and UV light (365 nm, C), respectively. (D, E) Low and high magnification SEM images of superhydrophobic AuNPs/silica film. (F) Profile of a water droplet (8 μL) on the AuNPs/silica film. (G, H) Photographs of water drops (dyed by trypan blue) on the hygroscopic paper before (G) and after (H) the modification with AuNPs/silica nanocomposites.

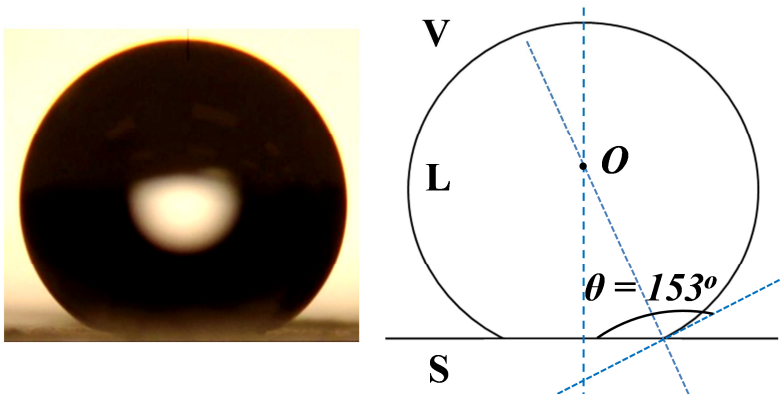

Figure S8. A photograph of a water drop (8 uL) on the on the AuNPs/silica film (left). The profile of the water drop for contact angle analysis using Young–Laplace method.

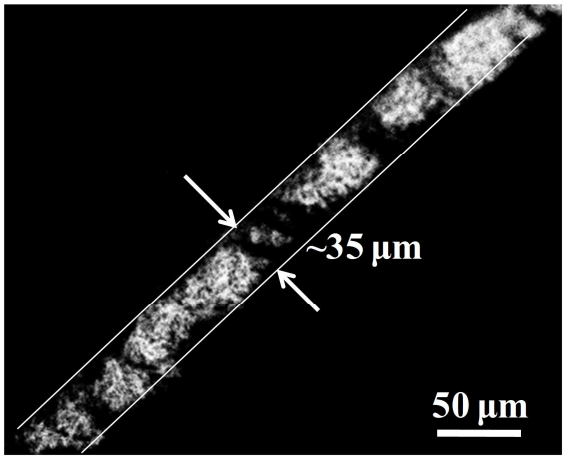

Figure S9. Cross-section SEM image of the luminescent and superhydrophobic film composed of AuNPs/silica nanocomposites.

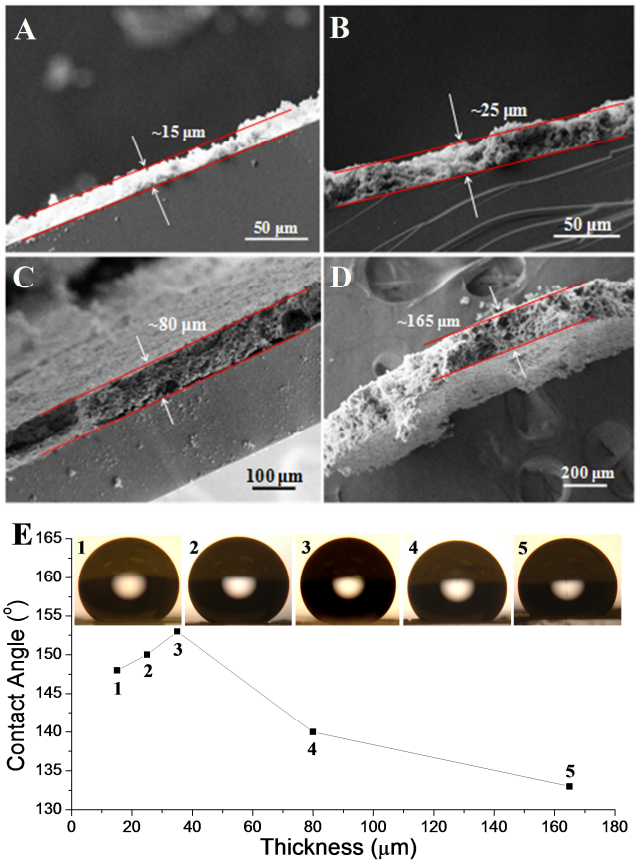

Figure S10. (A-D) Cross-section SEM images of the micro-thickness luminescent AuNPs/silica nanocomposite films with different thickness. (E) The relationship between the film thickness and the water contact angle. Inset: Water drop profiles in contact angle measurement (8 µL droplet size) on the corresponding surfaces.

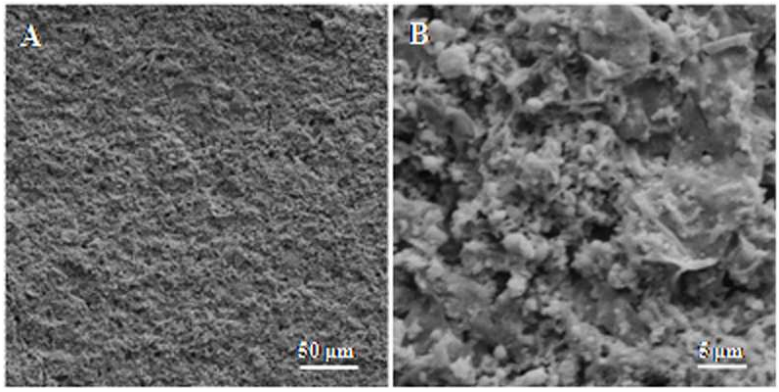

Figure S11. (A, B) SEM images of the luminescent AuNPs/silica nanocomposite film with the thickness of about 165 μm.

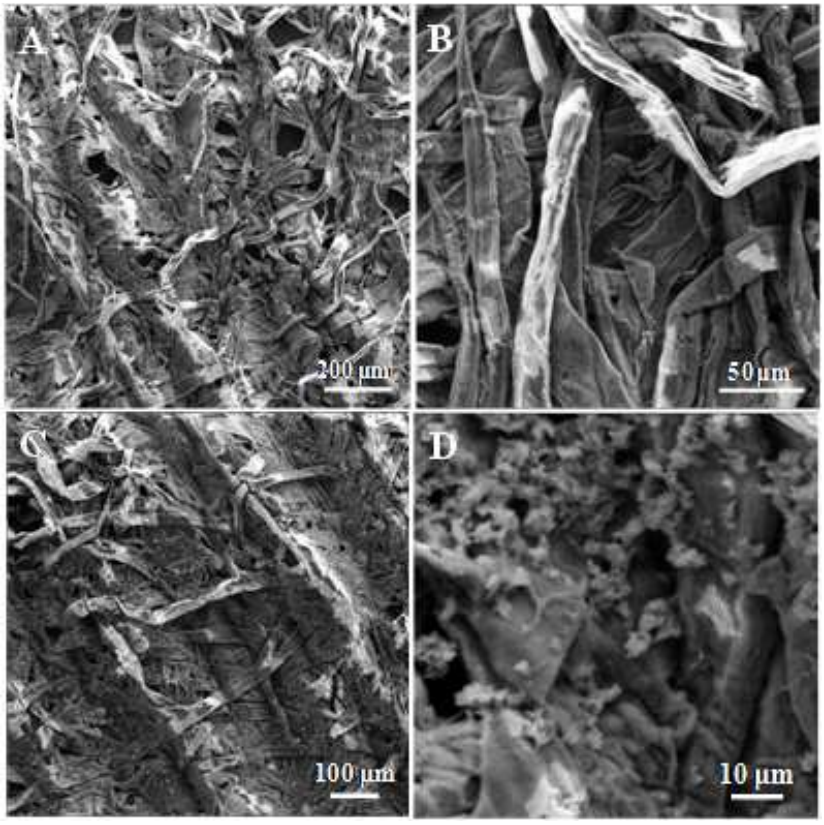

Figure S12. SEM images of hygroscopic paper without (A, B) and with (C, D) AuNPs/silica nanocomposites treatment.

References

[S1] I. Horcas, R. Fernandez, J. M. Gomez-Rodriguez, J. Colchero, J. Gomez-Herrero, A. M. Baro, *Rev. Sci. Instrum.* **2007**, *78*, 013705.