Supplementary Information

Directionally Electrodeposited Gold Nanoparticles into Honeycomb Macropores and their Surface–Enhanced Raman Scattering

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1. Chemicals:

The nanopolyoxometalates crystal Na₆(NH₄)₄[(Mn(H₂O)₃)₂(WO₂)₂(BiW₁₂O₃₃)₂] ({$\text{Mn}_2\text{Bi}_2\text{W}_{20}$}) was synthesized according to the reported method by B. Krebs (J. Am. Chem. Soc. 1998, 120, 7252). Dioctadecyldimethylammonium chloride (C₃₈H₈₀ClN, DODMACl) was purchased from Fluka. AuCl₃·HCl·4H₂O (HAuCl₄, analytical reagent, ≥ 47.8%) and KNO₃ were purchased for use. Rhodamine 6G (R6G) (high purity, 98%) was purchased from Aladdin. Triple distilled water was used to prepare solutions.

2. Experimental Procedures

Electrochemical Properties of Gold–Filled Honeycomb Film: Gold nanoparticles were beforehand electrodeposited on ITO–coated glass with the honeycomb film template. The above prepared ITO–coated glass was the working electrode. Pt plate and saturated calomel electrode (SCE) were counter electrode and reference electrode,
respectively. Pure nitrogen was bubbled for 15 min before measurement to remove the dissolved oxygen. Cyclic voltammetry (CV) was carried out in a 0.1 M KNO₃ aqueous solution from –0.6 V to 1.4 V at the different scan rate at room temperature.

**Morphologies Observation:** The morphologies were observed on a field-emission scanning electron microscope (FE-SEM, HITACHI, S4800) and an atomic force microscopy (AFM, Digital Instruments, NanoScope IIIa) in a contact mode.

![SEM images](image)

**Fig. S1** SEM images (a) of gold nanoparticles electrodeposited on bare ITO-coated glass without the honeycomb film templates, and (b) magnified image of the close-packed surface in (a).