Thiol-Induced *in situ* Synthesis of Bifunctional Au-SH/SO_3H-COP Enables Efficient and Recyclable Alkyne Hydration

Zhenzhong Li,* Haohao Chang, Zhiwei He, Zhongyang Su, Mingjian Duan

College of Chemistry and Chemical Engineering, Henan University of Science and Technology, Luoyang 471023, China

E-mail: zhenzli@haust.edu.cn

1. HRTEM images of Au-SH-COP

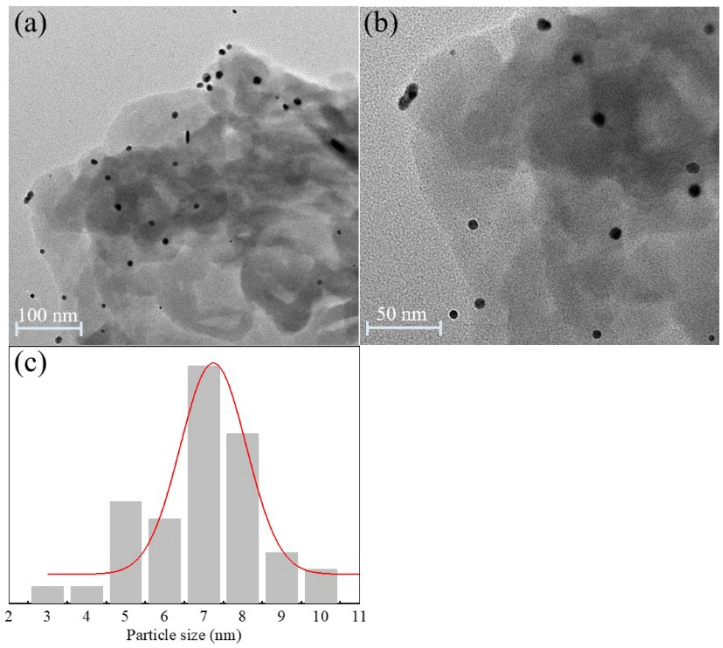

Figure S1. (a, b) HRTEM images of Au-SH-COP, (c) The corresponding Au particle size distribution diagram.

2. Mapping images of Au-SH/SO_3H-COP

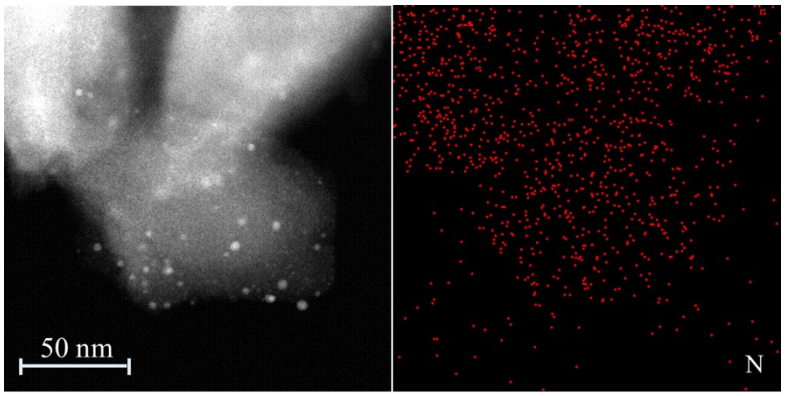

Figure S2. TEM images and the corresponding elemental mapping images of O (red).

3. Quantitative analysis of SH and SO_3H moieties

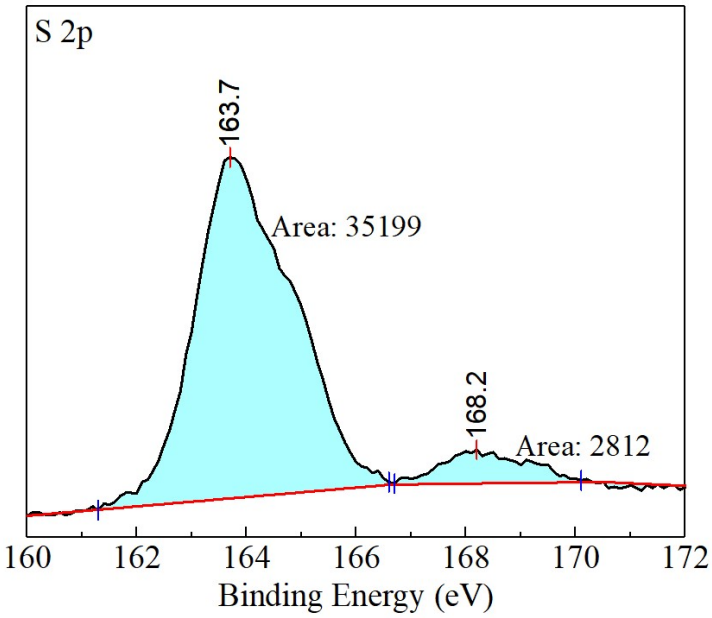

Figure S3 shows the S 2p XPS spectra of Au-SH/SO_3H-COP. Based on a quantitative analysis of the respective peak areas, the HS/SO_3H molar ratio was determined to be 13:1. The S content in Au-SH/SO_3H-COP, which was determined to be 4.1 mmol/g. therefore, the SO_3H content was estimated to be approximately 0.29 mmol/g. ICP analysis revealed the Au content of the material was 0.56 mmol/, corresponding to a molar Au/SO_3H ratio of approximately 2:1.

4. S 2p XPS spectra

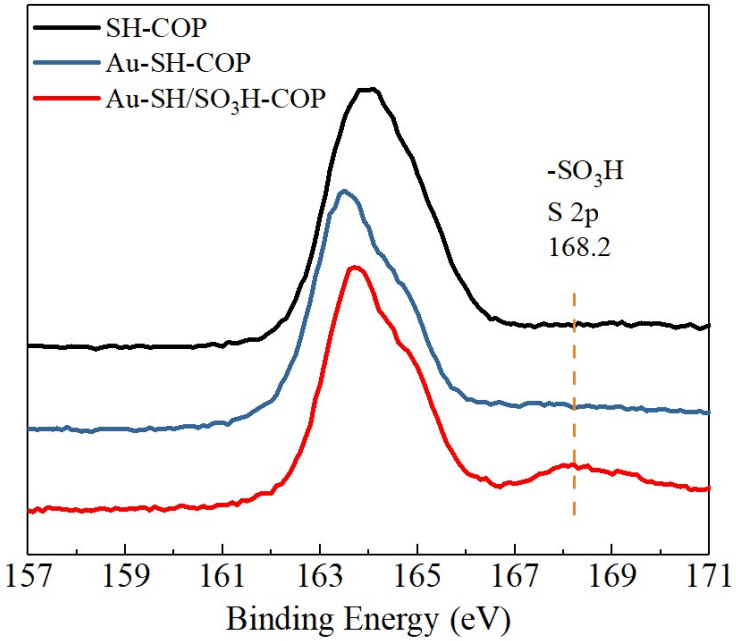

Figure S4. XPS spectra of SH-COP, Au-SH-COP and Au-SH/SO_3H-COP at the S 2p level.

5. XPS spectra of Au-SH/SO_3H-COP after six reaction cycles

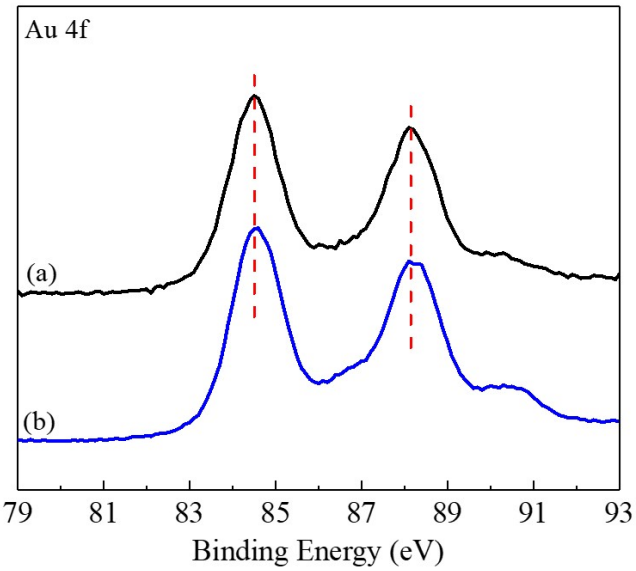

Figure S5. XPS spectra of (a) freshly prepared Au-SH/SO_3H-COP and (b) Au-SH/SO_3H-COP after six reaction cycles.

6. ICP-OES analysis

After the catalytic reaction, the Au-SH/SO_3H-COP catalyst was recovered by filtration and washed with methanol at room temperature. The combined filtrate was collected to quantify Au leaching by ICP-OES. Specifically, the methanol was evaporated, and the residual was dissolved in 5 mL aqua regia at 50 ℃ for 2 h. The resulting solution was then diluted to 25 mL with deionized water for ICP analysis. The measured Au concentration was below 0.1 ppm after the first reaction cycle, indicating negligible leaching from the catalyst.