Facile morphology control of gold(0) structures from aurophilic assemblies

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Supporting Information

Experimental Section

General procedures

All manipulations were performed under prepurified N_2 using standard Schlenk techniques. All solvents were distilled from appropriated drying agents. Literature methods were used to prepare Ag nanoparticles,[1] [Au(C≡C-C_5H_4N-CH_3)(PTA)]OTf,[2] [Au(C≡C-C_5H_4N-CH_3)(PTA)]I[2] and [Au(C≡C-C_5H_4N)(PTA)].[3]

Physical measurements

Infrared spectra were recorded on a FT-IR 520 Nicolet Spectrophotometer. ^{1}H-NMR (δ(TMS) = 0.0 ppm), ^{31}P{^{1}H}-NMR (δ(85% H_3PO_4) = 0.0 ppm) spectra were obtained on a Varian Mercury 400, Bruker 400 and Bruker DMX 500. Absorption spectra were carried out on a Varian Cary 100 Bio UV- spectrophotometer. Scanning electron microscopy (SEM) was carried out at 5 kV using a Neon40 Crossbeam Station (Zeiss) equipped with a field emission gun and an energy-dispersive X-ray detector (EDX). Transmission Electron Microscopy (TEM) were examined by a Tecnai™ Spirit TEM working at 80 kV. All the samples were prepared for observation by dilution in water, allowed by sonication, deposition and pouring on carbon covered copper TEM grids.

Synthesis of gold structures from [Au(C≡C-C_5H_4N)(PTA)] (A). 2 mL of a 20 mM solution of [Au(C≡C-C_5H_4N)(PTA)] in water was maintained under stirring for 15 minutes at room temperature. Then, 2 μL of a suspension of 100 mM Ag nanoparticles in water were added to the previous sample. 2 mL of acetone were added to the reaction mixture after maintaining it under heating at 60 ºC for 100 h. The resulting structures were collected after sonication for 5 seconds and centrifugation for 5 min at 3900 rpm.

Synthesis of gold structures from [Au(C≡C-C_5H_4N-CH_3)(PTA)]OTf (B)

Similar procedure was followed for the synthesis of **A** but using [Au(C≡C-C_5H_4N-CH_3)(PTA)]OTf [Au(C≡C-C_5H_4N-CH_3)(PTA)]I instead of [Au(C≡C-C_5H_4N)(PTA)].

Synthesis of gold structures from [Au(C≡C-C_5H_4N-CH_3)(PTA)]I (C)

Similar procedure was followed for the synthesis of **A** but using [Au(C≡C-C_5H_4N-CH_3)(PTA)]I [Au(C≡C-C_5H_4N)(PTA)] instead of [Au(C≡C-C_5H_4N)(PTA)].

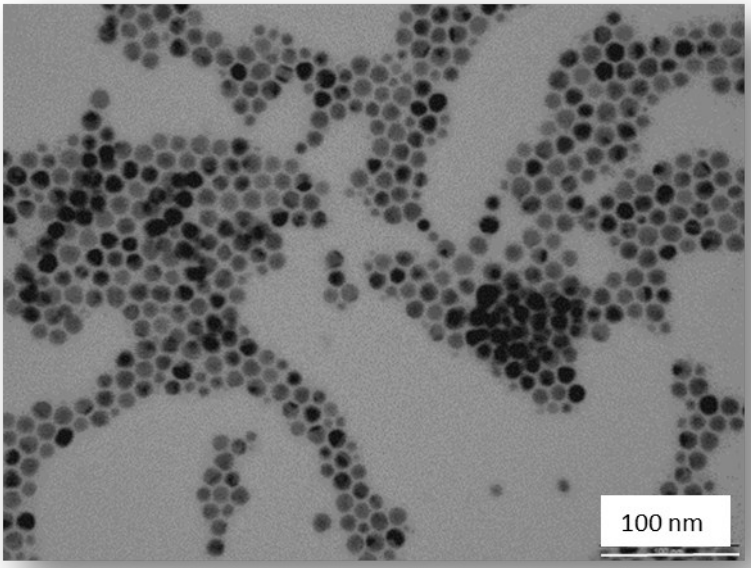

Figure S1. TEM image of the synthesized silver nanoparticles.

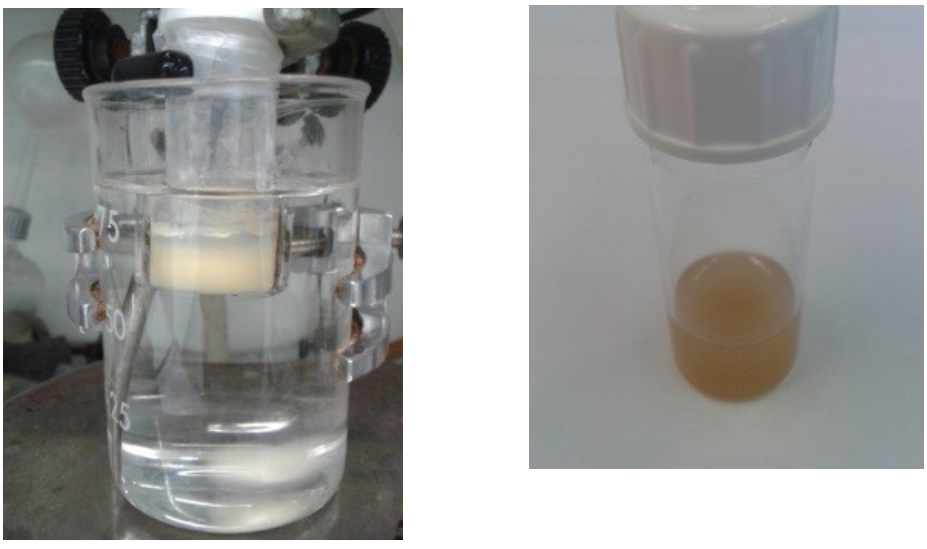

Figure S2. Images of the initial aqueous solution of **A** (left) and after reaction with Ag NPs for 100 h of stirring at 60 ºC (right).

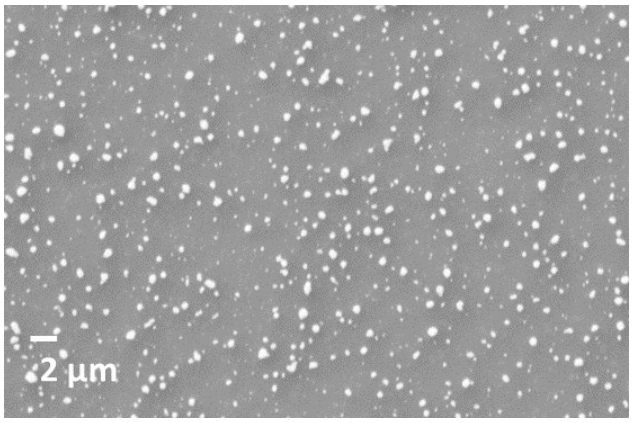

Figure S3. SEM image of the Au micellar microstructures derived from the reduction of **C** (extended view).

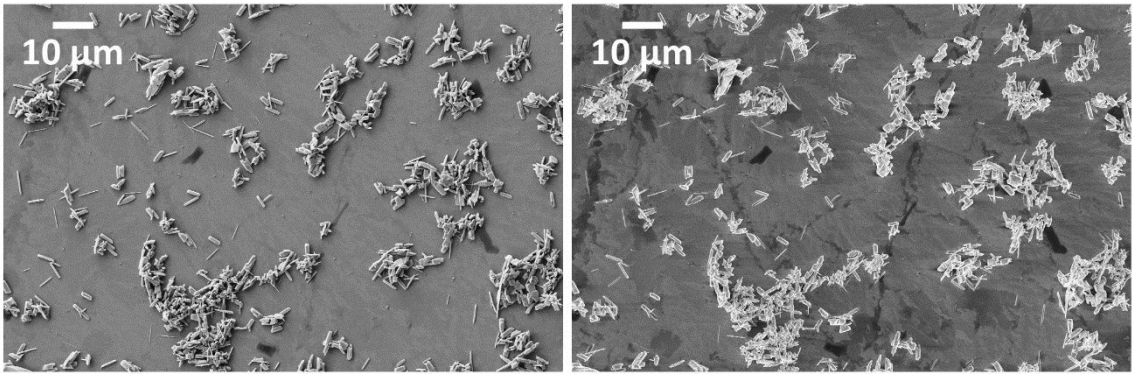

Figure S4. SEM image of the Au rod microstructures derived from the reduction of **A**. Left (secondary electron detector) and right (backscattered electron detector).

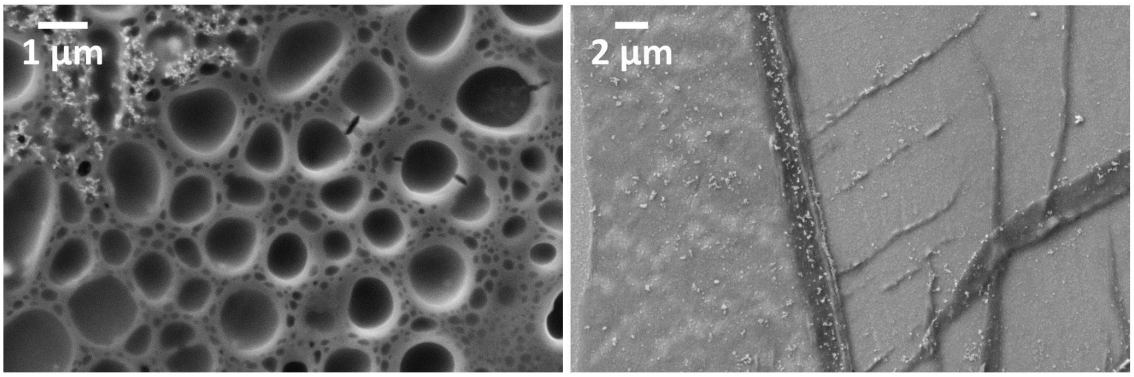

Figure S5. SEM characterization of the reaction of **A** with Ag NPs in a 1:1 ratio (left) and 1:10 ratio (right).

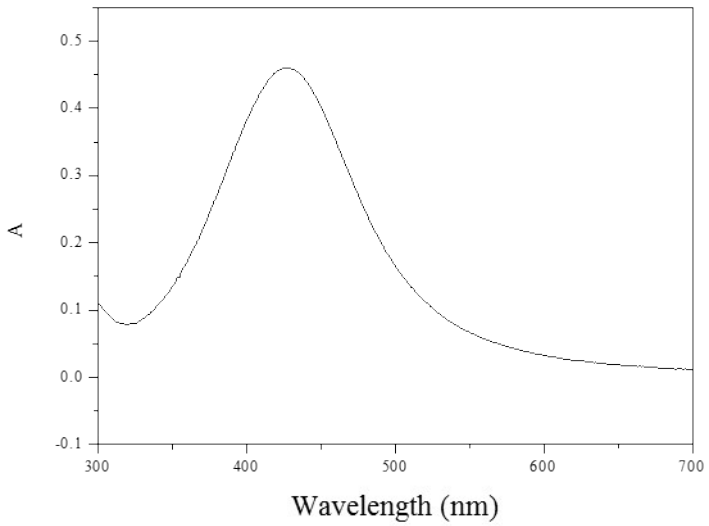

Figure S6. UV-vis spectrum of the synthesized Ag(0) nanoparticles in water.

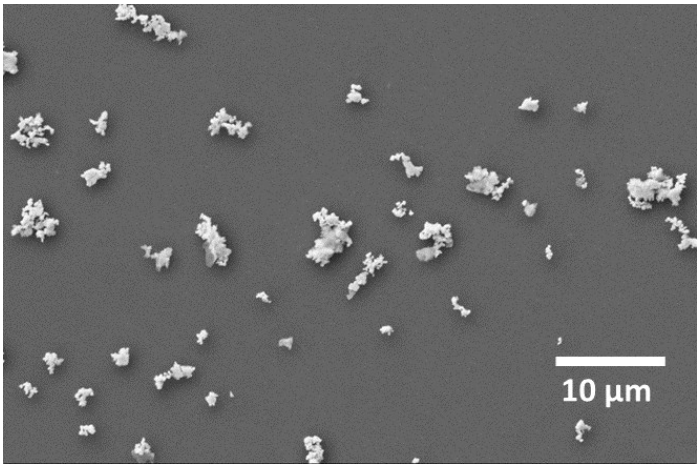

Figure S7. SEM characterization of the reaction of **A** with Ag NPs in 200:1 ratio in chloroform (extended view).

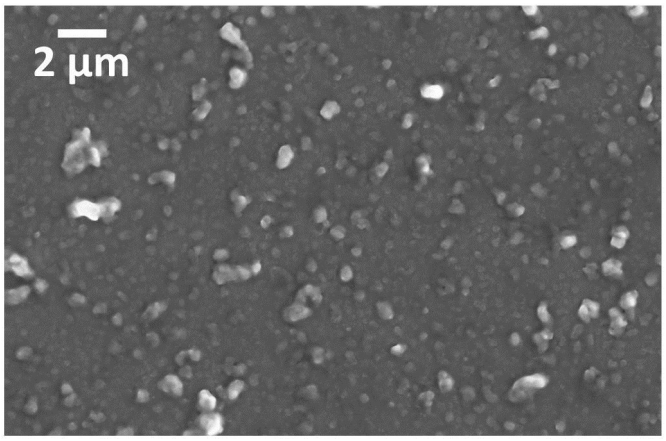

Figure S8. SEM images of the Au(0) spherical nanostructures derived from the reduction of **A** in methanol.

References

1 L.-Q. Zheng, X.-D. Yu, J.-J. Xu and H.-Y. Chen, *Talanta*, 2014, **118**, 90–95.

2 E. Aguiló, R. Gavara, C. Baucells, M. Guitart, J. C. Lima, J. Llorca and L. Rodríguez, *Dalton Trans.* 2016, **45**, 7328–7339.

3 R. Gavara, J. Llorca, J. C. Lima and L. Rodríguez, *Chem. Commun.*, 2013, **49**, 72–74.