Supplementary Material (ESI) for Chemical Communications

Supporting Information

Facile controlled preparation of gold nanoparticles with amphiphilic thiacalix[4]arene as reductant and stabilizer

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Part I: Experimental details

SI-1. Materials

Poly(ethylene glycol) monomethyl ether (MPEG550, Mn=550) came from Fluka Chemical Co.. p-Toluenesulfonyl chloride (99%) was purchased from ACROS Chemical Co.. Potassium carbonate, cesium carbonate, and all the solvents were purchased from Sinopharm Chemical Reagent Co., Ltd.

SI-2. Preparation of SCa-MPEG550 and SCa-TMPEG550

$CsCO_3$ (868 mg, 4.5 mmol) was added into a suspension of p-tert-butyl thiacalix[4]arene[1] (1.45 g, 2 mmol) in dry acetonitrile (150 ml). After the mixture was stirred at room temperature for 30 minutes, an acetonitrile solution (30 ml) of mPEG550-OTs[2] (2.70 g, 4 mmol) was added by drops during one hour. Then the solution was refluxed at 80 ^oC for another three days under N_2 atmosphere. The resulting yellow solution was allowed to rotation to remove the solvent, and the remains were re-dissolved in 50 ml CH_2Cl_2 and then washed with 20 ml deionized water for three times. The organic phase was collected and evaporated under vacuum. The crude products were purified by silica column chromatography with eluent (CH_2Cl_2:CH_3OH=35:1). The yellow pasty solid SCa-MPEG550 (1.46 g) and SCa-TMPEG550 (0.65 g) were obtained respectively.

Part II: Test conditions and result analysis

SII-1. Nuclear magnetic resonance (NMR)

The ^1H NMR and ^{13}C NMR spectra were recorded using Varian MERCURY plus-400 spectrometer with $CDCl_3$ or dimethyl sulfoxide-d_6 (DMSO-d_6) as solvent at 298 K.

SCa-MPEG550: ^1H NMR (400 MHz, $CDCl_3$, 298 K) δ=: 9.41 (s, 1H, Ar-OH), 9.16 (s, 2H, Ar-OH), 7.64 (s, 2H, ArH), 7.60 (s, 4H, ArH), 7.56 (s, 2H, ArH), 4.60 (t, 2H, J=4.70, ArO-CH_2), 4.18 (t, 2H, J=4.70, $ArOCH_2$-CH_2-O), 3.90 (t, 2H, J=4.70, $ArOCH_2CH_2O$-), 3.85 (t, 2H, J=4.70, $ArOCH_2CH_2O$-), 3.60-3.73 (br, 46H, -CH_2CH_2-O), 3.54 (t, 2H, J=4.70, CH_2-OCH_3), 3.77 (s, 3H, OCH_3), 1.22 (s, 9H, $ArC(CH_3)_3$), 1.21 (s, 18H, $ArC(CH_3)_3$), 1.18 (s, 9H, Ar-$C(CH_3)_3$). ^{13}C NMR (400 MHz, DMSO-d_6, 298 K) δ=: 155.67, 155.41, 155.17, 148.57, 142.71, 132.04, 131.57, 129.31, 120.70, 120.29, 119.17, 71.95, 70.46, 70.26, 70.00, 58.73, 34.99, 34.56, 34.48, 31.84, 31.65, 31.42.

In order to obtain a strong quinone signal, excess chloroaurate acid was used to prepare SCaQ-MPEG550 sample.

SCaQ-MPEG550: ^1H NMR (400 MHz, $CDCl_3$, 298 K) δ=: 8.08 (Ar-OH), 7.40 (ArH), 6.71, 6.57, 6.45 (quinone), 3.70-3.51 (PEG), 3.37 (PEG-OCH_3), 1.31-1.25 ($C(CH_3)_3$).

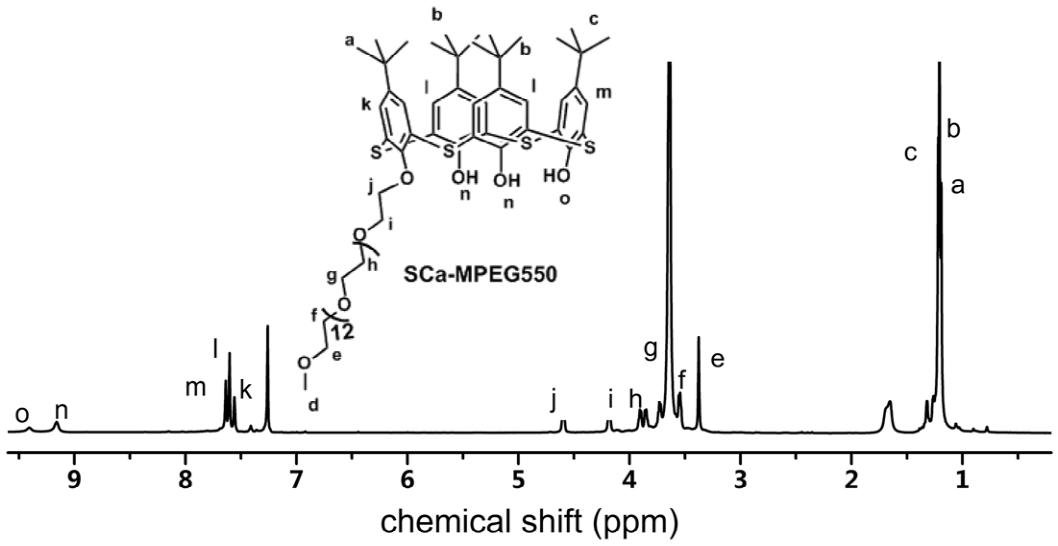

Figure S1. ^{1}H NMR spectrum of SCa-MPEG550 (400 MHz, in $CDCl_3$, 298 K).

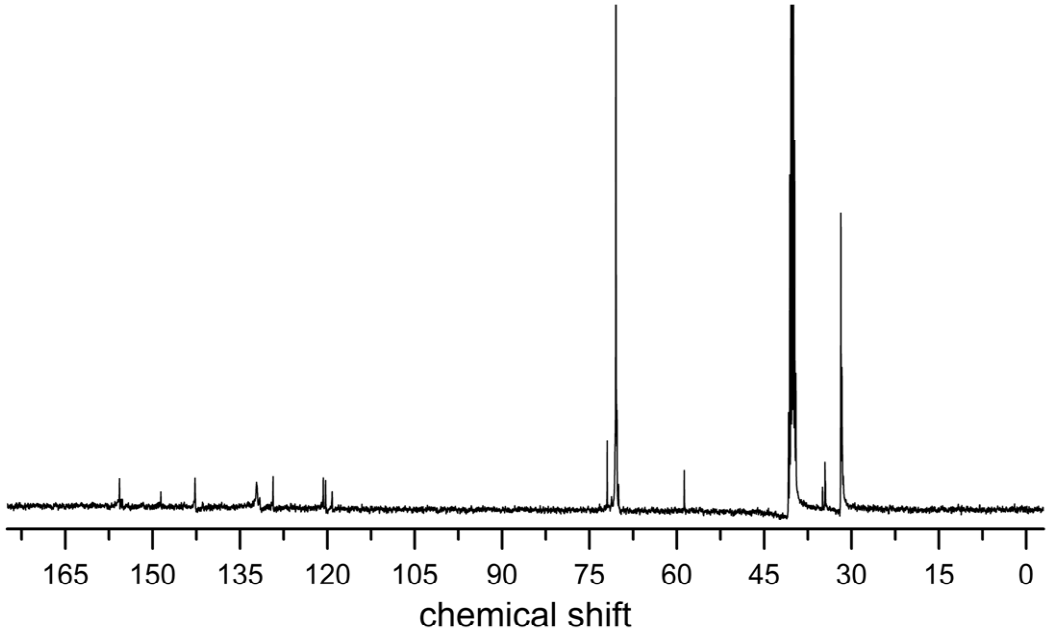

Figure S2. ^{13}C NMR spectrum of SCa-MPEG550 (400 MHz, in DMSO-d_6, 298 K).

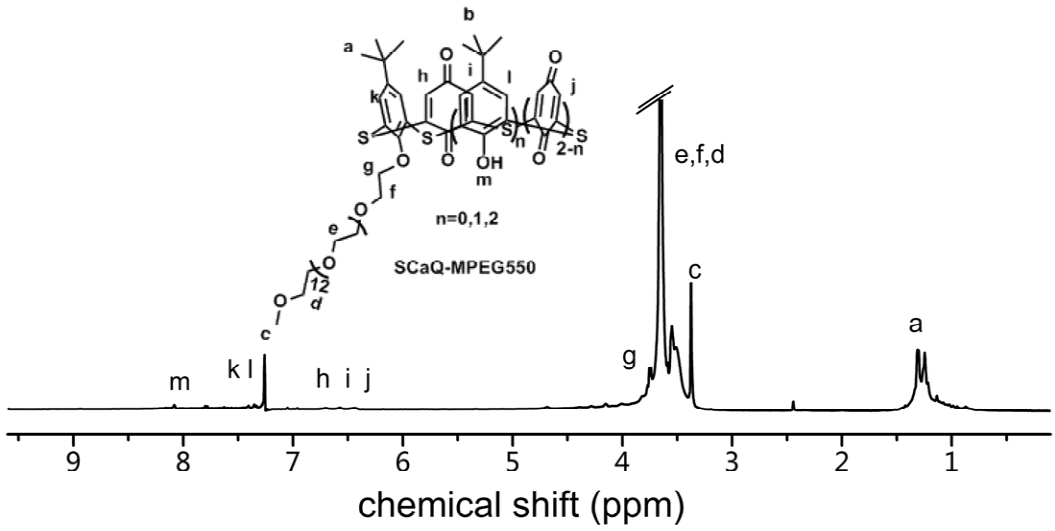

Figure S3. ^{1}H NMR spectrum of SCaQ-MPEG550 (400 MHz, in $CDCl_3$, 298 K).

SII-2. Fourier transform infrared spectroscopy (FTIR)

FTIR spectra were measured as KBr pellets on a Perkin Elmer Paragon 1000 spectrophotometer in the range of 4000-400 cm^{-1}.

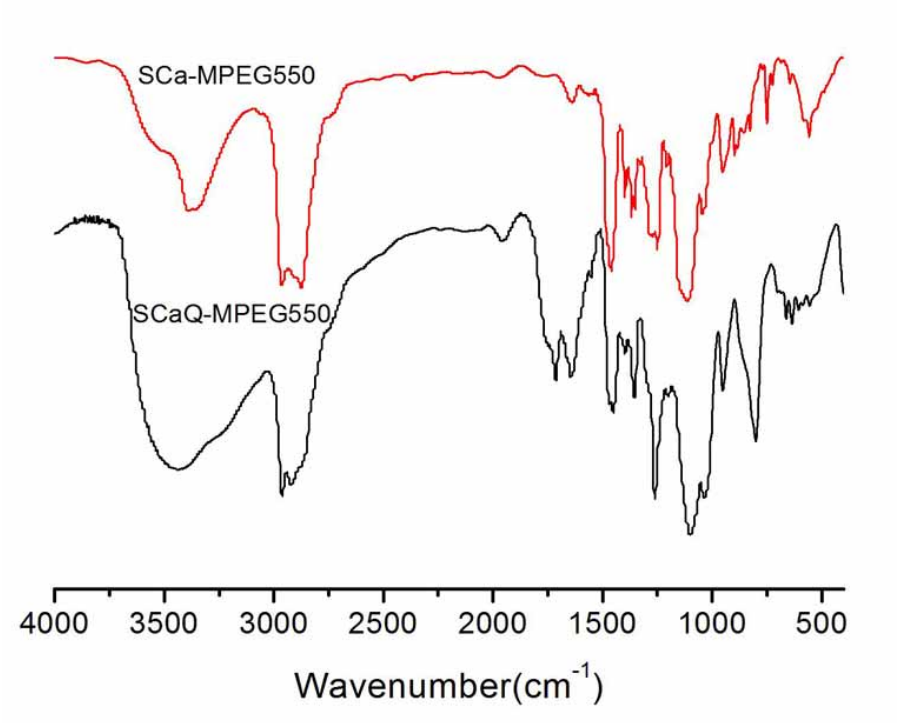

Figure S4. The infrared spectra of SCa-MPEG550 and SCaQ-MPEG550.

SII-3. UV-Vis spectra

The samples A-E were measured on the Perkin Elmer Lambda 20 UV-Vis spectrometer in the range of 350-700 nm.

SII-4. Dynamic light scattering (DLS)

The size distribution of the samples A-D was measured on Zetasizer Nano S photon correlation spectroscopy.

SII-5. High resolution transmission electronic microscopy (HRTEM)

The morphologies of samples A-D were observed at JEOL JEM-2010 transmission electron microscope, with an accelerating voltage of 200 kV.

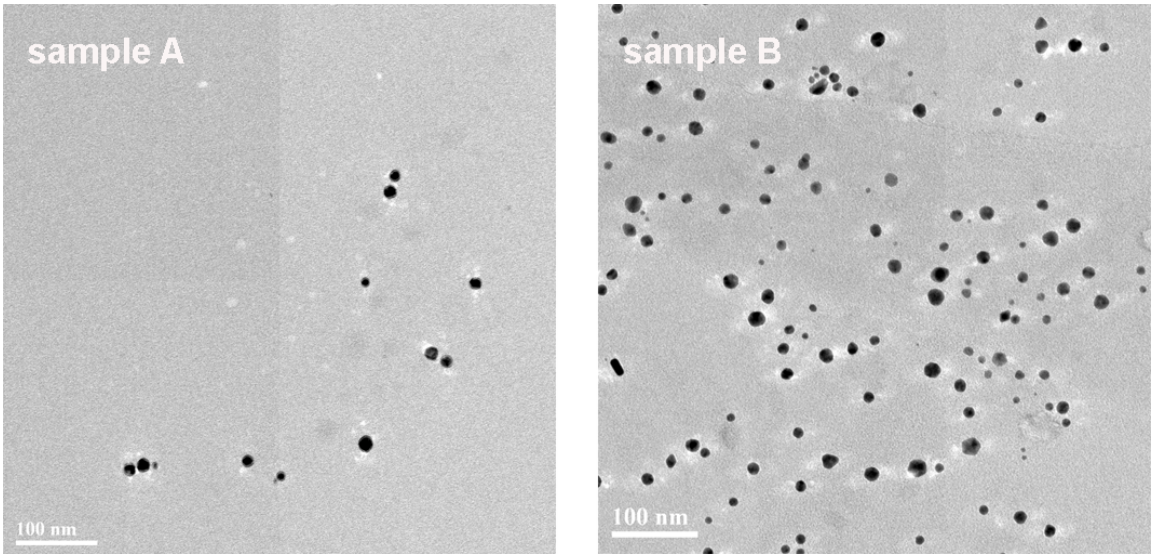

Figure S5. TEM images of samples A and sample B.

Part III: References

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