

Figure S1. ^1H NMR spectrum of 2-bromoisobutyrate ethyl disulfide. The ^1H NMR spectrum was recorded in deuterated chloroform.

2-Bromoisobutyrate ethyl disulfide was prepared by a reaction of 2-bromoisobutyryl bromide and 2-hydroxyethyl disulfide in dry THF. The signals of 2-bromoisobutyrate ethyl disulfide (Figure S1) at 4.46, 2.96, and 1.94 ppm represent the methylene protons next to the ester group (a), the methylene protons next to the sulfur group (b), and the methyl protons (peak c), respectively. The integral ratio of a:b:c is 4:4:12, which indicates the successful synthesis of 2-bromoisobutyrate ethyl disulfide.

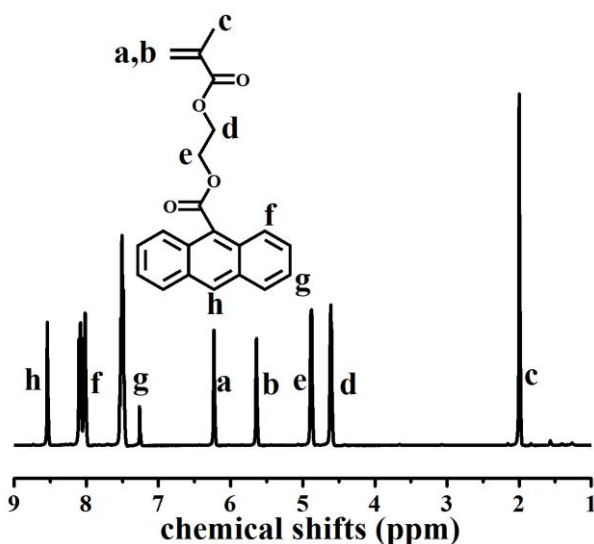


Figure S2. ^1H NMR spectrum of monomer AnMA. The ^1H NMR spectrum was recorded in deuterated chloroform.

AnMA monomer was synthesized by Mitsunobu reaction between anthracene-9-carboxylic acid and HEMA. The signals of AnMA (Figure S2) at 5.64, 6.23, and 2.00 ppm represent the protons on the double bond (a, b) and methyl protons next to the double bond (c). The signals at 4.61 and 4.88 ppm represent the two methylene protons between two ester groups (d, e). The signals at 7.51, 8.05 and 8.54 ppm represent the protons on anthracene groups (f, g, h). The integral ratio of a:b:c:d:e:f:g:h is 1:1:3:2:2:4:4:1, which indicates the successful synthesis of AnMA.

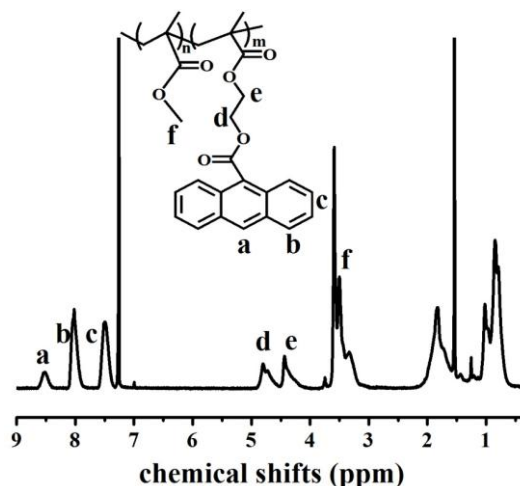


Figure S3. ^1H NMR of *DS*-poly(AnMA-*co*-MMA) initiated by 2-bromoisobutyrate ethyl disulfide. The ^1H NMR spectrum was recorded in deuterated chloroform.

DS-poly(AnMA-*co*-MMA) was synthesized by ATRP of AnMA and MMA. ^1H NMR spectrum of the polymer is shown in Figure S3. The signals at 7.48, 8.02 and 8.53 ppm represent protons on anthracene groups (peak a, b, c). The signals at 2.95 and 3.50 ppm represent the methylene protons next to sulfur group (peak g), the methyl protons of PMMA (peak f). The integral ratio of a:f:g is 4:22:206, which indicates the repeating unit numbers of AnMA and MMA on the polymer chains are 22 and 69, respectively. Based on ^1H NMR result the weight percentage of anthracene group in *DS*-poly(AnMA-*co*-MMA) is about 34.2 %. Anthracene groups on the polymer chains can be used for interfacial photodimerization. Based on gel permeation chromatography (GPC) results the apparent molecular weight and molecular weight distribution of *DS*-poly(AnMA-*co*-MMA) are 26.3 kg mol^{-1} and 1.30, respectively.

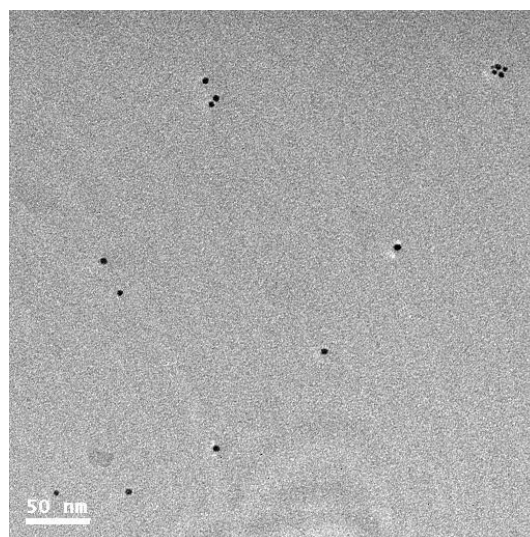


Figure S4. TEM image of gold nanoparticles (AuNPs) used in this research.

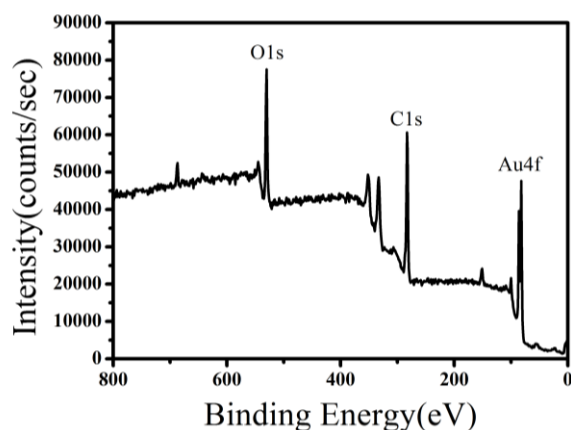


Figure S5. XPS spectrum of hybrid AuNPs.

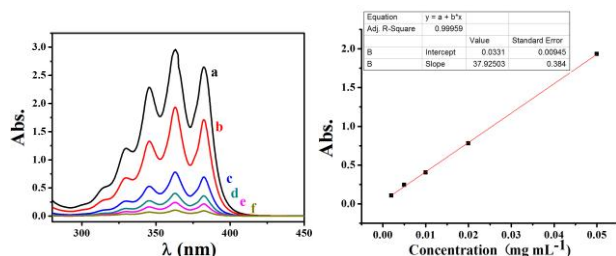


Figure S6. (a) UV absorbance of anthracene-9-carboxylic acid in THF at different concentrations, and (b) linear fitting as the benchmark of absorbance at 364 nm.

In Figure S56, the concentration of anthracene-9-carboxylic acid is 0.1, 0.005, 0.02, 0.01, 0.005, 0.002 mg·mL⁻¹, as the benchmark of absorbance at 364 nm, after linear fitting, the relationship of absorption value and the concentration is expressed as equation (1):

$$Abs. = 0.0331 + 37.92503 * C \dots\dots\dots (1)$$

Where, *Abs* is the absorbance of anthracene-9-carboxylic acid at 364 nm, *C* is the concentration of anthracene-9-carboxylic acid. When the concentration of *DS*-poly(*AnMA-co-MMA*) in THF is 0.018 mg·mL⁻¹, the max absorbance at 364 nm is 0.264, so the concentration of anthracene is about 0.0061 mg·mL⁻¹, which means the weight percentage of anthracene is about 33.8 %, and this value is very close to the ¹H NMR result.

Characterization. The apparent molecular weight and molecular weight distribution of the polymer were determined on a GPC equipped with a Hitachi L-2130 HPLC pump, Hitachi L-2350 column oven operated at 40 °C, three Varian PL columns with 5K-600K, 500-30K, and 100-10K molecular ranges, and a Hitachi L-2490 refractive index detector. THF was used as eluent at a flow rate of 1.0 mL/min. Molecular weights were calibrated on PS standards. Transmission electron microscopy (TEM) observations were carried out on a Tecnai G2 20 S-TWIN electron microscope equipped with a Model 794 CCD camera. TEM specimens were prepared by dipping copper grids into solutions and dried in air. UV-vis absorption spectra were recorded on a Shimadzu UV-2450 spectrophotometer using a quartz cell of 1 cm path length. Dynamic light scattering (DLS) measurements were conducted on a Zetasizer Nano ZS from Malvern Instruments equipped with a 10 mW HeNe laser at a

wavelength of 633 nm. The results were analyzed in CONTIN mode. Scanning electron microscopy (SEM) observations were conducted on a SHIMADZU SS-550 scanning electron microscope. XPS spectra were recorded using a Kratos Axis Ultra DLD spectrometer employing a monochromated Al K α X-ray source ($h\nu=1486.6$ eV), hybrid (magnetic/electrostatic) optics and a multichannel plate and DLD. XPS spectrum was recorded using an aperture slot of 300-700 microns.