Electronic Supplementary Material (ESI) for RSC Advances.

Supporting Information

The Soft Interactions of Aminated SiO_2 Nanoparticles with Fluorescent Partners: A Multi-functional Sensing Platform With Signal Amplification Effect

Ma Hengchang, Yang Zengming, Cao Haiying, Lei Lei, Lei ziqiang

Key Laboratory of Eco-Environment-Related Polymer Materials of Ministry of Education, KeyLaboratory of Polymer Materials of Gansu Province, College of Chemistry and Chemical Engineering,Northwest Normal University, Lanzhou 730070, China

Contents

General Information

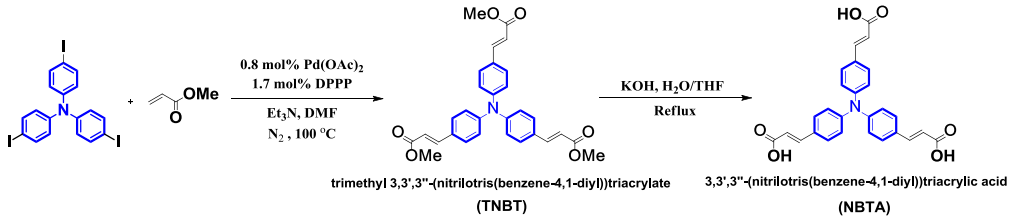
Materials

All commercially available reagents were purchased from Aladdin Co. and Energy Chemical Co. and used without further purification unless otherwise stated.

Tris(4-iodophenyl)amine(98%), Magnesium sulfate ($MgSO_4$, 99%), Methylacrylate (MA, 98%), and Palladium acetate ($Pd(OAc)_2$, 99%), 1,3-bis(diphenylphosphino)propane (DPPP,98%), Triethylamine(Et_3N, 99%), Potassium hydroxide (KOH, 98%)

Method

^1H NMR (400MHz), ^{13}C NMR (100MHz) were recorded on MERCURY spectrometer at 25 °C. Mass spectra were recorded on a HP5989B mass spectrometer. Fourier transform infrared (FT-IR) spectra were recorded on a DIGIL FTS3000 spectrophotometer using KBr tablets. UV spectra were measured on a TU-1901 spectrophotometer.

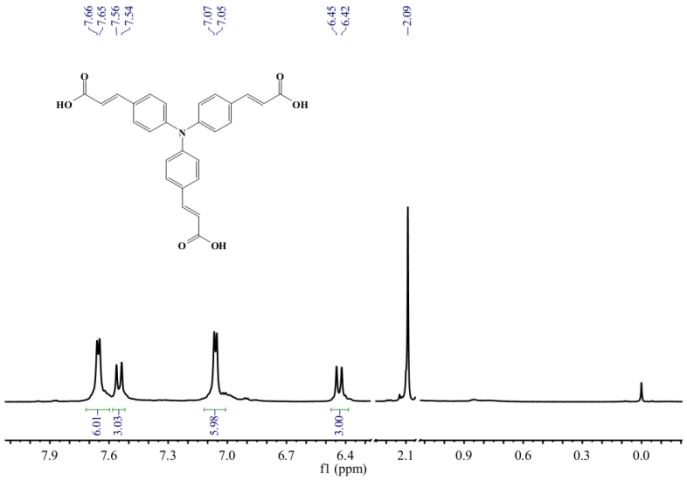


Scheme1. Synthetic procedures for TNBT and NBTA

Synthesis oftrimethyl 3,3',3''-(nitrilotris(benzene-4,1-diyl))triacrylate (TNBT). Tris(4-iodophenyl)amine (2.4 g, 5 mmol), methylacrylate (18 mmol, $Pd(OAc)_2$) (27 mg, 0.12 mmol), 1,3-bis(diphenylphosphino)propane (DPPP, 103.1 mg, 0.25 mmol) and Et_3N (1.8 g, 18 mmol) were added into 10 mL dry dimethylformamide (DMF) and stirred for 48 h at 100 ℃ under N_2 atmosphere. The reaction process was monitored by the disappearance of the tris(4-iodophenyl)amine. Then 20 mL water was charged, and the solution was extracted with CH_2Cl_2 (3×10 mL). The organic phases were dried with anhydrous magnesium sulfate, filtered, and concentrated CH_2Cl_2 solution under reduced pressure. The residue was purified by column chromatography on silica gel (300-400 mesh) with a mixture of ethyl acetate and petroleum ether as eluent (1:100 by volume), leading to the purified TNBT 2.24 g, yield: 90%.

Synthesis of 3,3',3''-(nitrilotris(benzene-4,1-diyl))triacrylic acid (NBTA).

TNBT (2.49 g，5 mmol), KOH (1.12 g，20 mmol) were added into the mixed solvent (THF:distilledwater = 6:1, 35 mL). Warming at 95 ^{0}C for 12 h, the solution was evaporated to remove THF under reduced pressure. Charging HCl solution into the aqueous phase, and the pH value was judged to 1.5，green solid was precipitated, filtered, and dissolved into acetone. Afterwards, $MgSO_4$ were added to the solution for exclude the small fraction of water. And then filtrating and evaporating were employed to the purified NBTA 2.1 g, yield: 95%. M.p. > 300 ^{0}C; ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.65 (d, J = 7.8 Hz, 2H), 7.55 (d, J = 15.8 Hz,1H), 7.06 (d, J = 7.8 Hz, 2H), 6.43 (d, J = 15.9 Hz, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 170.9, 145.6, 140.8, 129.3, 127.1, 121.4, 116.9 ppm. IR (KBr): 1684, 1592, 1507, 1397, 1318, 1270, 1176, 1118, 869, 819, 517 cm^{-1}.



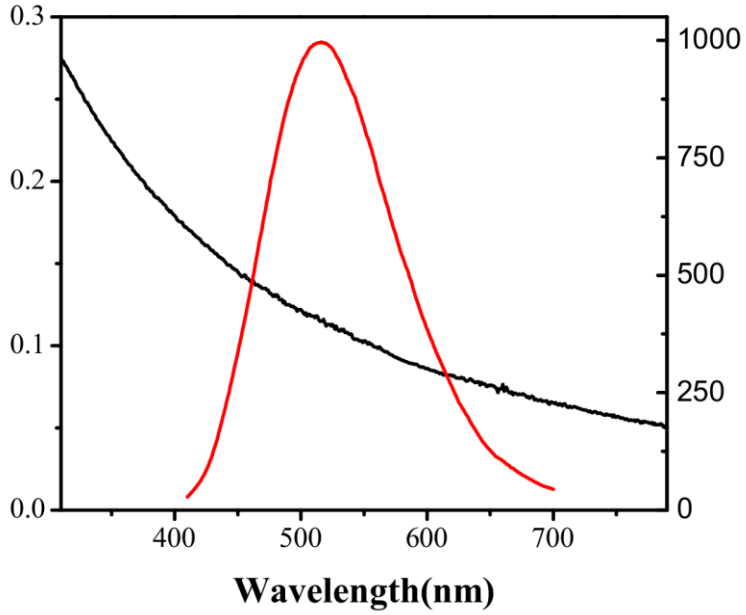


Fig.S1 The overlap of UV spectrum of SiO_2-NH_2NPs(black line) and the PL spectrum of NBTA(red line)