Porous films based on a conjugated polymer gelator for fluorescent detection of explosive vapors

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1. General Information

$^1$H and $^{13}$C NMR spectra were recorded at 298 K obtained with a Bruke Avance 300 NMR spectrometer. Number-average ($M_n$) and weight-average ($M_w$) molecular weights were determined by GPC on a Waters 410 instrument with polystyrene as standards and THF as eluent. MALDI-TOF was measured by a Bruker Daltonics Flex analysis system. UV-Vis absorption spectra were recorded by a Perkin-Elmer Lambda35 UV/vis spectrometer. PL spectra were recorded with a Perkin-Elmer LS50B spectrofluorometer. Thermal property of the polymer was analyzed with a Perkin-Elmer-TGA 7 instrument under nitrogen at a heating rate of 10 °C/min.

2. Synthesis Procedures

Monomer (1) and Monomer (2) were synthesized according to the published procedures.$^{1,2}$ In a 50 mL flask of flame-drying, Monomer (1) (0.5594, 1.0mmol), Monomer (2) (0.9870, 1.0mmol) and Pd(PPh$_3$)$_4$ (0.0011g, 0.001 mmol) were dissolved in 6 mL of degassed toluene and 2 mL of 2M aqueous potassium carbonate. The reaction mixture was vigorously stirred at 100 °C for 24h. After the reaction was finished, the polymer was purified by precipitation in methanol and air-dried overnight. The product was obtained as green floccule with a yield of 89%. $^1$H NMR (400 MHz, CDCl$_3$), δ (TMS, ppm): 7.97(m,6H) 7.74(m,3H) 7.45(s,3H) 7.07(s,2H) 6.33(s,2H) 4.34(m,2H) 4.11(s,4H) 3.73(s,4H) 1.87—1.22(m,64H) 0.87(m,15H);

GPC: $M_n = 1.29 \times 10^4$ g/mol PDI=2.48.

3. The Formation of P1 Gel

The gelator and the solvent were put in a screw-capped test tube and heated until the solid was dissolved. The solution was cooled to room temperature and turned upside down. The gelator is believed to form a stable gel if it can immobilize the solvent at this state.$^3$
Put several drops of the warm solution on the surface of a quartz substrate. After 5 minutes, the temperature will cool down to room temperature completely, and a polymer gel is formed, which can be used to test PL emission directly.

4. Film Preparation and Fluorescence Measurement

The film was fabricated by spin-coating the P1 solution of different concentrations with different spin rate on a quartz substrate. The film thickness was determined by a step profiler. A linear relationship of optical density (OD) with thickness was established and this allows the film thickness to be measured by OD.

The fluorescence response of the P1 films to the vapors of analytes was measured by inserting the prepared films into a quartz cuvette containing analytes at room temperature. A small quantity of the analyte was placed beneath some cotton wool inside a sealed quartz cuvette and left overnight to allow the analyte vapor to reach equilibrium (fully saturated). The cotton wool prevents the direct contact of the polymer film with the analytes and helps to maintain a constant saturated vapor pressure. Polymer film was placed in the quartz cuvette and fluorescence spectra were measured after exposing the film for a specific interval time.

5. Recovery Test

After exposure to explosive vapors, the fluorescence of P1 film can be recovered by exposure to the vapor of hydrazine for 1 hour. The recovered film showed similar quenching efficiency when it was re-exposed to the explosive vapors.
Fig S1. TGA curve of polymer P1.

Reference:


