Electronic Supplementary Information

From Nano Ribbon to Fibre by Concentration Control

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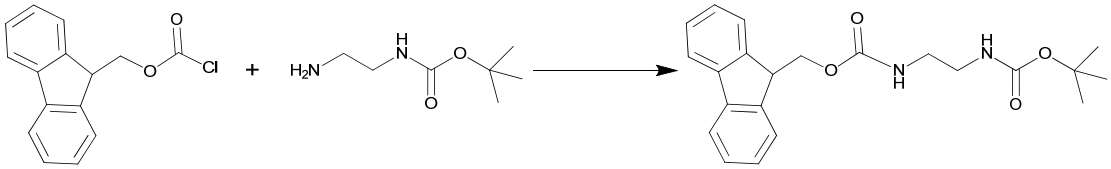
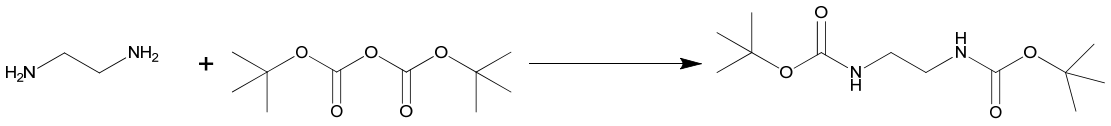
Experimental procedure

Synthesis



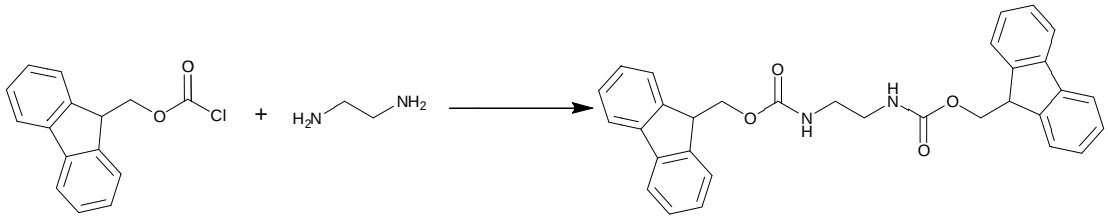
1

Mono-boc-protected ethylenediamine [1] (7.70 g, 50 mmol) was dissolved in dichloromethane (100 mL). The pH was adjusted to 8-9 using triethylamine. Subsequently, a solution of 9-fluorenylmethoxycarbonyl chloride (Fmoc-Cl) (25.7 g, 100 mmol) in dichloromethane (100 mL) was added in one portion. The mixture was allowed to stir for 12 h and the solvent was evaporated to dryness. The residue was washed with water and acetonitrile, dissolved in $CHCl_3$ and MeOH, and evaporated. The crude product was purified by column chromatography on silica gel with acetic ester/petrol ether (1:1, v/v) as the eluent to give **1** as a white solid.[2] Mp. 156-158°C. ^{1}H-NMR (400 MHz, $CDCl_3$): δ 7.77-7.75 (d, J = 8 Hz, 2H), 7.60-7.58 (d, J = 8 Hz, 2H), 7.42-7.38 (t, J = 8 Hz, 2H), 7.35-7.31 (t, J = 8 Hz, 2H), 5.18 (s, 1H), 4.78 (s, 1H), 4.42-4.40 (d, J = 8 Hz, 2H), 4.22-4.18 (t, J = 8 Hz, 1H), 3.28 (s, 4H), 1.44(s, 9H) ; ^{13}C NMR (100 MHz, $CDCl_3$): δ 156.9, 156.6, 144.0, 141.5, 127.8, 127.2, 125.2, 120.1, 79.8, 66.8, 47.4, 41.8, 40.7, 28.5. HR-MS: 405.1792 $[M+Na]^+$.



2

2 was synthesized according to ref. 3 and checked by melting point and ^{1}H NMR. Mp. 130-132°C. ^{1}H-NMR (400 MHz, $CDCl_3$): δ 4.28 (s, 1H), 3.23 (s, 4H), 1.44 (s, 18H).



3

To a mixture of ethylenedianmine (2 mmol) and $NaHCO_3$ (10%, 6 mL) in dioxane (3 mL), a solution of Fmoc-Cl (1.55 g, 6 mL) is slowly added at 0°C. The mixture is vigorously stirred at rt overnight. Then, ACOET (60 mL) and HCl 1M (30 mL) were added; the organic layer is separated, washed with bine and dried over Na_2SO_4. Evaporated of the solvent and purification by column chromatography using CH_2Cl_2: MeOH = 100: 1 as eluent, affords the pure products **3** as white solids. Mp. 227-228°C. ^{1}H-NMR (400 MHz, $CDCl_3$): δ 7.89-7.88 (d, J = 8 Hz, 4H), 7.68-7.66 (d, J = 8 Hz, 4H), 7.42-7.39 (t, J = 8 Hz, 4H), 7.33-7.30 (t, J = 8 Hz, 4H), 4.29-4.28 (d, J = 8 Hz, 4H), 4.20-4.18 (t, J = 8 Hz, 2H), 3.04(m, 4H), HR-MS calc for $C_{32}H_{28}N_2NaO_4$: 527.1947[M+Na]$^+$, found: 527.1961.

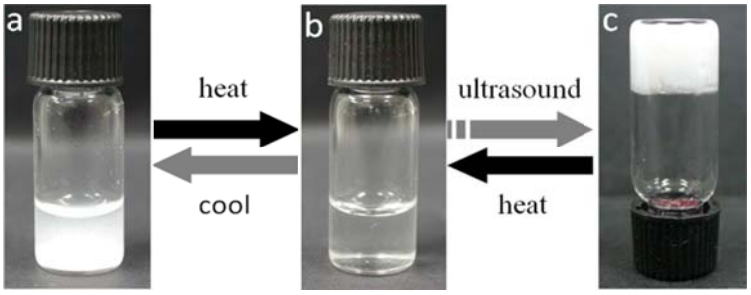


Fig. S1 a, 10 mg gelator and 0.5 mL ethanol were put into 1.5 mL glass vial; b, the gelator all dissolve into ethanol solution when heated in the water bath at 80 ºC; c, the gel were formed in the ultrasonic instrument (500 w, 40 KHz, 15 minutes).

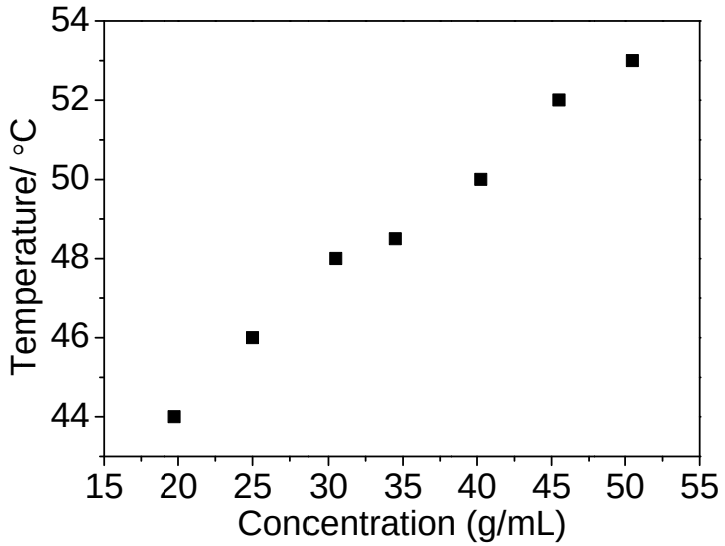


Fig. S2 The gel transition temperature with different concentration of **1** in ethanol

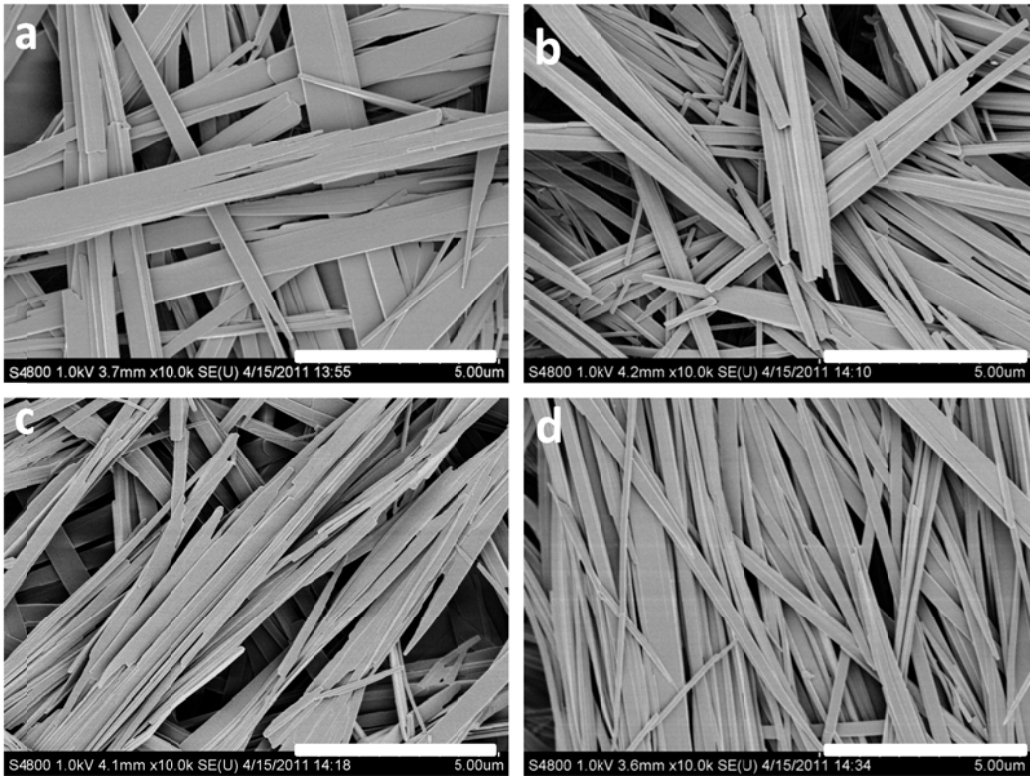


Fig. S3 SEM images of xerogel of **1** from ethanol in different concentration: (a) 20 mg mL^{-1}, (b) 30 mg mL^{-1}, (c) 40 mg mL^{-1} and (d) 50 mg mL^{-1}; all the scale bars are 5.0 μm

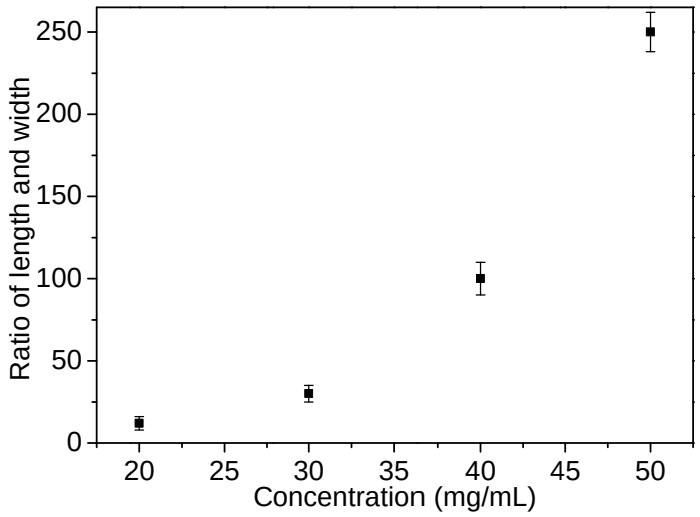


Fig. S4 The plot relating the aspect ratio and concentration

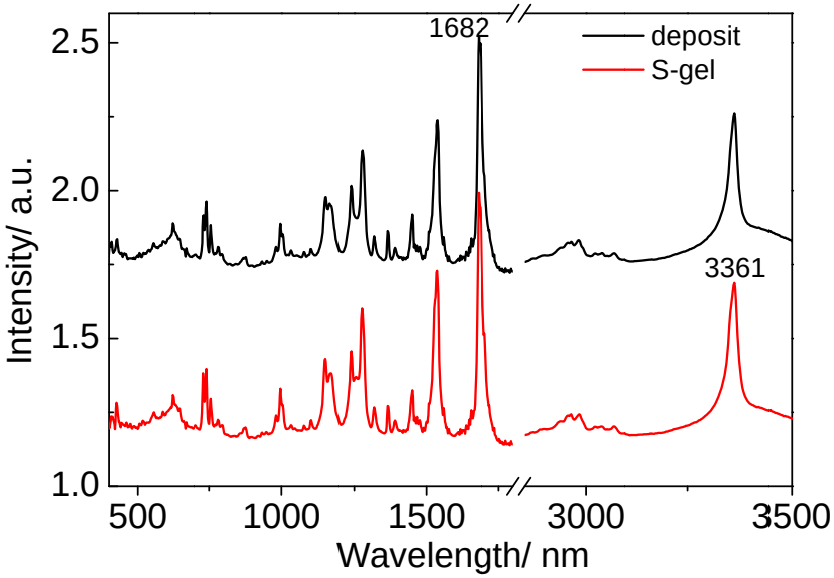


Fig. S5 IR spectra of 25 mg mL^{-1} hot sol **1** in ethanol after cooling at room temperature (the black line) and treated with sonication at room temperature for 5 min (the red line).

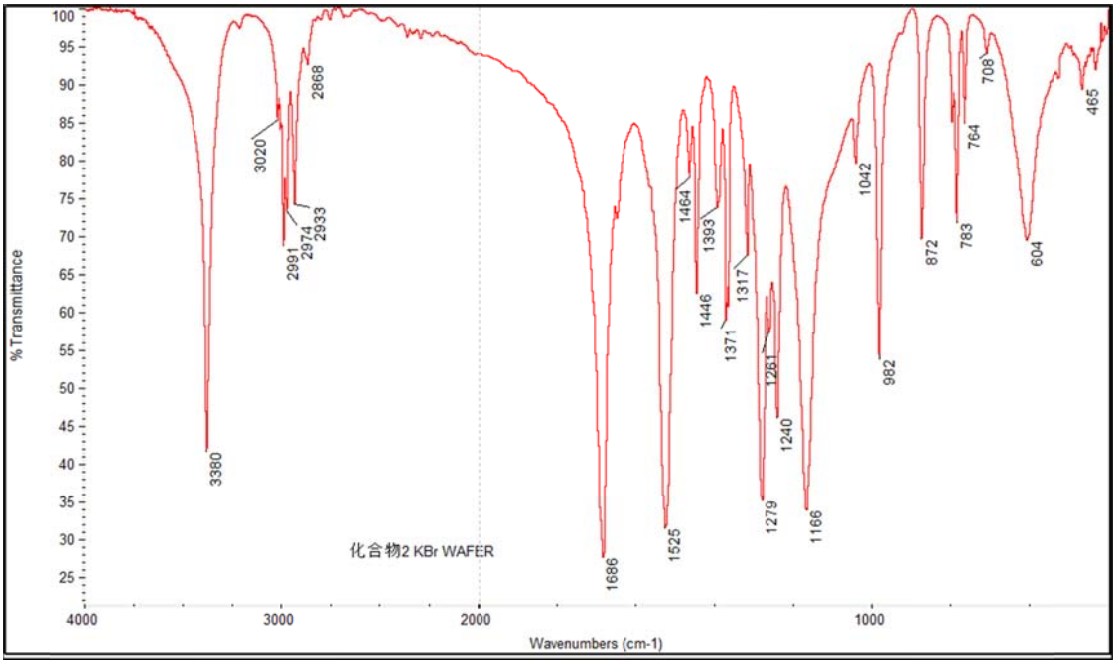


Fig. S6 IR spectra of powder of **2** from ethanol at room temperature.

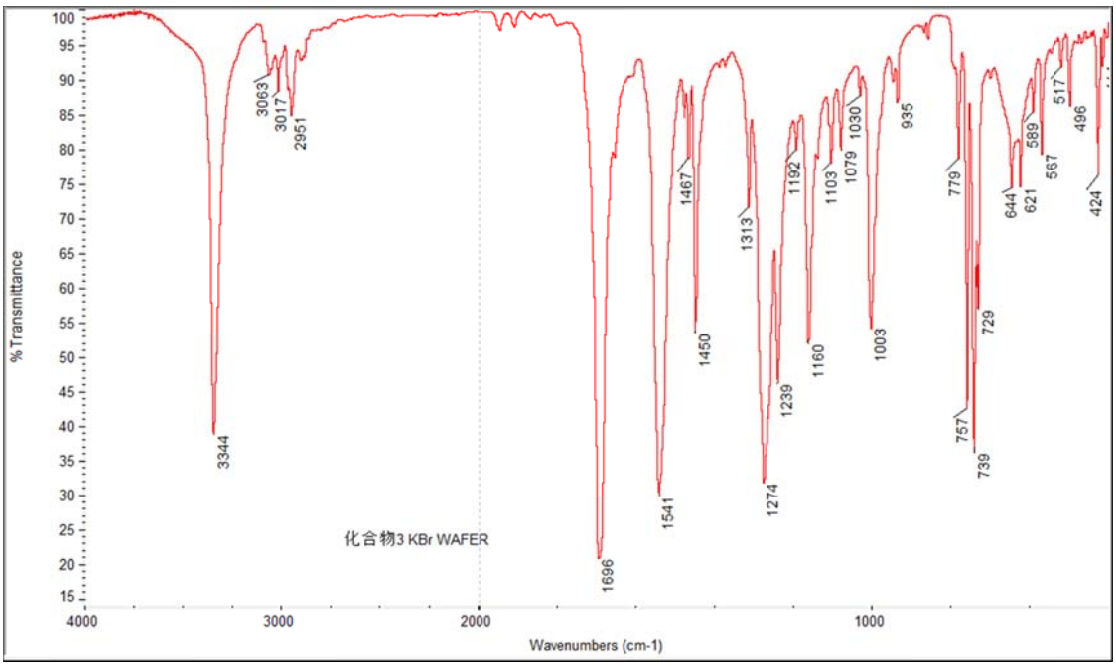
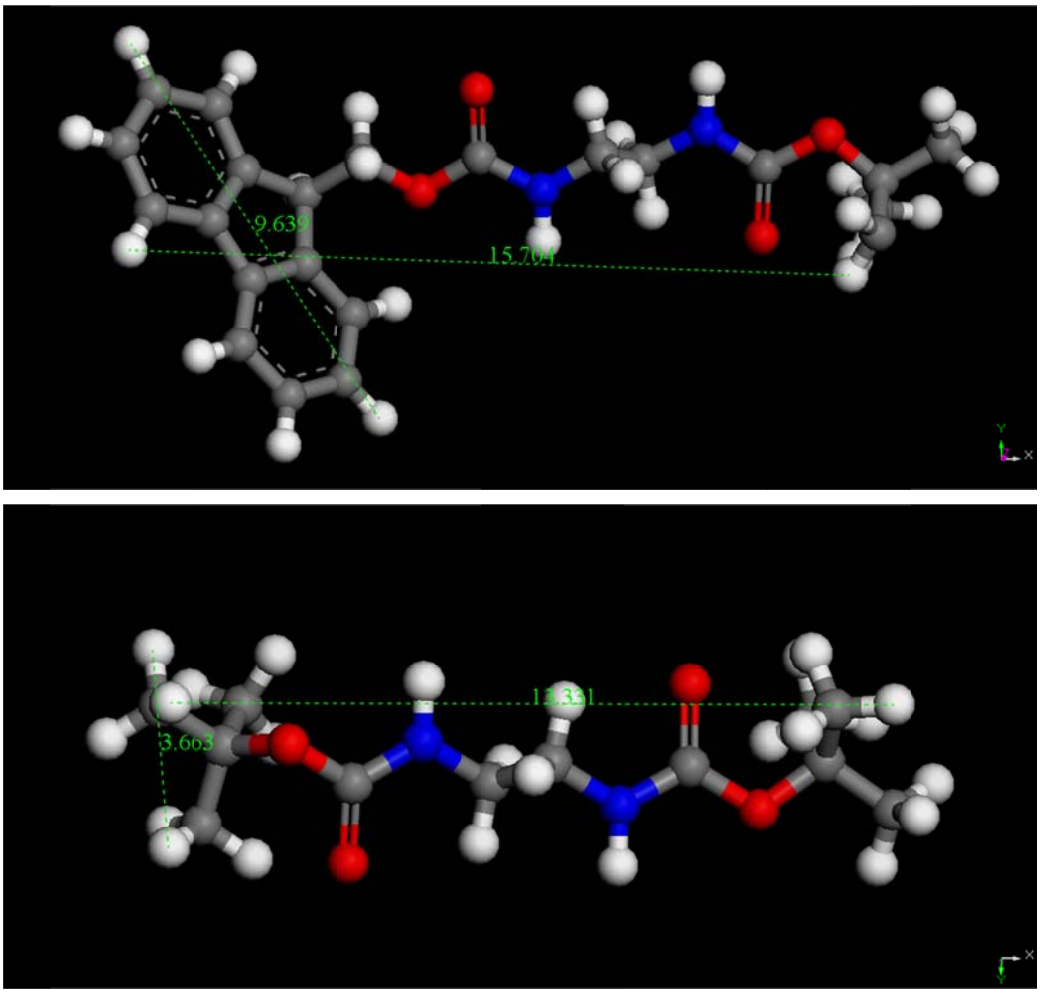


Fig. S7 IR spectra of powder of **3** from ethanol at room temperature.



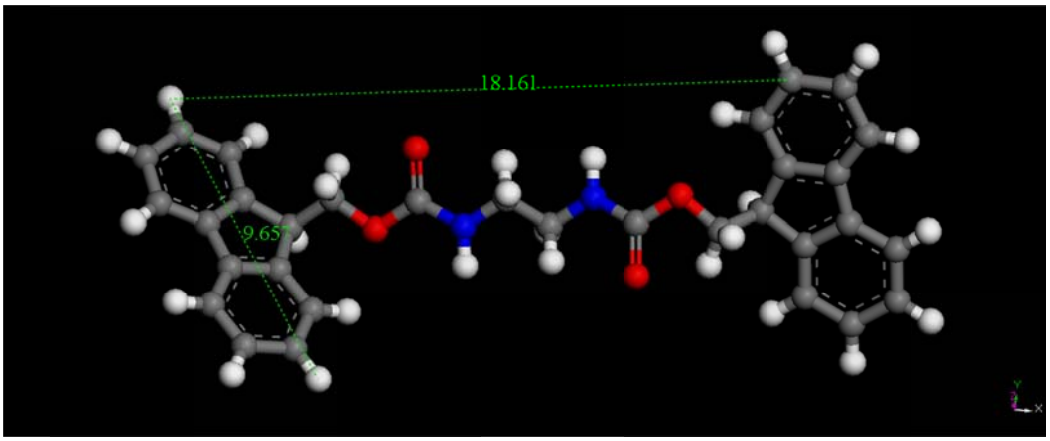


Fig. S8 Computer stimulation of molecule **1, 2** and **3** by the materials studio.

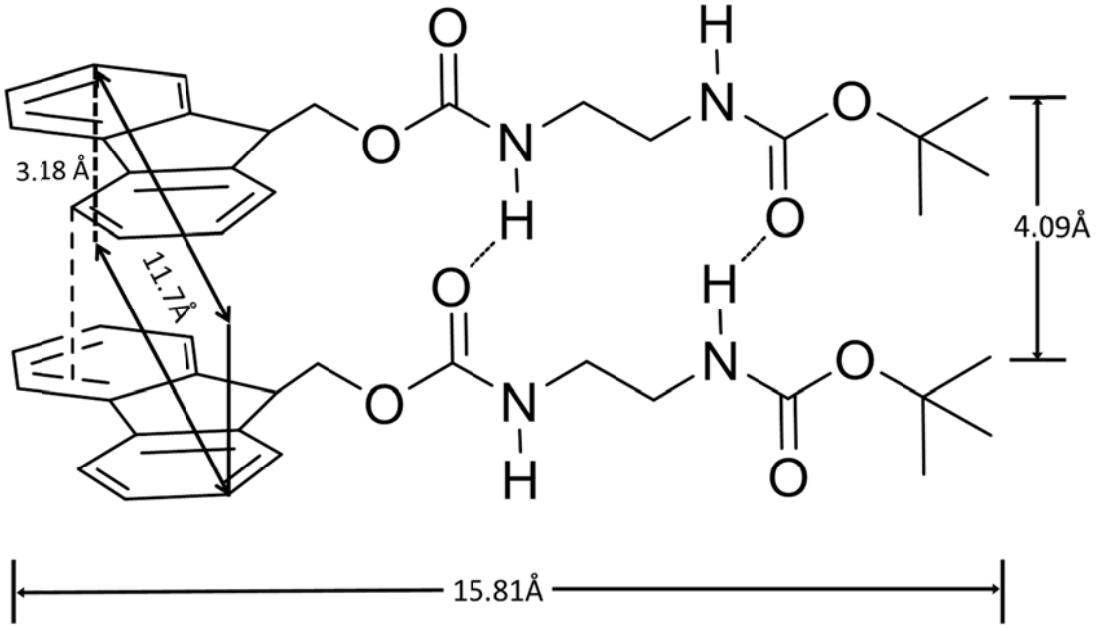


Fig. S9 Computational simulation of intermolecular interactions between mo**l**ecules of **1**

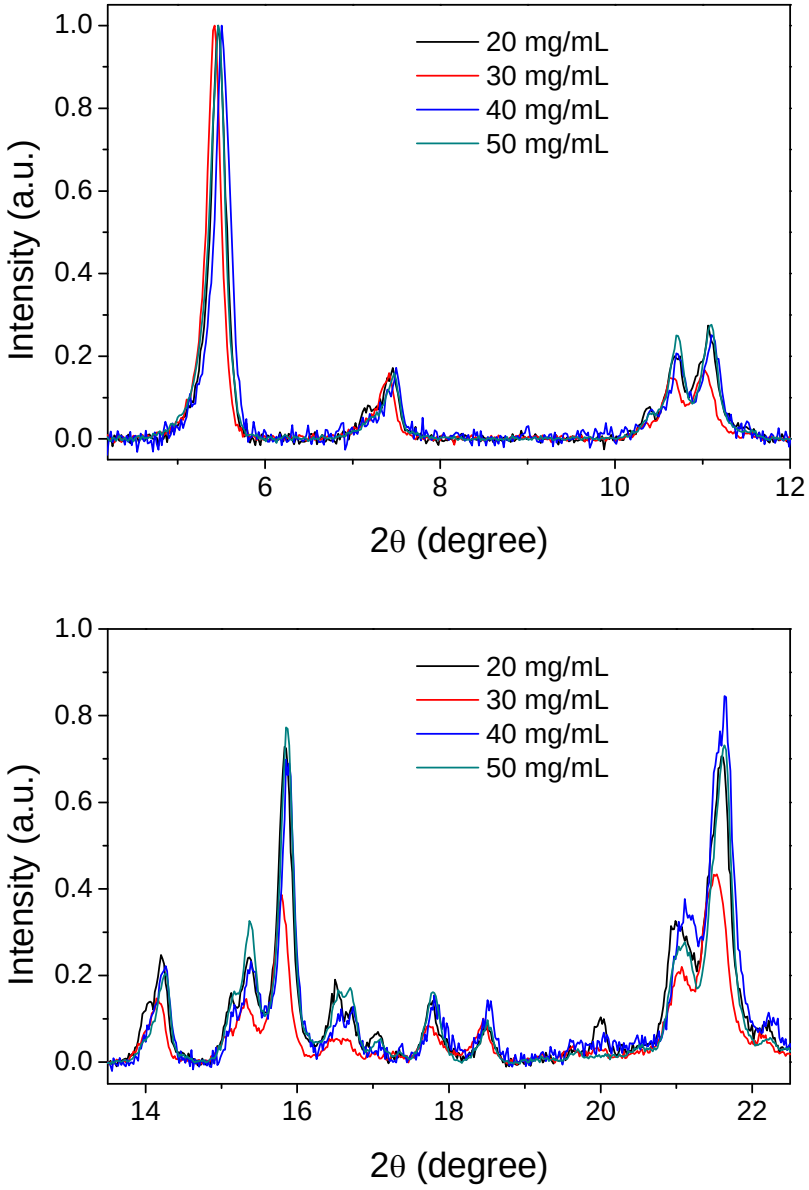


Fig. S10 XRD profile of the xerogel of **1** with different concentration in the 2θ range of (a) 4.2-12°and (b) 12- 22.5°at room temperature

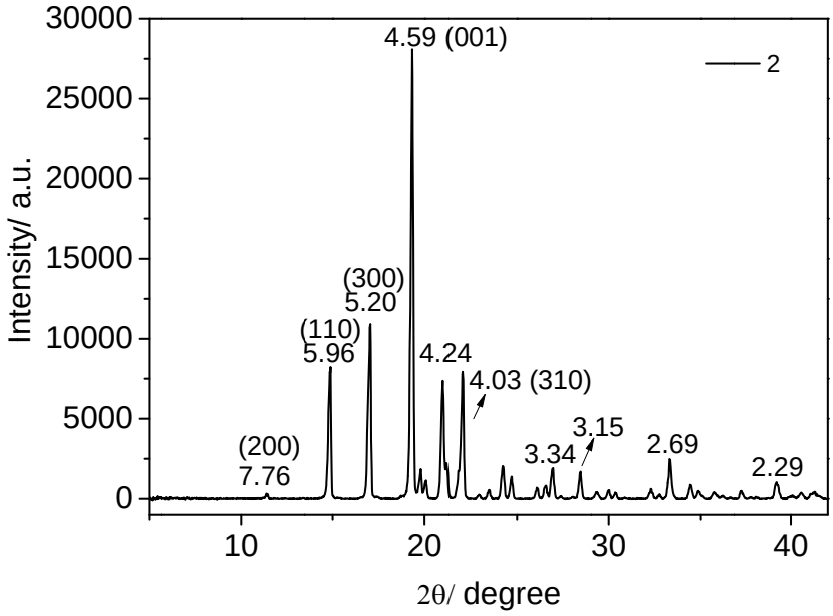


Fig. S11 XRD profile of (a) the powder of **2** in the 2θ range of 5-42° at room temperature

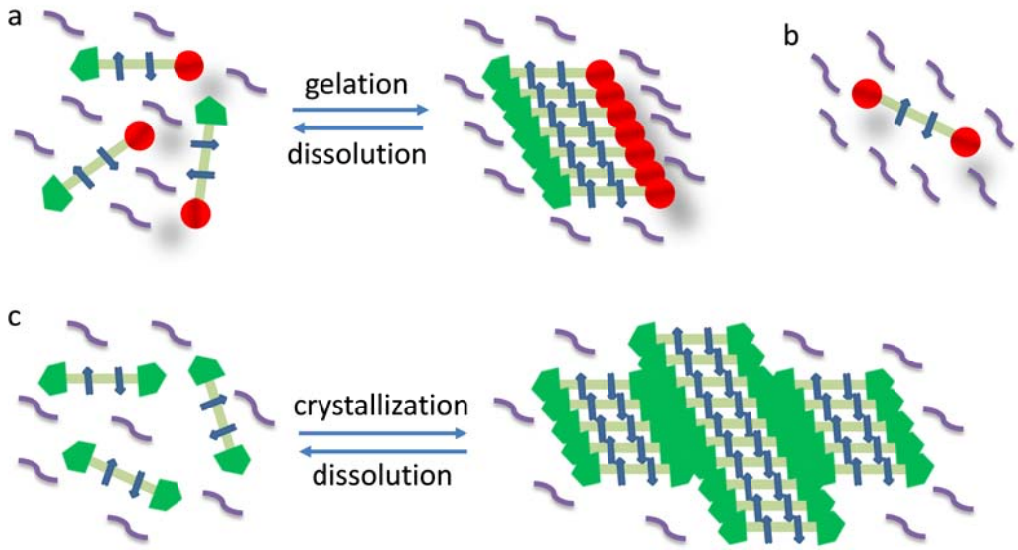


Fig. S12 The proposed mechanism for gelation, dissolution and crystallization in **1** (a), **2** (b) and **3** (c), with the green part representing fluorene group, red ball being tert-butyl group, blue arrows being amides and purple line being solvent.

Reference

1. I. H. Kim, C. Morisseau, T. Watanabe and B. D. Hammock, *J. Med. Chem.*, 2004, **47**, 2110-2122.
2. A. Watzke, M. Gutierrez-Rodriguez, M. Köhn, R. Wacker, H. Schroeder, R. Breinbauer, J. Kuhlmann, K. Alexandrov, C. M. Niemeyer and R. S. Goody, *Bioorgan. Med. Chem.*, 2006, **14**, 6288-6306.
3. T. T. Chang, S. V. More, I. Lu, J. C. Hsu, T. J. Chen, Y. C. Jen, C. K. Lu and W. S. Li, *Eur. J. Med. Chem.*, 2011, **46**, 3810-3819.

Electronic Supplementary Material (ESI) for CrystEngComm