

A Fluorescent Supramolecular Crosslinked Polymer Gel Formed by Crown Ether Based Host-Guest Interactions and Aggregation Induced Emission

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Abstract Based on the combination of B21C7/dialkylammonium salt host-guest interactions and tetraphenylethylene (TPE)-based aggregation-induced emission (AIE) effect, a fluorescent supramolecular crosslinked polymer gel was successfully prepared. Compared with the solution of TPE-containing small molecules, this gel exhibited remarkable fluorescence enhancement due to the AIE effect of TPE units. The “gelation induced fluorescence emission” phenomenon can be explained by the hindered intramolecular rotation of phenyl rings of TPE. Because of the reversibility and stimuli-responsiveness of the B21C7/dialkylammonium salt host-guest interactions, the transition between the fluorescent supramolecular crosslinked polymer gel and the disassembled sol with very weak fluorescence can be realized by adding pH and thermal stimuli. This novel material contributes to the development of supramolecular chemistry, polymer science and fluorescent materials and offers a new method to construct functional supramolecular materials.

Keywords: Fluorescent polymer gel; Aggregation-induced emission; Host-guest interactions; Responsiveness.

INTRODUCTION

Fluorescent self-assembled organogels are an important class of smart soft materials and have found diverse applications in optoelectronics, fluorescence sensors, *etc*^[1–5]. However, most π -conjugated molecules suffer from emission quenching when self-assembled although they are highly emissive in the solution state. The light emission of the self-assembled materials are debilitated by the severe intermolecular interactions which usually consume the excited state energy^[6–8]. Therefore, it is of great importance to develop tailored π -conjugated organogels with efficient emissions in the aggregated state. In 2001, Tang and co-workers observed that a series of silole derivatives were non-fluorescent in the solution state but became highly emissive in the aggregated state^[9]. The term “aggregation-induced emission” (AIE) was coined for this unusual phenomenon because the non-emissive molecules were induced to emit by aggregate formation. This unique phenomenon of AIE owes to the restriction of intramolecular torsional/rotational motions, and has potential applications in permitting the use of dye solutions with any concentration for bioassays and enabling the development of “turn on” or “light up” nanosensors^[10]. Especially, the AIE effect has gained much attention in organogel systems recently because the self-assembled chromophores show bright fluorescence in the gel state^[10–14].

Supramolecular crosslinked polymer gels are the aggregates of traditional polymers connected by noncovalent interactions^[15–22]. Among all the noncovalent interactions, host-guest interactions have been widely used in the construction of functional supramolecular materials because they combine multiple non-covalent interactions such as hydrogen bonding, π -stacking, charge transfer, and/or hydrophobic interactions between complementary compounds, forming complexes with fixed host-guest geometry and directionality^[23–33].

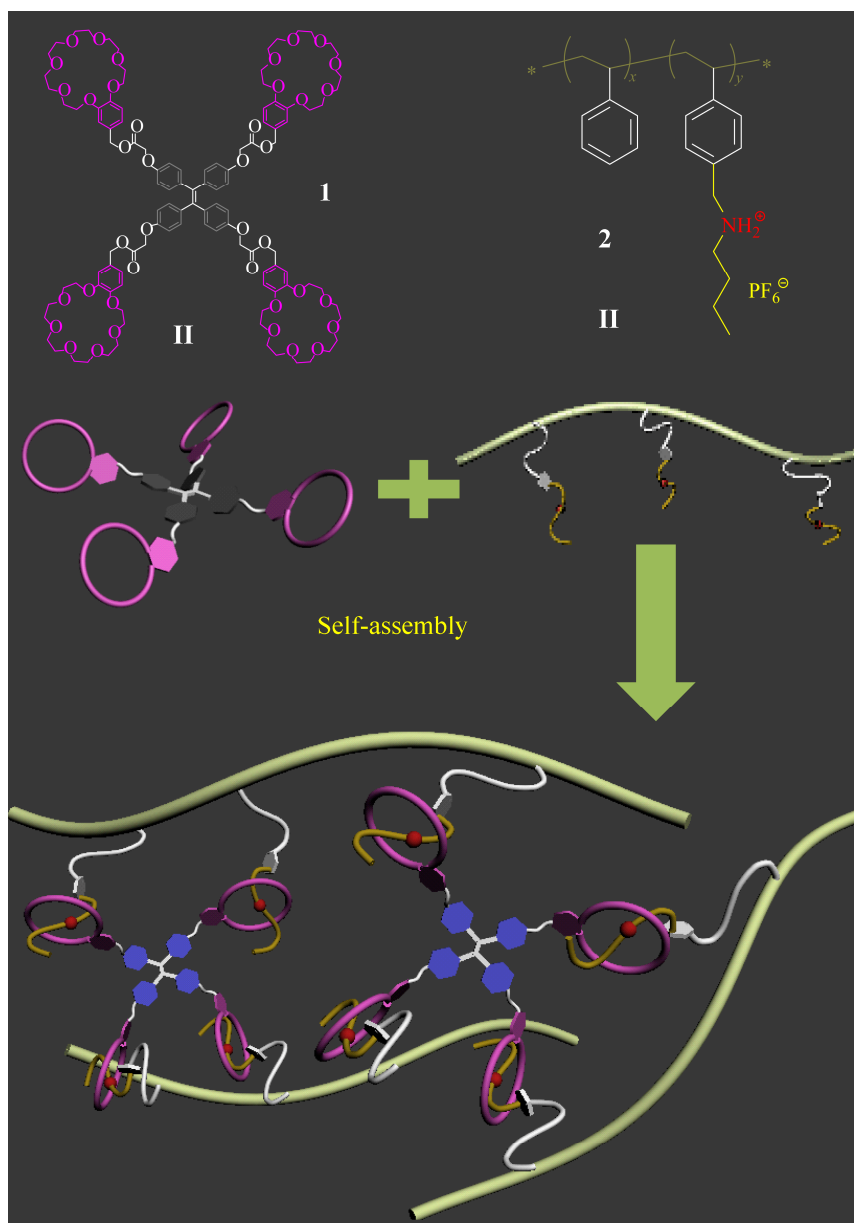
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Moreover, due to the reversibility and abundant stimuli-responsivenesses of host-guest interactions^[34–47], supramolecular crosslinked polymer gels based on host-guest interactions, one kind of the self-assembled polymer materials^[48–50], have exhibited fascinating properties: easier to process and recycle; acting as stimuli-sensitive materials in sensing, actuation, and controlled release; and self-healing ability^[18]. Although much progress has been made in the studies of these properties, fluorescent supramolecular crosslinked polymer gels based on host-guest interactions have been rarely reported^[15–22].

In this work, we report a fluorescent supramolecular crosslinked polymer gel formed by combining crown ether based host-guest interactions and AIE. As shown in Scheme 1, compound **1** has a tetraphenylethylene (TPE) moiety, a famous chromophore with the AIE effect, and four benzo-21-crown-7 (B21C7) units, and polymer **2** is a polystyrene (PS) polymer with pendent dialkylammonium salt moieties. The B21C7 host units



Scheme 1 Chemical structures of **1** and **2** and cartoon representation of the fluorescent supramolecular crosslinked polymer gel formed by self-assembly of **1** and **2**

bind the dialkylammonium salt guest moieties when **1** and **2** are mixed together in solution^[51, 52], thereby resulting in the formation of a supramolecular crosslinked polymer gel. At the same time, TPE moieties aggregate together, bringing about bright fluorescence. Therefore, a fluorescent supramolecular crosslinked polymer gel forms. Moreover, owing to the reversibility and stimuli-responsiveness of the B21C7/dialkylammonium salt recognition motif^[44], the reversible gel-sol transitions can be realized, which accompanies with the corresponding changes in the fluorescence intensity.

EXPERIMENTAL

Materials and Methods

All reagents were commercially available and used as supplied without further purification. Compound **4** and polymer **2** were prepared according to the published procedures^[53, 54]. NMR spectra were recorded with a Bruker Avance DMX 400 spectrophotometer using the deuterated solvent as the lock and the residual solvent or TMS as the internal reference. Low-resolution electrospray ionization mass spectra were recorded with a Bruker Esquire 3000 Plus spectrometer. High-resolution mass spectrometry experiments were performed with a Bruker Daltonics Apex III spectrometer. Molecular weights and molecular weight distributions were determined by gel permeation chromatography (GPC) with a Waters 1515 pump and Waters 1515 differential refractive index detector (set at 30 °C). It used a series of three linear Styragel columns (HT2, HT4, and HT5) at an oven temperature of 45 °C. The eluent was THF at a flow rate of 1.0 mL/min. A series of low polydispersity polystyrene standards was employed for the GPC calibration. Viscosity measurements were carried out with a Cannon-Ubbelohde semi-micro dilution viscometer at 25 °C in chloroform. Fluorescent confocal images were recorded on a Zeiss LSM 710 confocal fluorescence microscopy. The fluorescence spectra were recorded on a Perkin Elmer LS55 fluorescence spectrophotometer. Rheological data were obtained using a Physica MCR302 rheometer (Anton Paar) with cone-plate geometry (diameter of 25 mm, 2° cone, truncation height of 0.3 mm). Oscillatory frequency sweep experiments were performed from 0.1 rad/s to 300 rad/s with a strain in the linear region at 10 °C.

RESULTS AND DISCUSSION

Syntheses of **1** and **2**

Synthesis of compound **1**

The synthetic routes to **1** and **2** are shown in Scheme 2.

A mixture of compound **4** (0.950 g, 1.51 mmol), compound **5** (3.51 g, 9.08 mmol), 4-dimethylaminopyridine (DMAP, catalytic amount) and 1-(3'-dimethylaminopropyl)-3-ethylcarbodiimide hydrochloride (EDC, 1.16 g, 6.06 mmol) in CH₂Cl₂ (100 mL) was stirred for 48 h at room temperature, filtered, and concentrated to give a white solid, which was purified by flash column chromatography (CH₂Cl₂/CH₃OH, 20:1 *V/V*) to afford **1** as a white solid (1.43 g, 45%), mp: 118.2–119.7 °C. The ¹H-NMR spectrum of compound **1** is shown in Fig. 1.

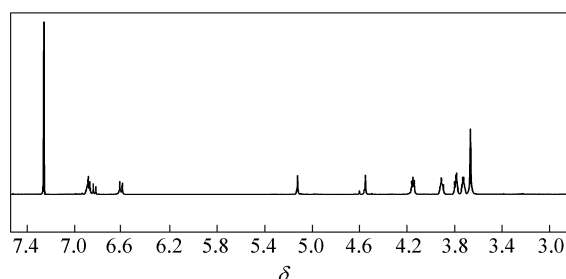
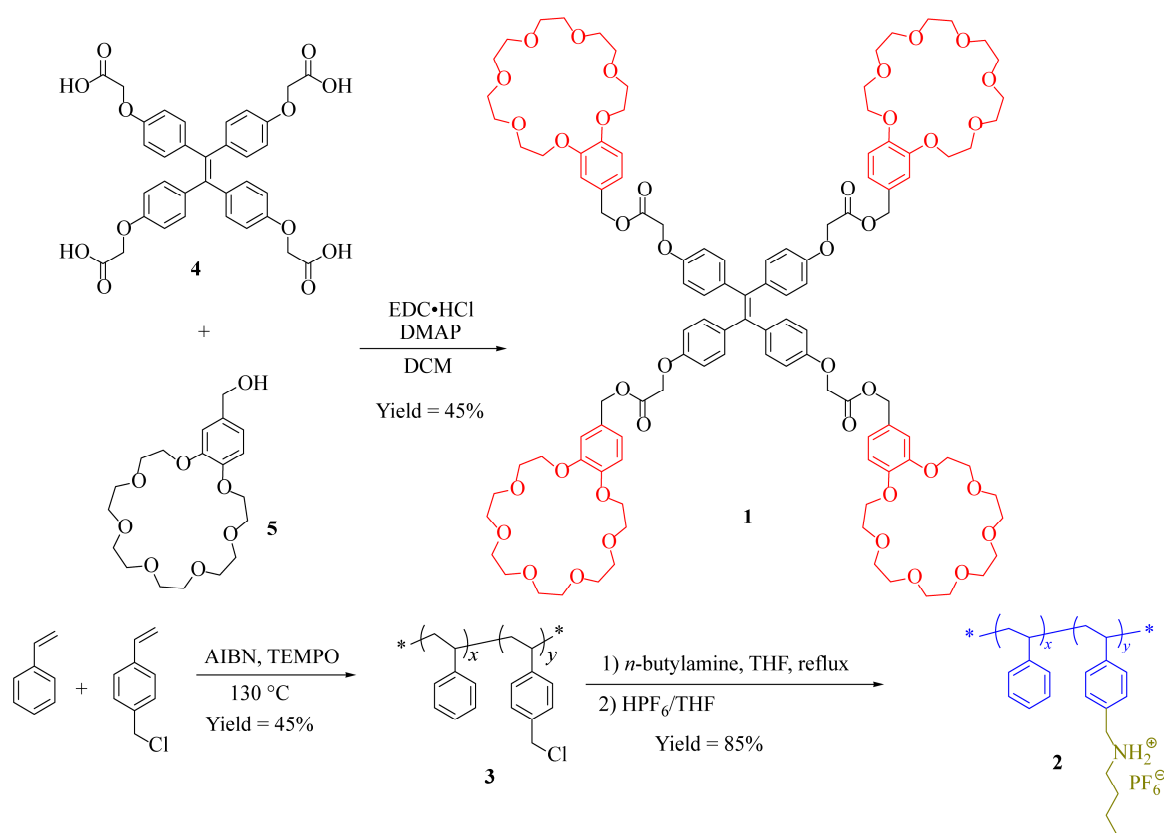


Fig. 1 ¹H-NMR spectrum (400 MHz, CDCl₃, 298 K) of **1**

Scheme 2 Synthetic routes to **1** and **2**

¹H-NMR (400 MHz, CDCl₃, 298 K, δ) 6.87–6.73 (m, 20H), 6.54 (s, 8H), 5.06 (s, 8H), 4.49 (s, 8H), 4.11–4.05 (m, 16H), 3.88–3.81 (m, 16H), 3.75–3.70 (m, 16H), 3.68–3.63 (m, 16H), 3.63–3.56 (m, 32H). The ¹³C-NMR spectrum of **1** is shown in Fig. 2. ¹³C-NMR (125 MHz, CDCl₃, 298 K, δ) 167.7, 155.1, 148.2, 147.8, 131.6, 127.2, 121.1, 113.7, 112.8, 70.0, 69.5, 68.7, 68.2, 66.0, 64.3, 28.7. LRESIMS is shown in Fig. 3: m/z 2124.3 [$M + Na$]⁺ (100%). HRESIMS: m/z calcd for [$M + Na$]⁺ C₁₁₀H₁₄₀NaO₄₀⁺, 2123.8899; found 2123.8912, error 0.6.

Synthesis of polymer **3**

A mixture of 2,2,6,6-tetramethylpiperidine-1-oxyl (TEMPO) (832 mg, 5.33 mmol, 2.5 eq.) and 2,2-azobisisobutyronitrile (AIBN, 350 mg, 2.13 mmol, 1.0 eq.) was added to a 50 mL Schlenk flask equipped with a stirring bar. This flask was then deoxygenated by degassing and backfilling with nitrogen for two times and charged with deoxygenated styrene (10.2 mL, 107 mmol, 25 eq.) and *p*-chloromethylstyrene (3.30 mL, 21.3 mmol, 5 eq.) through syringes. After three freeze-pump-thaw cycles, the reaction mixture was heated by immersing into a 130 °C oil bath for 72 h. The polymerization was stopped through cooling with an ice bath. The reaction mixture was dissolved in 50 mL of toluene and quickly passed through neutral Al₂O₃ column, and then precipitated into cold methanol (500 mL). The precipitate was collected by filtration and dried overnight in a vacuum to give **3** as a white powder (solution polymerization in xylene: 5.70 g, 45%; $M_{n, GPC}$ = 8.11 kDa, $M_{w, GPC}$ = 9.06 kDa, PDI = 1.11). The ¹H-NMR spectrum of polymer **3** is shown in Fig. 4. ¹H-NMR (400 MHz, CDCl₃, 298 K, δ) 7.23–6.21 (m, 40H, C₆H₅), 4.53 (m, 2H, Ar–CH₂–), 2.00–1.61 (m, mainchain, 4H), 1.53–1.18 (m, mainchain, 14H). The value of x/y is 7.2/1, x = 65, and y = 9.0 as measured by NMR and GPC.

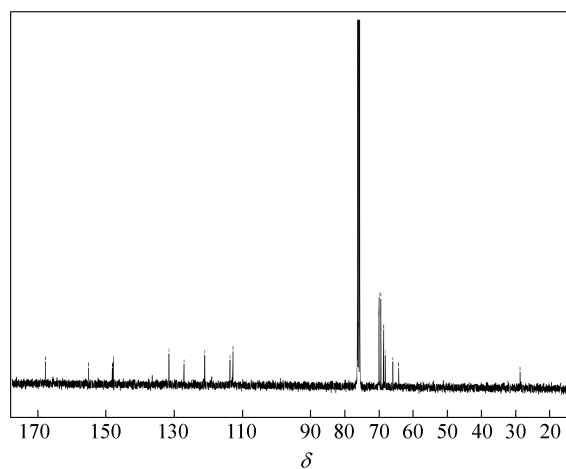


Fig. 2 ^{13}C -NMR spectrum (125 MHz, CD_3Cl , 298 K) of **1**

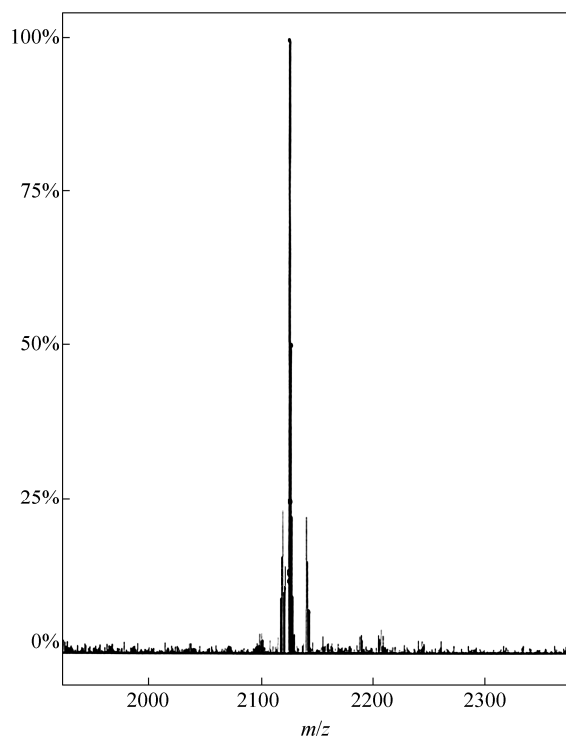


Fig. 3 Electrospray ionization mass spectrum of **1**

Synthesis of polymer 2

A mixture of polymer **3** (5.00 g, 0.616 mmol) and *n*-butylamine (6.01 mL, 61.6 mmol) in THF (50 mL) was stirred at reflux overnight, and then concentrated to precipitate into cold methanol (100 mL). The precipitate was collected by filtration and dried overnight in a vacuum to give a white powder (4.90 g). The white powder (4.90 g) and HPF_6 (5.45 mL, 61.6 mmol) in THF (50 mL) were stirred for 4 h at room temperature, and then concentrated to precipitate into cold methanol (100 mL). The precipitate was collected by filtration and dried overnight in a vacuum to afford a white powder polymer **2** (5.10 g, 85%). The ^1H -NMR spectrum of polymer **2** is shown in Fig. 5. ^1H -NMR (400 MHz, CDCl_3 , 298 K, δ) 7.23–6.11 (m, 40H, C_6H_5), 4.19 (s, 2H, $\text{Ar}-\text{CH}_2-$), 3.06–1.72 (m, mainchain and *n*-butyl moieties, 12H), 1.53–0.55 (m, mainchain and *n*-butyl moieties, 22H).

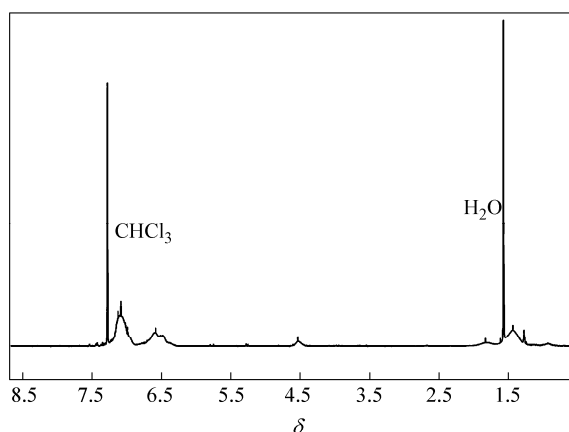


Fig. 4 ^1H -NMR spectrum (400 MHz, CDCl_3 , 298 K) of **3**

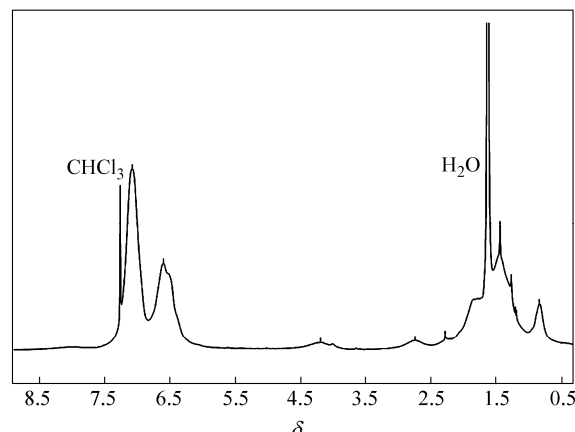


Fig. 5 ^1H -NMR spectrum (400 MHz, CDCl_3 , 298 K) of **2**

Characterizations of the Formation of the Supramolecular Crosslinked Polymer Gel

Compound **1** is a novel crown ether-functionalized TPE derivative, TPE-(B21C7)₄, which was synthesized *via* the esterification reaction of carboxy-modified TPE, TPE-(COOH)₄, with hydroxyl-functionalized B21C7. In TPE-(B21C7)₄, the outer four B21C7 units and the TPE core act as supramolecular recognizing functionalities and AIE-active motif, respectively. Polymer **2** was synthesized according to a previous literature. There were about 9.0 dialkylammonium salt moieties on a polymer chain for polymer **2** as calculated based on proton NMR spectra. It has been demonstrated that B21C7 can form [2]pseudorotaxanes with dialkylammonium salts^[51], thus a host-guest interactions crosslinked polymer network formed when **1** and **2** were mixed together.

To investigate the aggregation behavior driven by the B21C7/dialkylammonium salt host-guest interactions in solution, the mixture of **1** and **2** with equimolar B21C7 host moieties and dialkylammonium salt guest units was first characterized by measuring the solution viscosity (Fig. 6a) in chloroform using a Cannon-Ubbelohde semi-micro dilution viscometer. The specific viscosity of polymer **2** was also measured for comparison. Specific viscosity of polymer **2** versus concentration was almost linear over the concentration range of 1–8 g/dL. However, the specific viscosity of the mixture of **1** and **2** increased dramatically as the concentration increased, indicating that the B21C7/dialkylammonium salt recognition was responsible for the significant increase in viscosity^[54]. To confirm the formation of the supramolecular crosslinked polymer gel, rheological data were also collected to characterize the mixture of **1** and **2**. As shown in Fig. 6(b), the storage modulus (G') is larger than

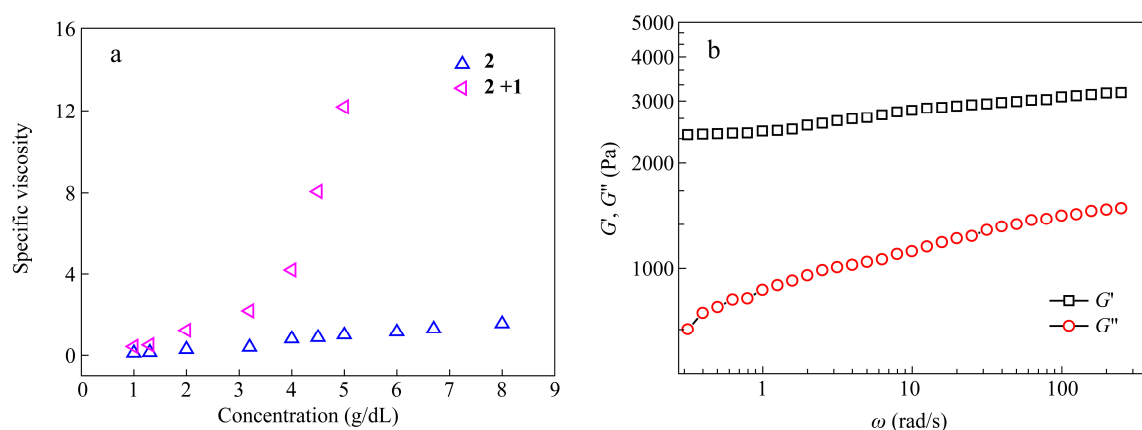


Fig. 6 (a) Specific viscosities of **2** and the mixture (1:1 molar ratio, B21C7/dialkylammonium salt) of **1** and **2**; (b) Storage modulus (G') and loss modulus (G'') versus frequency (ω) for the mixture (1:1 molar ratio, B21C7/dialkylammonium salt) of **1** and **2**

the loss modulus (G''), and the storage modulus G' is independent of the angular frequency ω , thereby indicating the gel formation^[15, 16]. Moreover, the supramolecular crosslinked polymer gel could be visualized macroscopically with the mixture of **1** and **2** in chloroform forming a gel, while a solution of polymer **2** without any specific interpolymer interaction could flow freely (Fig. 7a) at the same concentration of polymer **2** (5.00 mmol/L).

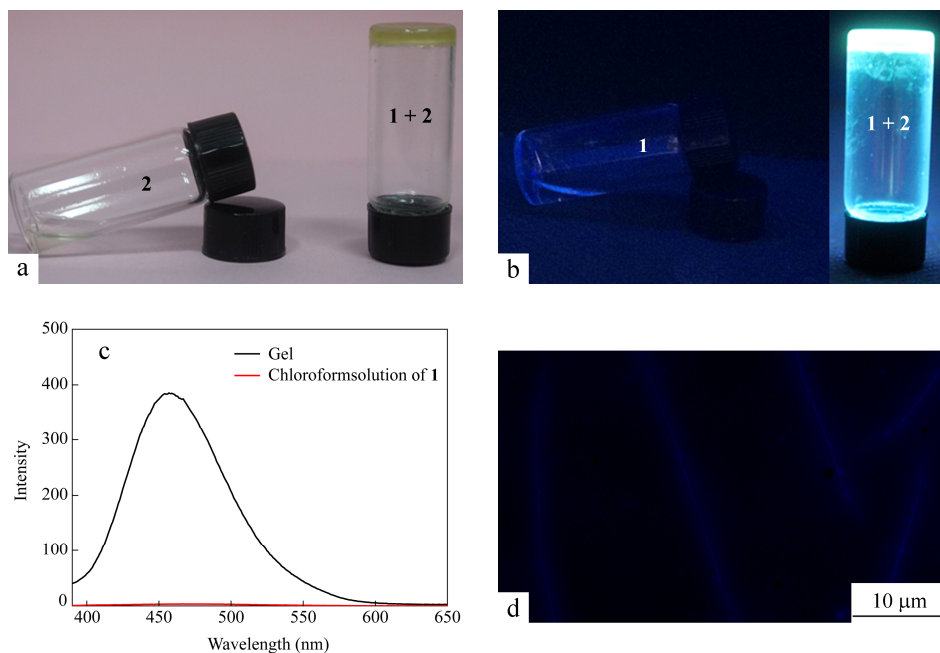


Fig. 7 (a) Photographic image of a solution of **2** (5.00 mmol/L) and a gel formed by self-assembly of **1** (1.25 mmol/L) and **2** (5.00 mmol/L); (b) Photographic image of a solution of **1** (1.25 mmol/L) and a gel formed by self-assembly of **1** (1.25 mmol/L) and **2** (5.00 mmol/L) observed upon UV irradiation at 365 nm; (c) Fluorescence spectra of the solution of **1** (1.25 mmol/L) and the gel formed by self-assembly of **1** (1.25 mmol/L) and **2** (5.00 mmol/L); (d) Fluorescence microscopy image of the electrospun nanofibers from a solution of **1** (1.00 mmol/L) and **2** (4.00 mmol/L).

Characterizations of the Fluorescent Supramolecular Crosslinked Polymer Gel

In order to compare the fluorescence intensity of the solution of **1** with that of the gel formed by the self-assembly of **1** and **2**, the fluorescences of both the gel and the solution of **1** with the same molar concentration of the TPE unit were examined (Fig. 7b). Figure 7(b) shows that the supramolecular crosslinked polymer gel was strongly fluorescent, while **1** was almost nonfluorescent in chloroform, indicating a peculiar example of “gelation induced fluorescence emission”^[10]. From the fluorescence spectra of the solution of **1** and the gel (Fig. 7c), it is obvious that the fluorescence of the gel was very strong and enhanced nearly 110 times compared with that of the solution of **1**. This remarkable fluorescence enhancement of the gel was owing to the AIE effect of TPE moieties. For the solution of **1**, the intramolecular rotation of phenyl rings of TPE induced the efficient nonradiative annihilation process and thus **1** was nearly nonemissive in solvent. However, for the gel, the intramolecular rotation of phenyl groups on TPE was hindered due to the aggregation through the B21C7/dialkylammonium salt host-guest interactions, which was the reason for the unique phenomenon of AIE^[10].

Interestingly, from a fluorescence microscopy photo (Fig. 7d), it can also be seen that the nanofibers electrospun from a solution of **1** and **2** exhibited intense blue fluorescence, resulting from the combination of B21C7/dialkylammonium salt host-guest interactions and the TPE-based AIE effect.

Responsiveness of the Fluorescent Supramolecular Crosslinked Polymer Gel

Because of the reversibility and environmental responsiveness of the B21C7/dialkylammonium salt host-guest interactions, the transitions between the fluorescent supramolecular crosslink polymer gel and the disassembled sol with very weak fluorescence were realized by adding pH and thermal stimuli (Fig. 8)^[51, 52]. As demonstrated in the previous studies, the B21C7/dialkylammonium salt recognition motif is sensitive to temperature^[52], so the reversible gel-sol transition could be controlled by the heating-cooling method. Accordingly, the change in the fluorescent intensity was also observed. Moreover, the solution pH value was increased by adding triethylamine (Et_3N) to deprotonate the dialkylammonium salt guest moieties^[52], losing their capacity of associating with the B21C7 host moieties and further dispersing the TPE molecules. After that, protonation of the dialkylammonium salt guest moieties was realized by adding trifluoroacetic acid to the solution^[52], recovering the host-guest interactions and aggregating the TPE molecules. This pH-controlled gel-sol transition also brought about the change in the fluorescent intensity.

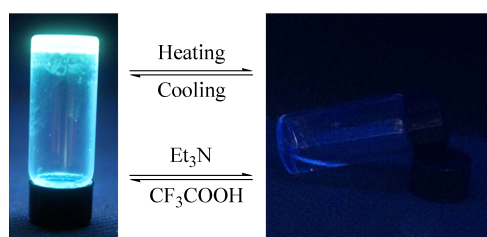


Fig. 8 Photographs demonstrating gel-sol processes of the polymer network using the heating-cooling method and changing pH

CONCLUSIONS

In summary, based on the combination of B21C7/dialkylammonium salt host-guest interactions and the TPE-based AIE effect, we have successfully prepared a fluorescent supramolecular crosslinked polymer gel. Compared with the solution of **1** with the same molar concentration of the TPE unit, this gel exhibited remarkable fluorescence enhancement due to the AIE effect of TPE moieties. The “gelation induced fluorescence emission” phenomenon can be explained by the hindered intramolecular rotation of phenyl rings of TPE. Because of the reversibility and environmental responsiveness of the B21C7/dialkylammonium salt host-guest interactions, the transitions between the fluorescent supramolecular crosslinked polymer gel and the disassembled sol with very weak fluorescence were realized by adding pH and thermal stimuli. This novel material contributes to the development of supramolecular chemistry, polymer science and fluorescent materials and offers a new method to construct functional supramolecular materials.

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