Super organogelator based on tetrathiafulvalene with four amide derivatives and its F$_4$TCNQ charge-transfer complex

Deyun Kong$^{a,b}$, Yan Xia$^a$, Dongfeng Li$^a$ and Ruibin Hou$^{a,b}$

$^a$School of Chemistry and Life Science, Changchun University of Technology, Changchun, P.R. China; $^b$Advanced Institute of Materials Science, Changchun University of Technology, Changchun, P.R. China

ABSTRACT

A new series of tetrathiafulvalene-based organogelators endowed with four hydrophobic chains incorporating amide groups was synthesised and characterised. The resulting transparent organogels were obtained with organic solvents such as cyclohexane, carbon tetrachloride and chlorobenzene. Additionally, the length of the alkyl chain influenced the gelation ability of organogels. Considering the results, we concluded that compounds were ‘super gelators’. Interestingly, the gelators reacted with 2,3,5,6-tetrafluoro-7,7,8,8-tetracyanoquinodimethane to form charge-transfer (CT) complexes and binary organogels. $^1$HNMR and FT-IR revealed that cooperation of hydrogen bonding, π–π and CT interactions was the main driving force for formation of the native and CT gels. The scanning electron microscopy images of native xerogels revealed characteristic gelation morphologies of three-dimensional cross-linking networks, whereas the morphologies of CT complex xerogels showed amorphous rod-like aggregates. X-ray powder diffraction studies suggested that both gelator and CT complex maintained lamellar molecular packing mode in organogel phase.

1. Introduction

In recent years, low molecular weight organogels (LMOGs), molecules whose molar mass is ≤3000, have attracted increasing interest because of their potential applications in the new soft material synthesis of inorganic nanomaterial templates, controlled drug release, light-harvesting systems, electronic devices, molecular logic gates and sensors. LMOGs are the materials used for gelator self-assembly through non-covalent interactions to form three-dimensional (3-D) networks that can immobilise a large amount of solvents. Generally, the cooperative effect of non-covalent forces mainly includes π–π stacking, hydrogen bonding, electrostatic forces and dipolar and van der Waals interactions. Therefore, particular functional sites, such as H-bond forming sites, π–π stacking units and long alkyl...
alkyl chains in the TTF terminal showed obvious differences on the gelling ability. For example, the gelator 4a, which contained n-dodecyl, could gelatinise cyclohexane and had the lowest critical gelation concentration (CGC) of 0.6 mg/mL. To the best of our knowledge, this is the lowest reported CGC value for TTF-based gelators. Interestingly, 4a–c could form stable binary gels with an electron acceptor (2,3,5,6-tetrafluoro-7,7,8,8-tetracyanoquinodimethane; F$_4$TCNQ) through charge-transfer (CT) and hydrogen bonding interactions.

2. Results and discussion

2.1. Synthesis

The synthetic procedures for target compounds 4a–c are summarised in Scheme 1. The reaction of bis(tetraethylammonium) bis (1,3-dithiole-2-thione-4,5-dithiol) zincate salt 1 with N-alkyl-2-chloroacetamide 2 in acetonitrile gave bipedal 1,3-dithiol-2-thione derivatives 3 in good yields (70–71%). Phosphite-mediated self-coupling of 3 eventually produced compounds 4a–c (50–53%).

2.2. Gelation behaviours

The gelation abilities of compounds 4a–c have been evaluated in a wide range of polar and apolar solvents by the ‘stable to inversion of the test-tube’ method and are summarised in Table S1 (Figure S1). The gelation test indicated that these compounds could all immobilise certain saturated hydrocarbons and aromatic solvents, such as cyclohexane, carbon tetrachloride and chlorobenzene, and formed transparent organogels, while other solvents formed opaque organogels (Figures 1(a)–(c) and S2–S4). Interestingly, organogels of 4a–c could entrap the guest chains, are necessary for gelation (19–21). The hydrogen bonding interaction, which has generated both theoretical and experimental interest in recent years, is an important interaction for the construction of supramolecular architectures such as amide, urea and hydroxyl groups (22–24). Tetrathiafulvalene (TTF) and its derivatives have been widely studied as strong electron donors in the research fields of field-effect transistors (25), sensors (26), liquid crystalline materials (27), conductors (28) and switches (29). Recently, several TTF-based LMOGs have been reported. The self-assembly of TTF derivatives through intermolecular interactions can lead to gelation of certain organic solvents that respond to oxidation/reduction reactions. For example, Becher et al. constructed oriented nanowires and organogels based on amphiphilic TTF-substituted macrocycles (30). Zhang et al. used the redox properties of the TTF moiety to tune gel–sol transition behaviours (31, 32). Amabilino et al. and Yin et al. have also detailed new gelators containing TTF moieties (33–38). Akutagawa et al. and Shinkai et al. separately described the organogels derived from TTF derivatives (39, 40). All of the results indicated that the use of intermolecular non-covalent interactions via a gelation process to construct TTF-based conducting bars is very appealing, and hydrogen bonding acts as an excellent scaffold to organise and hold the functional units.

Here, we describe a new series of LMOGs, 4a–c, based on an electroactive TTF (in Scheme 1). To enhance its gelation ability, four hydrophobic chains incorporating amide groups were connected to the TTF unit as the four hydrophobic chains incorporating amides likely facilitated the gelation through hydrophobic and multiple hydrogen bonding interactions. These compounds exhibited the typical redox chemistry for this family of compounds and formed thermo-reversible and stable organogels in saturated and aromatic hydrocarbons. Furthermore, different

Scheme 1. The synthetic route and molecular structures of gelators 4a–c.
molecules through intermolecular interactions. For example, 4b containing 0.25 wt% rhodamine B could gelate cyclohexane to form an organogel as indicated by their fluorescent colours (Figure 1(e)). Therefore, the system could be considered a binary organogel, which was also thermo-reversible. Once formed, the gel was stable in closed tubes for several months without crystallising or melting at ambient temperature.

The main characteristic of gels formed by small organic compounds is their thermo-reversible gel-to-sol transition that occurs by self-assembly and disassembly processes. The gel–sol phase transition temperature (Tgel) values of gelators having different chain lengths 4a–c at different concentrations in cyclohexane were determined to examine their thermostabilities by a convenient ball-drop method. In concurrence with the literature, the results showed that the Tgel values increased with increase in gelator concentration in cyclohexane (Figure 2). In cyclohexane, at the same concentration, the observed Tgel values of 4a–c decreased with the length of the end alkyl chains, thereby indicating that the length of the end alkyl chains had a pronounced influence on the formation and stability of the organogels.

An advantageous property of the donor–acceptor system is its ability to assemble and form a stable CT complex gel without forming a precipitate, which enhances the dimensionality of the electronic structure by non-covalent interactions. Interestingly, the CT complexes of 4 with F_{4}TCNQ could form binary gels through gelation of some saturated hydrocarbons. In fact, when 1 equiv. of F_{4}TCNQ was directly added on top of a cyclohexane gel of 4 under heating, the gel was gradually destroyed after several minutes and led to a green-yellow solution. Upon cooling, the solutions of CT complexes changed to the stable green-yellow binary gels (Figure 3). Furthermore, as seen in the solution system, UV–Vis spectra of the CT complex gels also showed two absorption bands in the 700–950 nm region (Figure S5), thus proving that the CT complex gels were undoubtedly formed.

### 2.3. Driving force analysis

The intermolecular π–π and S–S interactions caused by the TTF unit and multiple hydrogen bonding interactions attributed to the four amide units in 4 may be the driving force for the gelation. The $^1$HNMR spectra for the gel formed with compound 4b in d_{4}-o-dichlorobenzene at different temperatures and the corresponding solution at 393 K were measured (Figure 4). The signals around 6.58 ppm attributed to the protons of the amide units in 4b were gradually upfield shifted to 6.44 and 6.31 ppm, respectively, with increase in temperature as shown in

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**Figure 1.** (Colour online) Photographs of organogel 4b (a) 1.0 mg/mL in cyclohexane; (b) 1.3 mg/mL in carbon tetrachloride; (c) 5.7 mg/mL in benzene; and (d) with 0.25wt% rhodamine B in cyclohexane and the corresponding fluorescent organogel under UV light (wavelength: 365 nm).

**Figure 2.** (Colour online) Plots of T_{gel} vs. the concentration of 4 in cyclohexane.
Compounds 4a and 4c were also tested and a similar conclusion was also drawn by studying their FT-IR spectra shown in Figures S6–S7. The above-mentioned FT-IR Figure 4. This result implied that hydrogen bonding owing to the amide units in 4b existed in the gel phase, and it became weak after heating as anticipated. Furthermore, the signals around 3.58 ppm upfield shifted to 3.55 and 3.54 ppm, respectively, which were attributed to the protons of the –SCH2- in 4b, when the temperature was increased. This may be caused by the intermolecular π–π and S–S interactions caused by the TTF unit of 4b in the gel phase.

Additionally, the FT-IR spectra of the xerogel of 4b and its solution in CHCl3 were measured as depicted in Figure 5. The N–H stretching absorptions of amide units were shifted from 3447 and 3291 cm⁻¹ in solution to 3434 cm⁻¹ and 3290 cm⁻¹ in the gel state, and the C=O stretching absorption of amide units was shifted from 1645 to 1647 cm⁻¹, thereby suggesting that the hydrogen bonding between the amide groups was one of the main driving forces in the gelation. Similar spectral changes were observed both in the cyclohexane xerogel and the powder of 4b. The results are also shown in Figure 5.

Figure 6. (Colour online) FT-IR spectra of the CT complex xerogel of 4b with F4TCNQ (mole ratio = 1 : 1) from cyclohexane (a), native xerogel of 4b cyclohexane (b) and the F4TCNQ powder (c).

Compounds 4a and 4c were also tested and a similar conclusion was also drawn by studying their FT-IR spectra shown in Figures S6–S7. The above-mentioned FT-IR
spectral changes sufficiently demonstrated the presence of hydrogen bonding interactions of amide groups in the gel states.

In contrast, we focused our attention on the CN stretching band of F$_4$TCNQ in the CT complex gels (4 mM) in the FT-IR spectra (Figure 6). The CN stretching band of the 4b CT complex xerogel in cyclohexane appeared at 2193 cm$^{-1}$, exhibited a low frequency and shifted by 34 cm$^{-1}$ in comparison with that of F$_4$TCNQ, thus indicating that CT interaction had a key role in the formation of the CT complex gels. However, any shifts of the amide A, I and II bands at 3292, 1646 and 1554 cm$^{-1}$ in the CT complex gel with respect to the 4b xerogel were not observed, thereby implying that intermolecular hydrogen bonding interactions also had a key role in the formation of the CT complex gel.

2.4. Morphological characterisation

To investigate the differences between the aggregation mode of the transparent gel and that of the opaque gel, scanning electron microscopy (SEM) measurements were conducted using the cyclohexane xerogel, the n-hexane xerogel and the benzene xerogel of 4b (Figure 7). The SEM image of the cyclohexane xerogel of 4b (transparent) showed that the average length and width of the

Figure 7. SEM images of native xerogels of 4b from cyclohexane (a), 4b from benzene (b), 4c from n-hexane (c) and CT complex xerogel of 4b from cyclohexane (d).

Figure 8. XRD patterns of xerogel 4b from cyclohexane at a low concentration (0.6 mg/mL) (a) and xerogel 1b/F$_4$TCNQ (mole ratio = 1 : 1) from cyclohexane (b).
nanofibres were on the micrometer scale and approximately 200 nm, respectively. These results may indicate that molecules of compound 4b were self-assembled into fibrous aggregates that further piled to form cross-linking network structures. Similarly, the benzene and n-hexane xerogels of 4b (opaque) also had fibrous aggregates that cross-linked into 3-D networks, and the average width of the single fibres was approximately 0.8 μm. The width of fibers of 4b and 4c were thicker than that of 4a. The formation of uniform supramolecular aggregates suggested that the gelators were self-assembled in a hierarchical fashion to form a one-dimensional supramolecular polymer and larger superstructures in organogel systems. More interestingly, an evident change took place in the morphologies of the CT complex gels of gelators with F4TCNQ compared with those of the native gels, as shown in Figure 7(d). The supramolecular structures of native gels changed into amorphous rod-like aggregates. These changes in morphology showed that CT interaction played a key role in the formation of the CT complex gels.

As shown in Figure 8(a), the X-ray powder diffraction (XRD) pattern of the cyclohexane gel of 4b was characterised by reflection peaks at 6.76 and 3.41 nm−1 in the low-angle region. Their scatter vector ratio was 2:1, which could be indexed to (200), (100) diffractions. This result illustrated that a lamellar organisation with 1.84 nm of interlayer distance was formed in the gel phase. Considering that the molecular length of 4b was approximately 5.48 nm (by the CPK model), a mono-molecular oblique arrangement in the lamellar structure was expected. The reflection peak of q = 17.5 nm−1 in the wide-angle region was a typical π–π stacking distance of about 0.38 nm, which is characteristic of the π–π interaction between the individual molecules in the self-assembled structure. To reveal the change of molecular packing mode in CT complex gels, XRD analyses were conducted and the results are shown in Figures 8(b) and S8. Taking these XRD spectral data into account, the packing mode of 4b with F4TCNQ in the binary gels was discussed. The xerogels of CT complexes in cyclohexane exhibited diffraction peaks at 6.15 and 3.07 nm−1 in the low-angle region. Their scatter vector ratio was 2:1, which could be indexed to (200), (100) diffractions. This result illustrated that a lamellar organisation with 2.05 nm of interlayer distance was formed in the gel phase. Considering that the molecular length of 4b was approximately 5.48 nm (by the CPK model), a
monomolecular oblique arrangement in the lamellar structure was expected. On the basis of the XRD data, it was reasonable to suggest that the driving forces for gelation of CT complexes consisted mainly of a combined effect of intermolecular hydrogen bonding and CT interactions.

2.5. Electrochemical properties

The electrochemical properties of gelators 4a–c were evaluated by cyclic voltammetry (CV). CV measurements were performed in a dry dichloromethane solution of Bu4NBF4 (0.1 M) with a scan rate of 60 mV s⁻¹ at room temperature (Table 1). All gelators exhibited two reversible single-electron oxidation waves, which was the typical electrochemical behaviour of TTF. Moreover, the compounds showed a quasi-reversible reduction process. Both the first half-wave potential and second half-wave potential were also found to be higher than that of 2,3,6,7-tetrakis(methylthio) tetraphiafulvalene (TMT-TTF) because the introduction of four carbonylmethyl groups on the S atom of TTF resulted in a decrease in the electron-donating ability. The CV results indicated that there was less influence of the different alkyl chains on the solution electrochemical behaviour of the compounds.

2.6. Thermal properties

The thermal stability of a material is important for device longevity. Figure 10 shows the thermogravimetric analysis (TGA) of compounds 4a–c. TGA revealed relative thermal stability with an initial weight loss (5%) temperature of 221 °C that extended to 237 °C, which revealed that the gelators 4a–c had high thermal stability (Figure 10).

3. Conclusion

In summary, we have reported a new series of TTF-based, low molecular mass super gelators and demonstrated the supramolecular self-assembly process in common organic solvents. The gelators effectively gelated certain saturated hydrocarbons, which was attributed to the cooperative interplay of intermolecular π–π stacking, hydrogen bonding and S–S interactions as shown by FT-IR, 1H NMR and XRD studies. The gelation test results suggested that gelator 4a had the lowest CGC in cyclohexane. Furthermore, SEM showed that the morphology of the formed native xerogel cross-linked into 3-D networks. Importantly, the gelators could form stable CT complex gels with the stronger oxidant F4TCNQ rather than TCNQ, thereby indicating that the introduction of four amide groups to the TTF skeleton was effective in further reducing the electron-donating property. The CT complex gels were further confirmed by various spectroscopy, microscopy, XRD and electrochemistry techniques. The presented super gelators and their CT complexes, with excellent gelation ability and the inherent optoelectronic properties of TTF, may provide new opportunities in the development of ordered soft materials.

4. Experimental part

4.1. Materials and instruments

All chemicals were purchased from J&K Scientific (Beijing, China) and used as received without further purification. For chromatography, 100–200 mesh silica gel (Qingdao, China) was employed. Fluorescence spectra were determined on a fluorescence spectrophotometer (RF-540). NMR spectra were recorded on a Bruker AV-400 spectrometer and Infinity plus 400. Chemical shifts are reported in ppm using tetramethylsilane as the internal standard. The mass spectra were performed on a Shimadzu MALDI AXIMA-CFR×FE spectrometer. IR spectra were recorded on a Perkin Elmer 2400 instrument (KBr pressed disc method). MALDI-TOF-MS was performed on a Bruker Autoflex III...
smart bead using a 1,8,9-anthracenetriol (DITH) matrix. The SEM images were examined by JEOL JSM-5500LV. Compounds 1, 2 and 3 were synthesised according to our literature method (41, 42). The wt% is weight of rhodamine/(weight of cyclohexane + weight of rhodamine + weight of gelator) × 100%.

4.2. Synthesis

4.2.1. 2,3,6,7-tetrakis(acetododecylamidothio) tetrathedianulenevalene (4a)

Compound 3a (0.40 g, 0.62 mmol) was added to P(OEt)3 (15 mL) and the suspension was heated to 120 °C, causing dissolution within 1 min, leaving a yellow reaction mixture. The mixture was stirred for 5 h at 120 °C, cooled to room temperature. Addition of MeOH (50 mL) gave an orange solid, which was filtered, washed with MeOH and dried in vacuo to give 0.20 g. yield: 53%. M.p. = 197–198 °C. 1HNMR (CDCl3, 400 MHz) δ: 0.88 (t, J = 7.01 Hz, 12H), 1.36–1.17 (m, 112H), 3.25 (q, J = 6.89 Hz, 8H), 3.52 (s, 8H), 6.53 (s, 4H); FT-IR (KBr, cm−1): 3430, 3292, 2920, 1646, 1546, 1467, 1399, 721; MS (MALDI-TOF) m/z 1458.5 ([M+ 100], Calcd.: 1458.51.

4.2.2. 2,3,6,7-tetrakis(acetoxydodecanamidothio) tetrathedianulenevalene (4b)

Like 4a, orange powder, yield: 51%. M.p. = 177–198 °C. 1HNMR (CDCl3, 400 MHz) δ: 0.88 (t, J = 7.01 Hz, 12H), 1.35–1.15 (m, 112H), 3.25 (q, J = 6.89 Hz, 8H), 6.53 (s, 4H); FT-IR (KBr, cm−1): 3433, 3292, 2920, 1646, 1546, 1467, 1399, 721; MS (MALDI-TOF) m/z 1570.73 ([M+ 100], Calcd.: 1570.7.

4.2.3. 2,3,6,7-tetrakis(acetoxydodecanamidothio) tetrathedianulenevalene (4c)

Like 4a, orange powder, yield: 50%. M.p. = 128–129 °C. 1HNMR (CDCl3, 400 MHz) δ: 0.88 (t, J = 7.01 Hz, 12H), 1.36–1.17 (m, 128H), 3.26 (q, J = 6.89 Hz, 8H), 3.51 (s, 8H), 6.48 (s, 4H); FT-IR (KBr, cm−1): 3433, 3293, 2920, 1646, 1552, 1467, 1401, 720; MS (MALDI-TOF) m/z 1458.5 ([M+ 100], Calcd.: 1458.51.

Supplemental material

Supplemental data for this article can be accessed online here: http://dx.doi.org/10.1080/10610278.2016.1175565

Disclosure statement

No potential conflict of interest was reported by the authors.

Funding

This work was supported by the National Science Foundation of China [grant number 21442004]; the National Natural Science Foundation of Jilin Province [grant number 21502008].

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