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# Synthesis and self-assembly of novel hydrazide derivatives containing multi-alkoxy chains with different lengths

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ABSTRACT

We report on the synthesis and self-assembly of two novel types of hydrazide derivatives, e.g. 1,4-bis[(3,4,5-trialkoxyphenyl)-dihydrazide]-2,5-diethylphthalate (**C-Tn**, $n = 7$, 10, 12 and 16) and *N′*,*N′*-bis[3,4,5-tris(dodecyloxy)benzamido]-pyromellitic diimide (**BI-C-T12**). They were confirmed to exhibit strong gelation ability in several apolar organic solvents, such as benzene, 1,2-dichloroethane and chloroform. SEM images of the xerogels revealed that the gels consist of twist fibrous aggregates. Both FT-IR and $^1$H NMR studies confirmed that the intermolecular hydrogen bonding and van der Waals interactions were the major driving forces for the self-assembly of the molecules during gelling.



## 1. Introduction

Self-assembling processes are common throughout nature and technology [1]. Self-assembled materials, such as liquid crystals and organogels, formed by non-covalent bonding have attracted much attention because they are good candidates for the next generation of materials, for which dynamic function, environmental benignity, and low energy processing are required [2,3].

Organogels, in which organic solvents are gelled by low molecular-weight compounds (organogelators), have attracted much interest as a result of their unique features and potential applications for new organic soft materials [4]. Most of the organogelators reported in the literatures [5–10], can form a three-dimensional network by self-organization through non-covalent interactions such as hydrogen bonding, van der Waals interactions, π-stacking, and coordination. In our previous papers, we reported the gelatinization behavior of a series of wedge-shaped [11], twin-tapered [12] and side-on [13] hydrazide derivatives with different alkoxy chains.

Here, we report the synthesis of the newly designed hydrazide derivatives, e.g., 1,4-bis[(3,4,5-trialkoxyphenyl)-dihydrazide]-2,5-diethylphthalate (**C-Tn**, $n = 7$, 10, 12 and 16) and *N′*,*N′*-bis[3,4,5-tris(dodecyloxy)benzamido]-pyromellitic diimide (**BI-C-T12**) and their gelatinization behaviors. Our results showed that the intermolecular hydrogen bonding between –C═O group and –N-H group and van der Waals interactions are the main driving forces for the self-assembly of the molecules.

## 2. Experimental

### 2.1. Characterization

Melting point (mp) was measured on a WRX-1S instrument. Infrared (IR) spectra were recorded with a Perkin-Elmer spectrum one B spectrometer. $^1$H NMR spectra were recorded on a Varian-Unity spectrometer at 300 MHz using tetramethylsilane (TMS) as an internal standard. The FE-SEM observation was carried out using a JSM-6700F field emission scanning electron microscope. The samples were dried overnight in a vacuum before the observation. The dried gels were sputtered using a gold target.

*Gelation test:* A mixture of a weighed gelator in organic solvents (1 mL) in a sealed test tube was heated around the boiling point until a clear solution appeared. The test tube was allowed to stand for 4 h in refrigeratory thermostated 5 °C. The gel formation was evaluated by the "stable to inversion of a test tube" method [14].

### 2.2. Synthesis

For all the reactions, magnetic stirrers were used. Commercially available reagents were used without further purification. All reactions were monitored by thin layer chromatography (TLC). Compounds were visualized with UV light at 254 nm and 365 nm.

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Two types of hydrazide derivatives named as **C-Tn** ($n$ = 7, 10, 12 and 16) and **BI-C-T12** were synthesized according to the synthetic route in Scheme 1. The chemical structures of the hydrazide compounds were confirmed by FT-IR, $^1$H NMR spectroscopy and elemental analysis.

*2.2.1. Synthesis of 1,4-bis[(3,4,5-trialkoxyphenyl)-dihydrazide]-2,5-diethylphthalate (**C-Tn**, n = 7, 10, 12 and 16)*

*2.2.1.1. 3,4,5-Triheptyloxybenzoic hydrazide (**T7**).* Methyl gallate (MG) (3.68 g, 0.02 mol) was dissolved in 80 mL dry acetone, 1-bromoheptane (10.8 g, 0.06 mol), anhydrous potassium carbonate ($K_2CO_3$) (15 g), KI (1.0 g), were added. The reaction mixture was refluxed for about 50 h. The hot reaction mixture was filtered and the residue was thoroughly washed twice with acetone. The solvent was removed from the combined filtrates in a rotary evaporator, and the residual product (3,4,5-triheptyloxybenzoic acid methyl ester) was collected. The ester and hydrazine monohydrate (20 mL) were added into 40 mL ethanol, and the mixture was refluxed for about 50 h. The reaction mixture was then frozen and the product precipitated. The product was filtered and recrystallized from methanol to give 4.9 g white solid (yield 63%, mp 107–108 °C).

$^1$H NMR (*d*-DMSO ppm from TMS, 300 MHz) 10.71 (s, 1H), 7.16 (s, 4H), 4.40 (s, 2H), 3.80 (t, 2H, $J$ = 4.5 Hz), 3.78 (t, 1H), 1.65-1.88 (m, 24H), 1.00 (t, 9H, $J$ = 9.0 Hz).

FT-IR (KBr disc cm$^{-1}$) 3317, 3258, 2954, 2928, 1625, 1580, 1519, 1498, 1466, 1425, 1390, 1348, 1303, 1242, 1116, 1069, 1041, 1011, 949, 921, 861, 842, 810, 788, 767, 752, 724, 663, 590.

*2.2.1.2. 3,4,5-Tridecyloxybenzoic hydrazide (**T10**).* White powder (yield 62%, mp 112–113 °C). $^1$H NMR ($CDCl_3$ ppm from TMS, 300 MHz) 10.71 (s, 1 H), 7.16 (s, 4H), 4.39-4.40 (s, 2H), 3.80 (t, 2H, $J$ = 4.5 Hz), 3.78 (t, 1H), 1.65–1.88 (m, 42H), 1.00 (t, 9H, $J$ = 9.0 Hz).

FT-IR (KBr disc cm$^{-1}$) 3265, 2954, 2920, 2850, 1627, 1581, 1526, 1498, 1468, 1425, 1389, 1347, 1242, 1120, 991, 859, 786, 757, 720, 664.

*2.2.1.3. 3,4,5-Tridodecyloxybenzoic hydrazide (**T12**).* White powder (yield 60%, mp 114–115 °C). $^1$H NMR ($CDCl_3$ ppm from TMS, 300 MHz) 10.71 (s, 1H), 7.16 (s, 4H), 4.39-4.40 (s, 2H), 3.80 (t, 2H, $J$ = 4.5 Hz), 3.78 (t, 1H), 1.65-1.88 (m, 54H), 1.00 (t, 9H, $J$ = 9.0 Hz).

FT-IR (KBr disc cm$^{-1}$) 3265, 2954, 2920, 2850, 1627, 1581, 1526, 1498, 1468, 1425, 1389, 1347, 1242, 1120, 991, 859, 786, 720, 664.

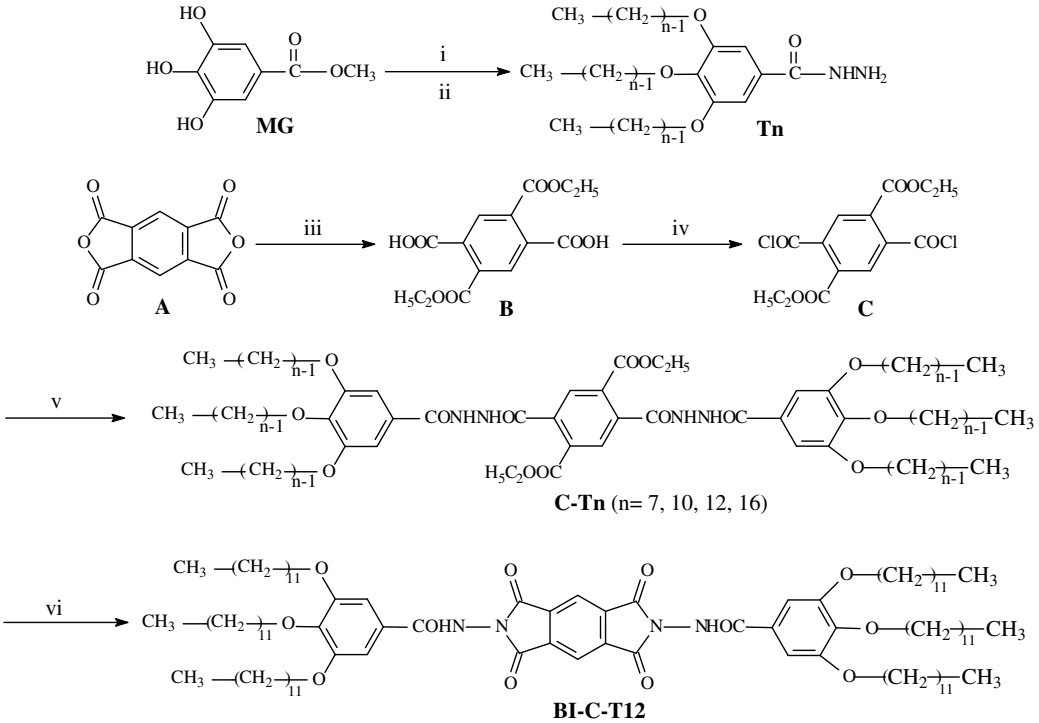
*2.2.1.4. 3,4,5-Tricetyloxybenzoic hydrazide (**T16**).* White powder (yield 63%, mp 117–118 °C). $^1$H NMR ($CDCl_3$ ppm from TMS, 300 MHz) 10.71 (s, 1 H), 7.16 (s, 4H), 4.39–4.40 (s, 2H), 3.80 (t, 4H, $J$ = 4.5 Hz), 3.78 (t, 2H), 1.65–1.88 (m, 78H), 1.00 (t, 9H, $J$ = 9.0 Hz).

FT-IR (KBr disc cm$^{-1}$) 3265, 2954, 2920, 2850, 1627, 1581, 1498, 1468, 1425, 1389, 1347, 1242, 1120, 991, 859, 786, 720, 665.

*2.2.1.5. Diethyl 2,5-dicarboxylterephthalate (**B**).* Phthalic anhydride (**A**) (4.36 g, 20 mmol) was dissolved in 120 mL ethanol, and the mixture was refluxed for about 8 h [15]. After ethanol being vaporized, the reaction mixture was frozen for about 24 h and the crude product precipitated. The precipitate was filtered, washed thoroughly with water, dried completely, and recrystallized from ether to give 2.75 g white crystal (yield 44%, mp 280–281 °C).

$^1$H NMR (*d*-DMSO ppm from TMS, 300 MHz) 13.82 (s, 2H), 7.98 (s, 2H), 4.29-4.31 (q, 4H), 1.29 (t, 6H, $J$ = 6.0 Hz).

FT-IR (KBr disc cm$^{-1}$) 2985, 2960, 2941, 2666, 2581, 1731, 1702, 1505, 1496, 1432, 1391, 1369, 1304, 1258, 1163, 1142, 1105, 1018, 918, 860, 794, 736, 654, 574.




**Scheme 1.** Synthetic route for novel hydrazide derivatives. Reagents and conditions: (i) $C_nH_{2n+1}Br$, acetone, $K_2CO_3$, reflux 50 h; (ii) hydrazine monohydrate, ethanol, reflux, 50 h; (iii) ethanol, reflux 8 h; (iv) $SOCl_2$, reflux 15 h; (v) Tn, THF, pyridine, rt 10 h; (vi) pyridine/toluene (1/5, V/V), reflux 96 h.

*2.2.1.6. 1,4-Bis[(3,4,5-triheptyloxyphenyl)-dihydrazide]-2,5-diethylphthalate (**C-T7**).* Compound **B** (1.55 g, 5 mmol) was dissolved in thionyl chloride ($SOCl_2$) and refluxed for 15 h. The superfluous $SOCl_2$ was vaporized from the reaction mixture to get terephthaloyl chloride (**C**). Compound **C** was dissolved in 70 mL tetrahydrofuran (THF), then 3,4,5-triheptyloxybenzoic hydrazide (4.78 g, 0.01 mol) and 3 mL pyridine was added, the reaction mixture was cooled to 0–4 °C by using an ice-bath and stirred for about 8 h. The mixture was poured into 500 mL water and product was collected through filtration and dried completely. The product was recrystallized from chloroform to give 4.15 g white powder (yield 68%, mp 225–226 °C).

$^1$H NMR (*d*-DMSO ppm from TMS, 300 MHz) 10.34 (s, 2H), 10.01 (s, 2H), 7.96 (s, 2H), 7.02 (s, 4H), 4.30 (q, 4H), 3.96 (t, 2H, *J* = 6.0 Hz), 3.80 (t, 4H), 1.60–1.76 (m, 12H), 1.27–1.47 (m, 66H), 0.88 (t, 18H, *J* = 3.0 Hz).

FT-IR (KBr disc cm$^{-1}$) 3183, 2955, 2926, 2856, 1728, 1666, 1600, 1578, 1498, 1455, 1383, 1337, 1299, 1236, 1131, 1117, 1021, 914, 851, 826, 772, 733, 665, 538.

Anal. Calcd for $C_{70}H_{110}N_4O_{14}$: C, 68.29%; H, 8.94%; N, 4.55%. Found: C, 68.33%; H, 9.14%; N, 4.45%.

*2.2.1.7. 1,4-Bis[(3,4,5-tridecyloxyphenyl)-dihydrazide]-2,5-diethylphthalate (**C-T10**).* White powder (yield 59%, mp 214–215 °C). $^1$H NMR ($CDCl_3$ ppm from TMS, 300 MHz) 10.70 (s, 2H), 10.60 (s, 2H), 7.86 (s, 2H), 6.97 (s, 4H), 4.24-4.26 (q, 4H), 3.92 (t, 2H, *J* = 6.0 Hz), 3.67 (t, 4H), 1.67-1.79 (m, 12H), 1.26-1.47 (m, 90H), 0.88 (t, 18H, *J* = 4.5 Hz).

FT-IR (KBr disc cm$^{-1}$) 3311, 3178, 2954, 2923, 2870, 2852, 1730, 1670, 1603, 1579, 1498, 1456, 1383, 1338, 1299, 1238, 1173, 1118, 1046, 1020, 986, 916, 842, 763, 721, 664, 595.

Anal. Calcd for $C_{88}H_{146}N_4O_{14}$: C, 71.26%; H, 9.85%; N, 3.78%. Found: C, 71.52%; H, 10.14%; N, 3.57%.

*2.2.1.8. 1,4-Bis[(3,4,5-tridodecyloxyphenyl)-dihydrazide]-2,5-diethylphthalate (**C-T12**).* White powder (yield 53%, mp 190–191 °C). $^1$H NMR ($CDCl_3$ ppm from TMS, 300 MHz) 10.37 (s, 2H), 10.15 (s, 2H), 7.96 (s, 2H), 7.01 (s, 4H), 4.28-4.30 (q, 4H), 3.95 (t, 2H, *J* = 6.0 Hz), 3.78 (t, 4H), 1.61-1.72 (m, 12H), 1.26-1.47 (m, 114H), 0.88 (t, 18H, *J* = 4.5 Hz).

FT-IR (KBr disc cm$^{-1}$) 3313, 3178, 2954, 2922, 2851, 1730, 1672, 1603, 1579, 1497, 1457, 1388, 1339, 1300, 1241, 1119, 1045, 1017, 895, 842, 721, 663, 538.

Anal. Calcd for $C_{100}H_{170}N_4O_{14}$: C, 72.73%; H, 10.30%; N, 3.39%. Found: C, 72.91%; H, 10.45%; N, 3.05%.

*2.2.1.9. 1,4-Bis[(3,4,5-tricetyloxyphenyl)-dihydrazide]-2,5-diethylphthalate (**C-T16**).* White powder (yield 48%, mp 174–175 °C). $^1$H NMR ($CDCl_3$ ppm from TMS, 300 MHz) 10.01 (s, 2H), 9.78 (s, 2H), 8.04 (s, 2H), 7.03 (s, 4H), 4.31–4.33 (q, 4H), 3.96 (t, 2H, *J* = 3.0 Hz), 3.86 (t, 4H), 1.62–1.73 (m, 12H), 1.26–1.35 (m, 162H), 0.87 (t, 18H, *J* = 7.5 Hz).

FT-IR (KBr disc cm$^{-1}$) 3183, 2984, 2918, 2870, 2850, 1728, 1670, 1603, 1579, 1498, 1468, 1426, 1383, 1337, 1300, 1239, 1174, 1123, 1046, 1016, 985, 969, 917, 842, 770, 720, 663, 593.

Anal. Calcd for $C_{124}H_{218}N_4O_{14}$: C, 74.92%; H, 10.98%; N, 2.92%. Found: C, 74.99%; H, 10.99%; N, 2.52%.

*2.2.2. Synthesis of N′,N′-bis[3,4,5-tris(dodecyloxy)benzamido]-pyromellitic diimide (**BI-C-T12**)*

Compound **C-T12** (1.81 g, 0.01 mol) was dissolved in 150 mL pyridine/toluene (1:5, V/V) and stirred at 80 °C for about 96 h. The superfluous solvent was evaporated from the reaction mixture, then cooled residue was poured into 150 mL water and product was collected through filtration and dried completely. The product was recrystallized from dichloromethane to give 0.97 g pale yellow solid (yield 62%, mp 173–174 °C).

$^1$H NMR ($CDCl_3$ ppm from TMS, 300 MHz) 8.45 (s, 2H), 8.06 (s, 2H), 7.10 (s, 4H), 4.03 (t, 12H, *J* = 6.0 Hz), 1.62-1.84 (m, 12H), 1.27-1.47 (m, 114H), 0.88 (t, 18H, *J* = 4.5 Hz).

FT-IR (KBr disc cm$^{-1}$) 3255, 2923, 2852, 1793, 1750, 1663, 1585, 1492, 1468, 1429, 1390, 1338, 1221, 1119, 1021, 914, 840, 760, 721, 709, 605, 559.

Anal. Calcd for $C_{96}H_{158}N_4O_{12}$: C, 73.94%; H, 10.14%; N, 3.59%. Found: C, 73.96%; H, 10.16%; N, 3.39%.

## 3. Results and discussion

### 3.1. Synthesis

The synthetic route for the novel hydrazide derivatives is shown in Scheme 1. Compound **C-Tn**'s were prepared through the condensation of terephthaloyl chloride (**C**) with 3,4,5-trialkoxybenzoic hydrazide (**Tn**), while **BI-C-T12** was obtained based on intramolecular dealcoholizing of **C-T12**.

As can be seen from the synthetic scheme, partially esterification of the phthalic anhydride (**A**) results in a mixture of diethyl 2,5-dicarboxylterephthalate (**B**), diethyl 2,4-dicarboxylterephthalate (B') containing two carboxyls and other by-products. Most B' and other esterifiable products can be easily removed from the mixture through recrystallization from ethanol [15]. Because of good solublility in ether than that of compound **B**, compound **B'** can be completely eliminated from the mixture of **B** and **B'**, then the pure compound **B** being obtained. The diimide compound **BI-C-T12** was converted from the amic ester **C-T12** in solvent (pyridine/toluene, Volume ratio: 1:5) by intramolecular dealcoholizing condensation under feeblish alkalinity condition. And the state of compound **BI-C-T12** took on evenly graininess solid via the new and succinct step, compared to that of traditional means of diimide [16].

### 3.2. Self-assembly of hydrazide derivatives

The organogelation property of the hydrazide derivatives were tested in several solvents and the minimum gel concentration (MGC) of both **C-Tn** and **BI-C-T12** in benzene, 1,2-dichloroethane and chloroform were listed in Table 1. Compared with that of **BI-C-T12**, **C-Tn** showed lower MGC indicating that **C-Tn**'s are more effective as gelators than **BI-C-T12**. The MGCs of **C-Tn**'s decreased with the increase of length of the terminal alkyl chains, suggesting that **C-Tn** with longer terminal chains possessed better gel ability.

To obtain visual insights into the aggregation mode of these gelators in organogels, the image observation of the xerogels was performed on a field emission scanning electron microscopy (FE-SEM). Fig. 1 shows the SEM images of the xerogels of **C-T12** and **BI-C-T12** from 1,2-dichloroethane. It can be seen that the SEM image of **C-T12** xerogels consists of bundles of fibers which are entangled to for network which are responsible for sustaining the solvents therein. The fiber diameters are 100 nm. Formation of long fibers indicates that strong directional intermolecular interaction

**Table 1**
The minimum gel concentrations (MGC) of **C-Tn** and **BI-C-T12**

| Solvent | MGC (wt%) | | | | |
|---|---|---|---|---|---|
| | C-T7 | C-T10 | C-T12 | C-T16 | BI-C-T12 |
| Benzene | 2.21 | 1.94 | 1.54 | 1.51 | 3.06 |
| 1,2-Dichloroethane | 1.96 | 1.62 | 1.48 | 1.45 | 2.28 |
| Chloroform | Solution | 1.85 | 1.69 | 1.63 | Solution |

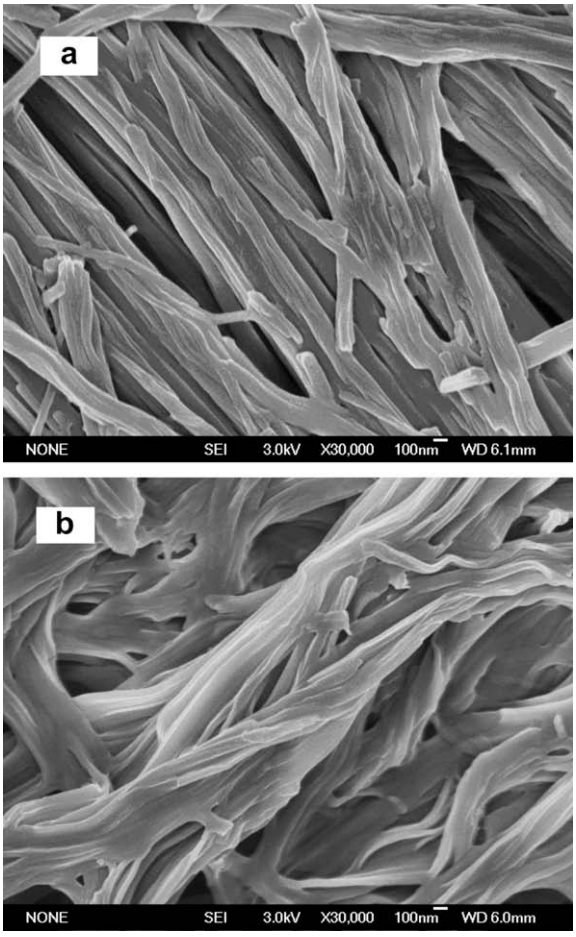



**Fig. 1.** FE-SEM images of **C-T12** (a) and **BI-C-T12** (b) xerogels from 1,2-dichloroethane.

might exist, which are the driving forces for the self-assembly of the molecules.

In order to explore the driving forces for the self-assembly of the molecules, Fourier-transform infrared (FT-IR) and concentration dependent $^1$H NMR spectroscopic experiments were performed.

The formation of elongated fiber-like aggregates indicates that the self-assembly of **C-T12** is driven by strong directional intermolecular interactions. To ascertain whether hydrogen bonding plays a role in the gelation process, FT-IR spectrum of the **C-T12** organogel was examined. The presence of –N–H stretching vibrations at 3175 cm$^{-1}$ and –C═O stretching vibrations at 1670 cm$^{-1}$ for **C-T12** in the gel state unambiguously suggested that the hydrogen bonding through –N–H. . .O═C– exists in the gelation process. As are the –N–H and –C═O stretching vibrations for **BI-C-T12** in the gel state (as seen in Fig. 2).

The hydrogen bonding was further confirmed to be the intermolecular one through the $^1$H NMR studies. These results strongly indicated that –N–H groups were exclusively involved in intermolecular hydrogen bonding [17] and play an important role in the gelation process. In the $^1$H NMR dilution studies, the amidic protons of compounds **C-Tn** showed a strong concentration dependence, for example, reducing the concentration of **C-T12** in CDCl3 from $10^{-1}$M to $10^{-2}$ M causes both NH-1 (near to alkoxy phenyl, $\Delta\delta$ = 1.00 ppm) and NH-2 (near to central phenyl, $\Delta\delta$ = 0.84 ppm) to shift upfield remarkably (as seen in Fig. 3), which strongly indicates that the two amide protons in **C-T12** participate in intermolecular hydrogen bonding.

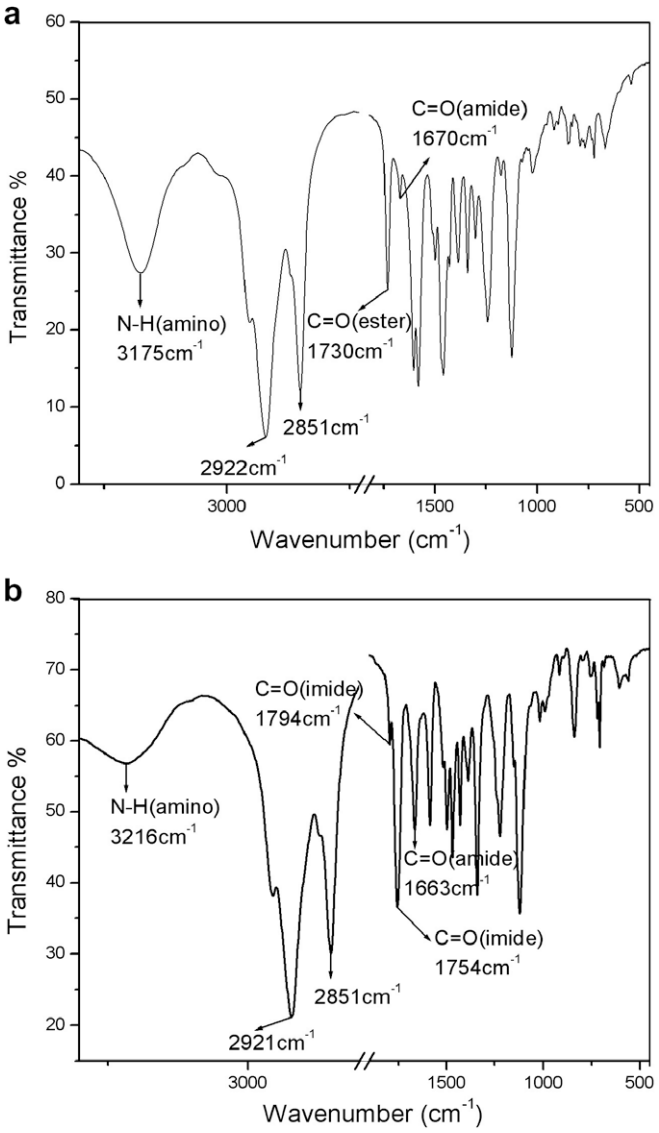



**Fig. 2.** FT-IR spectra of **C-T12** (a) and **BI-C-T12** (b) xerogels from 1,2-dichloroethane.

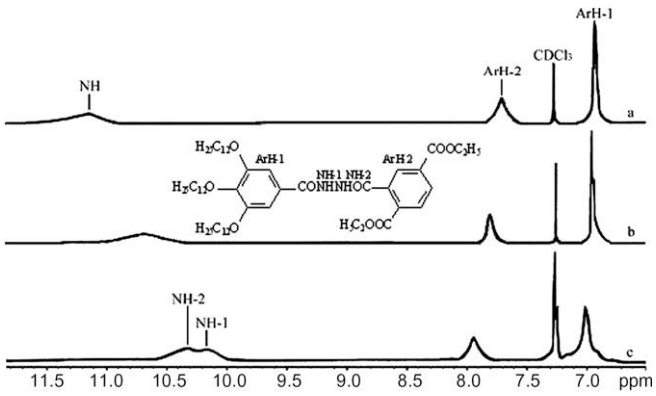



**Fig. 3.** Set of partial $^1$H NMR spectra of **C-T12** in different concentration $CDCl_3$ at room temperature (300 MHz): (a) $10^{-1}$ M, (b) $5 \times 10^{-2}$ M, (c) $10^{-2}$ M. The inset represented half molecular structure of **C-T12**.

Furthermore, the antisymmetric ($\nu_{as}$) and symmetric ($\nu_s$) $CH_2$ stretching vibrational modes of **C-Tn** are utilized to probe alkyl

**Table 2**
Summary of $-CH_2-$ stretching vibrations of hydrazide derivatives in xerogels

| Assignments | IR frequencies ($cm^{-1}$) | | | | |
|---|---|---|---|---|---|
| | **C-T7** xerogel | **C-T10** xerogel | **C-T12** xerogel | **C-T16** xerogel | **BI-C-T12** xerogel |
| $\nu_{as}(CH_2)$ | 2924 | 2923 | 2922 | 2929 | 2921 |
| $\nu_s(CH_2)$ | 2853 | 2852 | 2851 | 2859 | 2851 |

chain conformations. With the end-on alkyl chain length prolonging, for **C-Tn** xerogel (except for **C-T16** [18]), the $\nu_{as}(CH_2)$ and $\nu_s(CH_2)$ appear in the ranges of 2921–2924 and 2851–2853 $cm^{-1}$ (Table 2), indicating a significant population of the trans conformation [19]. The lower wavenumber shift reveals a decrease in the fluidity of the alkyl chains due to the strong organization of the alkyl groups via van der Waals interaction. Consequently, the driving forces for organogelation followed by entanglement of the self-assembled nanofibers are mainly hydrogen bonding and van der Waals interactions.

In many cases, organogelations need a heating dissolution process. The organogels show the same structural properties (FT-IR and $^1H$ NMR) as those made by the hydrazide derivatives and the same gel properties such as the thermoreversibilities. Furthermore, this procedure can apply for some solvents that gelators **C-Tn** and **BI-C-T12** can gel, as shown in Table 1. In summary, we revealed the effects of hydrogen bonding and van der Waals forces on the organogelation using new hydrazide-based gelators with various numbers of hydrogen bonding sites as well as alkyl chain lengths and the organogel formation at room temperature. For the hydrazide-based organogelators, gelator **C-T12** possessing the four potential hydrogen bonding sites and dodecyl groups at both terminals has the best organogelation ability. Using 1,2-dichloroethane and benzene as the vectors for organogelators **C-Tn** and **BI-C-T12** playing their roles, organogelation at room temperature can be achieved.

## 4. Conclusions

Based on the SEM observations, FT-IR and $^1H$ NMR spectroscopy methods, we studied the self-assembly of the hydrazide compounds **C-Tn** and **BI-C-T12** in solution. Investigating the minimum concentrations of gelling, aggregation morphology and structure of gels, and gelation driving forces, the results showed, (1) intermolecular hydrogen bonding origining from –C═O group and –N–H group existed in hydrazide compounds **C-Tn** and **BI-C-T12**, respectively, and it could freeze organic solvent forming gel; (2) the change of the number of hydrogen bonding donor-acceptor and the lengths of alkyl chains of hydrazide derivatives affected gelling ability remarkably, it indicated that intermolecular hydrogen bonding and van der Waals interactions were the main driving forces for the formation of self-assembled gel. Furthermore, the longer the end-on attached alkyl chains, the stronger the gelation ability.

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