Electronic Supplementary Information for

Heat-set gel formed from easily accessible gelators of succinamic acid derivative (SAD) and primary alkyl amine (R-NH_2)

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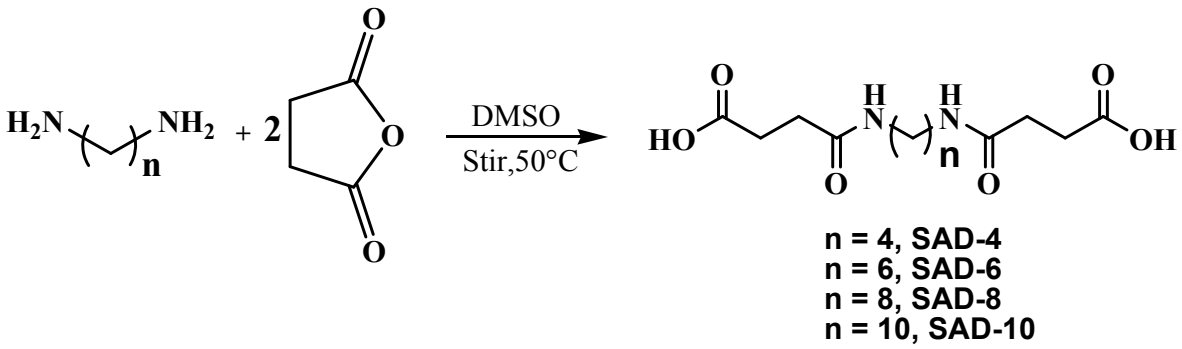
1）Synthesis and characterization of succinamic acid derivatives (SAD-4, SAD-6, SAD-8, SAD-10)



Scheme S1 The synthesis of succinamic acid derivatives: SAD-4, SAD-6, SAD-8, SAD-10

All starting materials were available from commercial suppliers and used without further purification. SAD-4 was synthesized as follows: A mixture of 1,4-butanediamine (0.1 mol) and butanedioic anhydride (0.2 mol) was dissolved in 15 mL DMSO in a round-bottomed flask equipped with a condenser. After stirring for about 4 h at 50 °C the solution was mixed with H_2O (45 mL). White solid was obtained after filtration. The method for preparing SAD-6, SAD-8, and SAD-10 is similar to that of SAD-4 except using 1,6-hexanediamine, 1,8-octadiamine and 1,10-diaminodecane instead of 1,4-butanediamine, respectively. Highly pure products were gotten after recrystallized in mixed solvents of ethanol/water (v/v = 1:1) for four times, respectively. The corresponding satisfactory ^{1}HNMR, FT-IR, melting point and mass spectrum data were showed below.

SAD-4: N-[4-(3-Carboxy-propionylamino)-butyl]-succinamic acid

White solid. mp: 181-183℃. Yield >60%.
^{1}H NMR (400MHz, DMSO-d_6, δ, ppm): 12.05 (S, 2H), 7.80 (t, J = 5.4 Hz, 2H), 3.0 (d, J = 5.6 Hz, 4H), 2.40 (t, J = 6.8 Hz, 4H), 2.27 (t, J = 7.0 Hz, 4H), 1.35 (t, J = 2.8 Hz, 4H).
IR (KBr)：3311, 3066, 2944, 2880, 1696, 1632, 1547, 1470, 1424, 1325, 1280, 1198, 1037, 917, 801, 695, 638, 546 cm^{-1}
MS-EI [M-H]$^-$ calcd. for $C_{12}H_{20}N_2O_6$: m/z 288.1; found: 287.1.

SAD-6: N-[6-(3-Carboxy-propionylamino)-hexyl]-succinamic acid

White solid. mp: 180-182℃. Yield >50%.
^{1}H NMR (400MHz, DMSO-d_6, δ, ppm): 12.02 (S, 2H), 7.79 (t, J = 5.8 Hz, 2H), 3.02-2.97 (m, 4H), 2.49 (t, J = 1.6 Hz, 4H), 3.39 (t, J = 7.0 Hz, 4H), 2.27 (t, J = 8.0 Hz, 4H), 1.35 (t, J = 6.4 Hz, 4H).
IR (KBr)：3311, 3057, 2936, 2867, 1696, 1640, 1537, 1471, 1421, 1313, 1250, 1185, 945, 693, 637, 561, 482 cm^{-1}
MS-EI [M-H]$^-$ calcd. for $C_{14}H_{24}N_2O_6$: m/z 316.2; found: 315.1.

SAD-8: N-[8-(3-Carboxy-propionylamino)-octyl]-succinamic acid

White solid. mp: 175-177℃. Yield >50%.
^{1}H NMR (400MHz, DMSO-d_6, δ, ppm): 12.02 (S, 2H), 7.78 (t, J = 5.4 Hz, 2H), 3.01-2.97 (m, 4H), 2.41-2.38 (m, 4H), 2.27 (t, J = 7.0 Hz, 4H), 1.35 (t, J = 6.4 Hz, 4H), 1.22 (m, 8H).

IR (KBr)：3312, 3058, 2931, 2857, 1696, 1640, 1537, 1470, 1422, 1319, 1277, 1196, 951 cm^{-1}
MS-EI $[M-H]^-$ calcd. for $C_{16}H_{28}N_2O_6$: m/z 344.2; found: 343.1.

SAD-10: N-[10-(3-Carboxy-propionylamino)-decyl]-succinamic acid

White solid. mp: 174-176℃. Yield >50%.
^1H NMR (400MHz, DMSO-d_6, δ, ppm): 12.01 (S, 2H), 7.78 (t, J = 5.6 Hz, 2H), 3.01-2.96 (m, 4H), 2.39 (t, J = 7Hz, 4H), 2.27 (t, J = 7.0 Hz, 4H), 1.35 (t, J = 6.2 Hz, 4H), 1.22 (s, 12H).
IR (KBr)：3315, 3055, 2928, 2854, 1695, 1640, 1536, 1471, 1423, 1314, 1273, 1197, 946, 799, 692, 635, 583, 501 cm^{-1}
MS-EI $[M-H]^-$ calcd. for $C_{18}H_{32}N_2O_6$: m/z 372.2; found: 371.2.

2) Gelation data

Table S1 Gelation data of succinamic acid derivatives (**SAD-4, SAD-6, SAD-8, SAD-10**)

	Compounds	SAD-n			
	Solvents	**SAD-4**	**SAD-6**	**SAD-8**	**SAD-10**
1	Tetralin	P	P	P	P
2	Benzene	P	P	P	P
3	Toluene	P	P	P	P
4	o-Xylene	P	P	P	P
5	m-xylene	P	P	P	P
6	p-xylene	P	P	P	P
7	Mesitylene	P	P	P	P
8	Nitrobenzene	P	P	P	P
9	Chlorobenzene	P	P	P	P
10	o-Dichlorobenzene	P	P	P	P
11	H_2O	S	P	P	P
12	ethyl acetate	P	P	P	P
13	Ethanol	S	P	P	P
14	n-amyl alcohol	P	P	P	P
15	Methanol	S	P	P	P
16	Chloroform	P	P	P	P
17	Isopropyl ether	P	P	P	P
18	Cyclohexane	P	P	P	P
19	DMSO	S	S	S	S
20	Petroleum ether	S	S	P	P
21	Ethylene glycol	S	S	P	P
22	Methylene Chloride	P	P	P	P

S = solution; P = precipitate.

Table S2 Gelation data of primary alkyl amine R-NH_2 (**R_{10}, R_{12}, R_{14}, R_{16}, R_{18}**)

	Compounds / Solvents	R-NH_2				
		R_{10}	**R_{12}**	**R_{14}**	**R_{16}**	**R_{18}**
1	Tetralin	S	S	S	S	S
2	Benzene	S	S	S	S	P
3	Toluene	S	S	S	S	S
4	o-Xylene	S	S	S	S	S
5	m-xylene	S	S	S	S	P
6	p-xylene	S	S	S	S	P
7	Mesitylene	S	S	S	S	P
8	Nitrobenzene	S	S	S	S	S
9	Chlorobenzene	S	S	S	S	S
10	o-Dichlorobenzene	S	S	S	S	S
11	H_2O	INS	INS	INS	INS	INS
12	ethyl acetate	S	S	S	S	S
13	Ethanol	S	S	S	S	S
14	n-amyl alcohol	S	S	S	S	S
15	Methanol	S	S	S	S	S
16	Chloroform	S	S	S	S	S
17	Isopropyl ether	S	S	S	P	P
18	Cyclohexane	S	S	S	S	S
19	DMSO	S	S	S	S	P
20	Petroleum ether	S	S	S	S	S
21	Ethylene glycol	S	S	S	S	P
22	Methylene Chloride	S	S	S	P	P

S = solution; P = precipitate; INS = insoluble.

Table S3 Gelation data of **4·10**, **4·12**, **4·14**, **4·16**, and **4·18**

	Gelators	SAD-4 and R-NH_2 in 1:2				
	Solvents	**4·10**	**4·12**	**4·14**	**4·16**	**4·18**
1	Tetralin	2.04	1.45	C	C	C
2	Benzene	1.22	2.79	C	C	C
3	Toluene	3.38	2.06	INS	C	C
4	o-Xylene	2.24	6.9	INS	C	C
5	m-xylene	2.29	2.48	C	INS	INS
6	p-xylene	2.29	2.48	INS	INS	INS
7	Mesitylene	2.62	2.83	INS	INS	INS
8	Nitrobenzene	1.0	0.96	C	C	C
9	Chlorobenzene	1.49	1.17	C	C	C
10	o-Dichlorobenzene	1.27	0.89	C	C	C
11	H_2O	S	P	P	P	P
12	ethyl acetate	C	INS	INS	INS	INS
13	Ethanol	S	P	P	P	P
14	n-amyl alcohol	S	INS	INS	INS	INS
15	Methanol	S	P	P	P	P
16	Chloroform	INS	INS	INS	INS	INS
17	Isopropyl ether	C	INS	INS	INS	INS
18	Cyclohexane	C	INS	INS	INS	INS
19	DMSO	P	P	P	P	P
20	Petroleum ether	C	INS	INS	INS	INS
21	Ethylene glycol	C	INS	INS	INS	INS
22	Methylene Chloride	INS	INS	INS	INS	INS

Numerical values indicate minimum gelator concentration in wt% in the formation of heat-set gel; S = solution; C = colloid; P = precipitate; INS = insoluble.

Table S4 Gelation data of **6·10**, **6·12**, **6·14**, **6·16**, and **6·18**

	Gelators / Solvents	SAD-6 and R-NH_2 in 1:2				
		6·10	**6·12**	**6·14**	**6·16**	**6·18**
1	Tetralin	C	1.12	1.35	C	C
2	Benzene	C	1.16	1.6	C	C
3	Toluene	C	1.25	1.76	C	C
4	o-Xylene	2.33	1.19	1.73	C	C
5	m-xylene	C	1.26	1.7	INS	INS
6	p-xylene	S	C	2.48	INS	INS
7	Mesitylene	C	1.25	2.61	INS	INS
8	Nitrobenzene	C	0.77	0.81	C	C
9	Chlorobenzene	C	0.85	1.1	C	C
10	o-Dichlorobenzene	C	0.72	0.94	C	C
11	H_2O	S	P	P	P	P
12	ethyl acetate	INS	INS	INS	INS	INS
13	Ethanol	S	P	P	P	P
14	n-amyl alcohol	S	INS	INS	INS	INS
15	Methanol	S	P	P	P	P
16	Chloroform	C	INS	INS	INS	INS
17	Isopropyl ether	INS	INS	INS	INS	INS
18	Cyclohexane	INS	INS	INS	INS	INS
19	DMSO	C	P	P	P	P
20	Petroleum ether	INS	INS	INS	INS	INS
21	Ethylene glycol	C	INS	INS	INS	INS
22	Methylene Chloride	INS	INS	INS	INS	INS

Numerical values indicate minimum gelator concentration in wt% in the formation of heat-set gel; S = solution; C = colloid; P = precipitate; INS = insoluble.

Table S5 Gelation data of **8·10**, **8·12**, **8·14**, **8·16**, and **8·18**

	Gelators	SAD-8 and R-NH_2 in 1:2				
	Solvents	**8·10**	**8·12**	**8·14**	**8·16**	**8·18**
1	Tetralin	C	1.33	1.01	C	C
2	Benzene	C	1.96	1.23	C	C
3	Toluene	C	2.64	1.56	C	C
4	o-Xylene	C	1.47	1.31	C	C
5	m-xylene	C	2.0	1.75	INS	INS
6	p-xylene	S	2.22	1.75	INS	INS
7	Mesitylene	C	1.43	1.61	INS	INS
8	Nitrobenzene	C	1.03	0.63	C	C
9	Chlorobenzene	C	1.56	1.48	C	C
10	o-Dichlorobenzene	C	0.95	0.58	C	C
11	H_2O	S	P	P	P	P
12	ethyl acetate	INS	INS	INS	INS	INS
13	Ethanol	S	P	P	P	P
14	n-amyl alcohol	S	INS	INS	INS	INS
15	Methanol	S	P	P	P	P
16	Chloroform	C	INS	INS	INS	INS
17	Isopropyl ether	INS	INS	INS	INS	INS
18	Cyclohexane	INS	INS	INS	INS	INS
19	DMSO	C	P	P	P	P
20	Petroleum ether	INS	INS	INS	INS	INS
21	Ethylene glycol	C	INS	INS	INS	INS
22	Methylene Chloride	INS	INS	INS	INS	INS

Numerical values indicate minimum gelator concentration in wt% in the formation of heat-set gel; S = solution; C = colloid; P = precipitate; INS = insoluble.

Table S6 Gelation data of **10·10**, **10·12**, **10·14**, **10·16**, and **10·18**

	Gelators	SAD-10 and R-NH_2 in 1:2				
	Solvents	**10·10**	**10·12**	**10·14**	**10·16**	**10·18**
1	Tetralin	C	C	C	2.83	INS
2	Benzene	C	C	C	2.6	3.31
3	Toluene	C	C	C	3.15	6.12
4	o-Xylene	C	C	INS	2.77	6.03
5	m-xylene	C	C	INS	2.81	6.16
6	p-xylene	S	C	INS	2.81	6.16
7	Mesitylene	C	C	C	2.64	3.36
8	Nitrobenzene	C	C	1.20	1.20	1.06
9	Chlorobenzene	C	C	C	2.19	1.6
10	o-Dichlorobenzene	C	C	C	1.39	3.35
11	H_2O	S	S	P	P	P
12	ethyl acetate	INS	INS	INS	INS	INS
13	Ethanol	S	P	P	P	P
14	n-amyl alcohol	S	INS	INS	INS	INS
15	Methanol	S	P	P	P	P
16	Chloroform	C	INS	INS	INS	INS
17	Isopropyl ether	INS	INS	INS	INS	INS
18	Cyclohexane	INS	INS	INS	INS	INS
19	DMSO	C	P	P	P	P
20	Petroleum ether	INS	INS	INS	INS	INS
21	Ethylene glycol	C	INS	INS	INS	INS
22	Methylene Chloride	INS	INS	INS	INS	INS

Numerical values indicate minimum gelator concentration in wt% in the formation of heat-set gel; S = solution; C = colloid; P = precipitate; INS = insoluble.

Table S7 Gelation data of fatty acids (dodecanoic acid, tetradeconic acid, palmitic acid) and dicarboxylic acids (succinic acid, sebacic acid).

	Compounds / Solvents	Acids				
		Dodecanoic acid	Tetradeconic acid	Palmitic acid	Succinic acid	Sebacic acid
1	Tetralin	S	S	S	P	P
2	Benzene	S	S	S	P	P
3	Toluene	S	S	S	P	P
4	o-Xylene	S	S	S	P	P
5	m-xylene	S	S	S	P	P
6	p-xylene	S	S	S	P	P
7	Mesitylene	S	S	S	P	P
8	Nitrobenzene	S	S	S	P	P
9	Chlorobenzene	S	S	S	P	P
10	o-Dichlorobenzene	S	S	S	P	P
11	H_2O	INS	INS	INS	P	P
12	ethyl acetate	S	S	S	S	S
13	Ethanol	S	S	S	S	S
14	n-amyl alcohol	S	S	S	S	S
15	Methanol	S	S	S	S	S
16	Chloroform	S	S	S	P	P
17	Isopropyl ether	S	S	S	P	P
18	Cyclohexane	S	S	S	P	P
19	DMSO	S	S	S	S	S
20	Petroleum ether	S	S	S	P	P
21	Ethylene glycol	P	P	P	S	S
22	Methylene Chloride	S	S	S	P	P

S = solution; P = precipitate; INS = insoluble.

Table S8 Gelation data of conventional gels transformed from heat-set gels by addition of fatty acids.

Gelators / Fatty acids	SAD-n and R-NH_2 in 1:2			
	6·12	**6·14**	**8·12**	**8·14**
Dodecanoic acid	CG	CG	CG	CG
Tetradeconic acid	CG	CG	CG	CG
Palmitic acid	CG	CG	CG	CG

CG = conventional gel

Table S9 Gelation data of thermo-irreversible gel gels transformed from het-set gels by addition of dicarboxylic acids.

Gelators / Dicarboxylic acids	SAD-n and R-NH_2 in 1:2			
	6·12	**6·14**	**8·12**	**8·14**
Succinic acid	SHG	SHG	SHG	SHG
Sebacic acid	SHG	SHG	SHG	SHG
SAD-6	SHG	SHG	SHG	SHG
SAD-8	SHG	SHG	SHG	SHG
SAD-10	SHG	SHG	SHG	SHG

SHG = thermo-irreversible gel

3) Obtaining of xerogels

The xerogels of heat-set gels were obtained by drying the gels in a vacuum oven at a temperature over Tgel for 24 h.

4) Growth of single crystal of 6•14

0.5 mmol (0.158 g) SAD-**6** and 1.0 mmol (0.214 g) R_{14}-NH_2 were dissolved in 20 mL mixed solution of ethanol and distilled water (v/v = 1:1). The mixed solution was allowed to stay at ambient temperature. After three weeks, colorless crystals suitable for single-crystal X-ray diffraction analysis were obtained by filtration.

5) Date of differential scanning calorimetry (DSC)

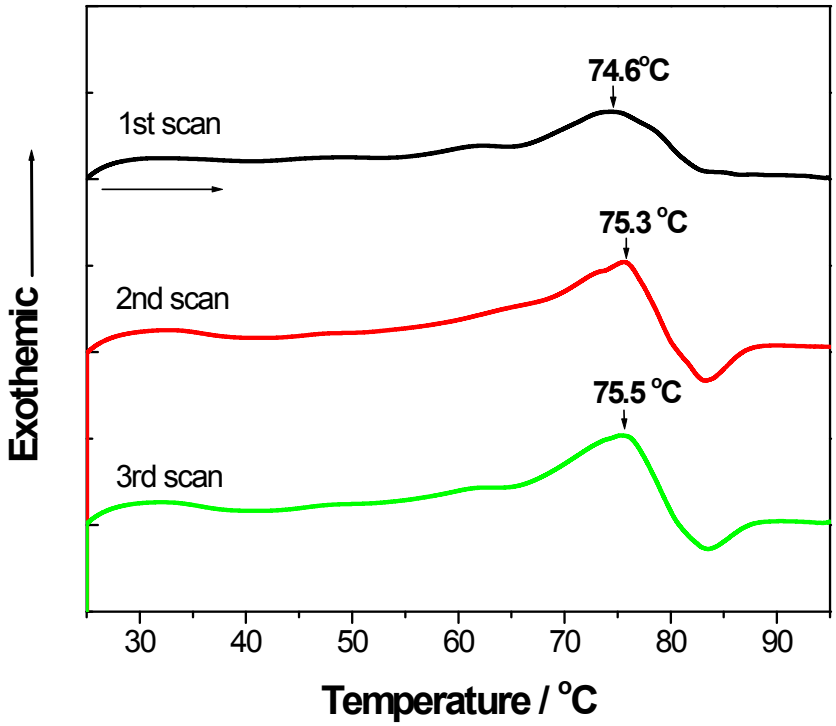


Fig. S1 Differential scanning calorimetry (DSC) thermograms for **6·14** composite in tetralin (1.88 wt.%).

6) FT-IR results

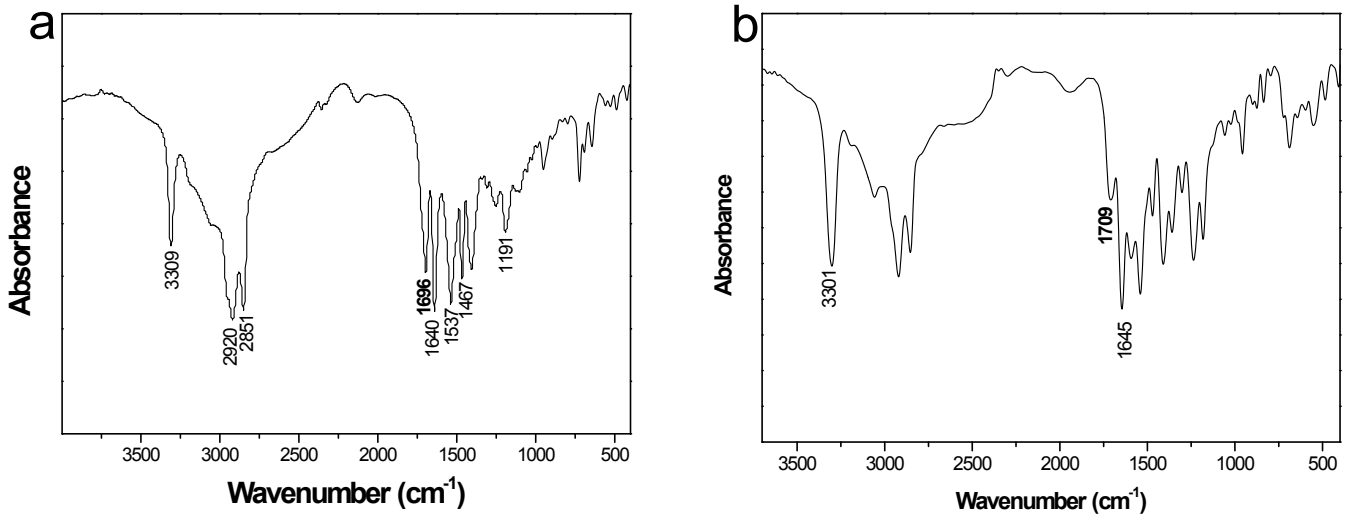


Fig. S2 FT-IR spectra. a, xerogel of **6·14** with dodecanoic acid in 1:2:3 molar ratio. b, xerogel of **6·14** with SAD-6 in 1:2:1 molar ratio.

7) SA-XRD patterns

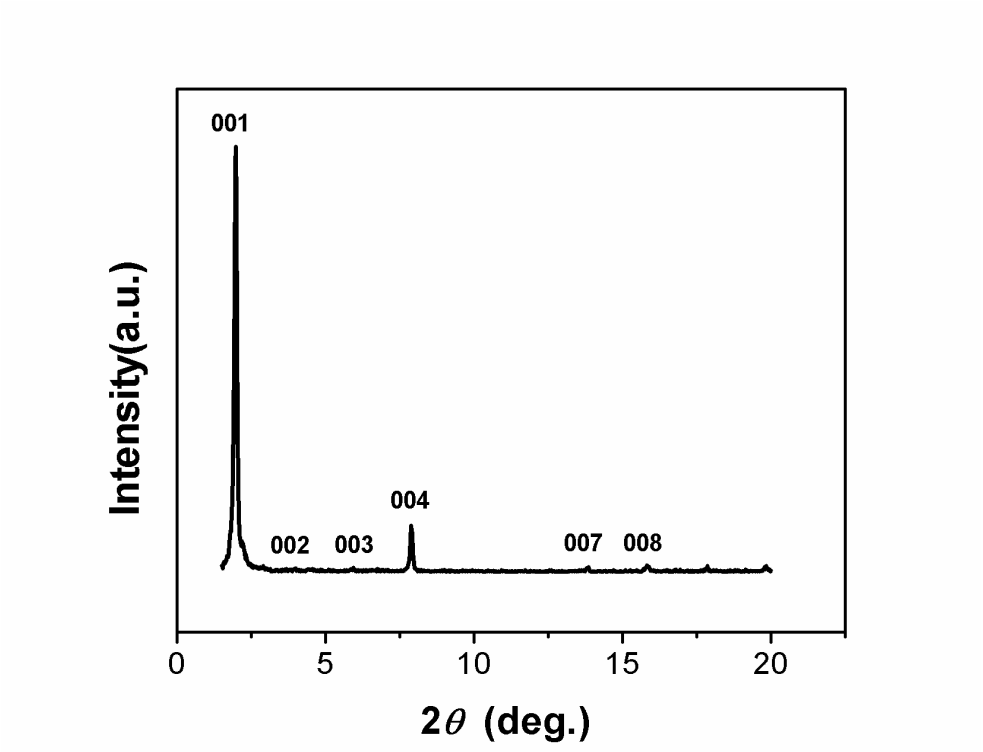


Fig. S3 SA-XRD patterns of **6·14** xerogel

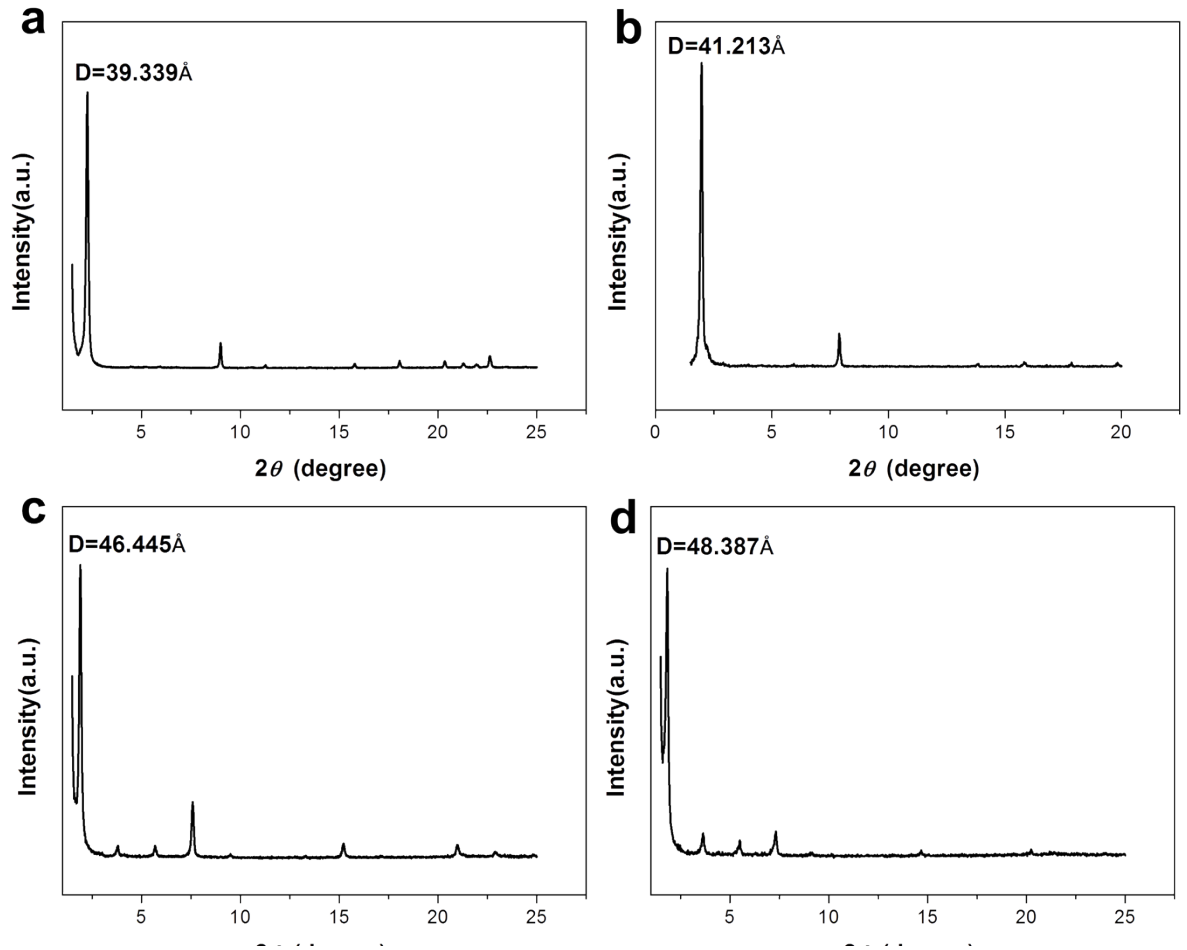


Fig. S4 SA-XRD patterns of heat-set gels in tetralin. a, **4**·**12**. b, **6**·**12**. c, **8**·**14**. d, **10**·**16**. SA-XRD patterns reveal that the *d*-spacing of the heat-set gel of **4**·**12**, **6**·**12**, **8**·**14** and **10**·**16** are 39.339, 41.213, 46.445 and 48.387 Å, respectively. The evaluated molecular length (L_2 = R_n-NH_2 + SAD-n + R_n-NH_2) by optimized CPK model are 51.506,54.376, 62.565 and 70.131 Å, respectively.

8) Crystal structure

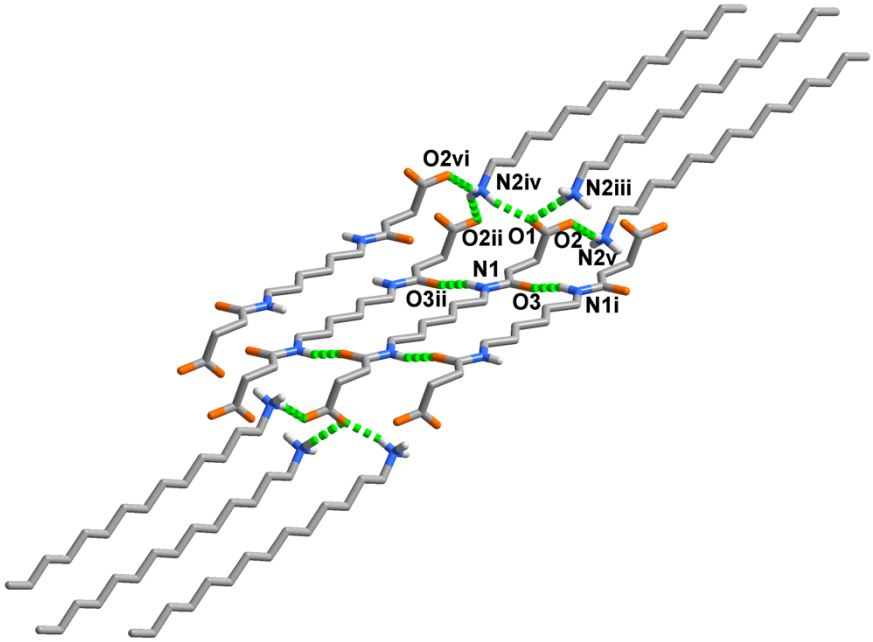


Fig. S5 Crystal structure of **6·14** (Symmetric codes: *i* = 1 + *x*, *y*, *z*; *ii* = -1 + *x*, *y*, *z*; *iii* = 2 + *x*, 1 + *y*, *z*; *iv* = 1 + *x*, 1 + *y*, *z*; *v* = 2 + *x*, 2 + *y*, *z*; *vi* = -1 + *x*, -1 + *y*, *z*).

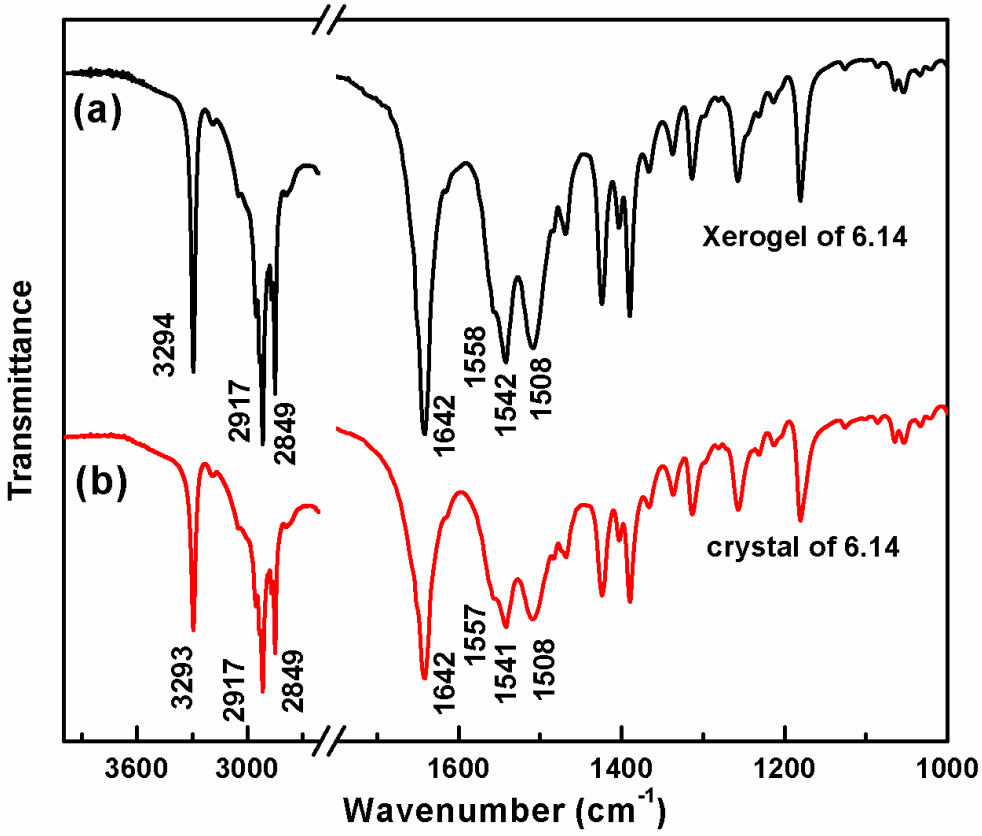


Fig. S6 IR spectra of **6**·**14** (a) xerogel, (b) crystals.

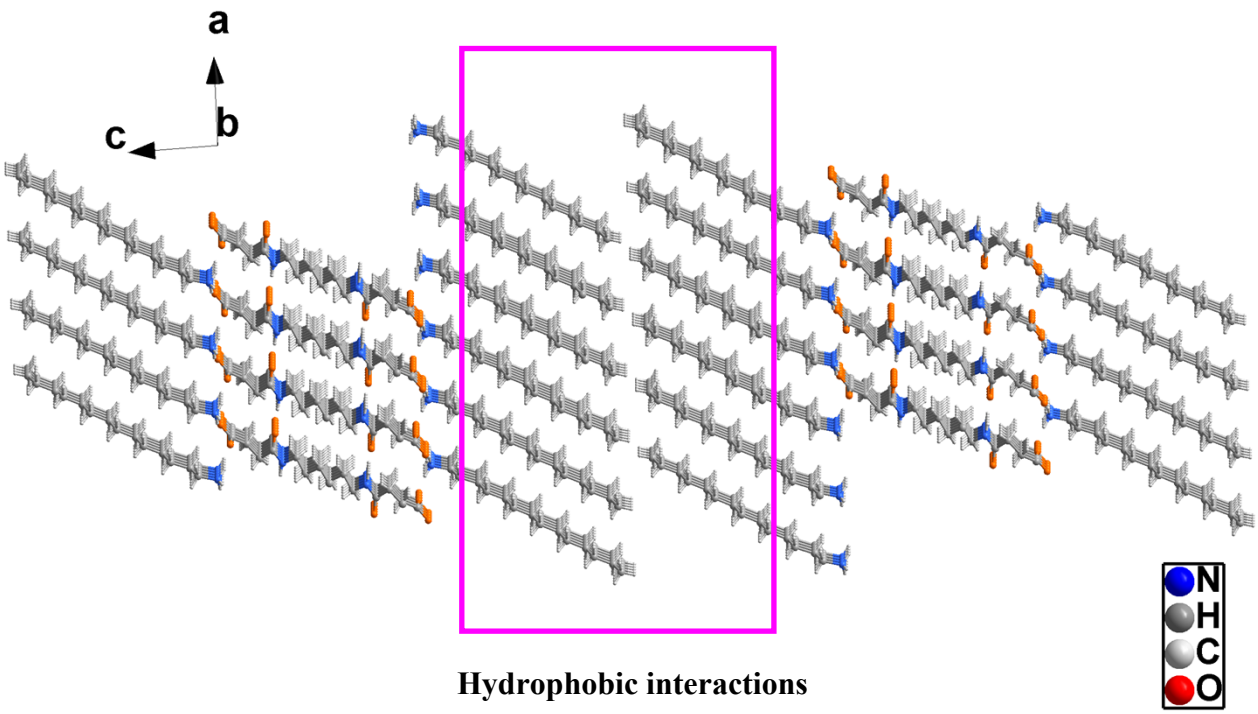


Fig. S7 3D supramolecular structure of **6·14** composite.