Supplementary Material (ESI) for Chemical Communications

Supplementary Information

Enantioselective Nanofiber-Spinning of Chiral Calixarene Receptor with Guest

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Materials and Methods

Materials. All reagents and solvents were chemical pure (CP) grade or analytical reagent (AR) grade and were used as received. Even chloroform used as gelled solvent did not need to remove the added ethyl alcohol as protecting reagent. *D*-(+)-dibenzoyltartaric acid (99%), *L*-(+)-dibenzoyltartaric acid (99%), (*R*)-α-methylbenzylamine (98%) and (*S*)-α-methylbenzylamine (98%) bought both from Aldrich Chemical Co. Ltd and from chemical companies in China gave same results. *p-tert*-Butylcalix[4]arene and calix[4]arene starting materials were prepared according to supporting reference (*S1*) and (*S2*) respectively.

Measurements. ^{1}H NMR spectra were measured on a Bruker AV 400 spectrometer at 400 MHz at 298 K. Circular dichrorism (CD) spectra were recorded on a JASCO J-810 spectrometer. Field emission scanning electron microscopy (FE-SEM) images were taken on a FEI Sirion200 electron microscope operating at 5 kV or 10 kV. Transmission electron micrographs (TEM) were recorded on a FEI Technai G2 20 electron microscope at 200 kV. Atomatic Force Microscopy (AFM) images were got on a VEECO Nano Scope IV instrument using tapping mode, and the showed AFM images are phase amplitude.

Powder X-ray diffraction (XRD) pattern were measured on a χ'Pert PRO diffraction instrument. The scan parameters and peak list are as following:

Anchor Scan Parameters

Dataset Name:	xiaojiao
File name:	E:¥X'Pert Data¥zhengyansong¥20060703¥xiaojiao.xrdml
Comment:	0.5-5 degree
Measurement Date / Time:	7/3/2006 2:45:55 PM
Operator:	dell
Raw Data Origin:	XRD measurement (*.XRDML)
Scan Axis:	Gonio
Start Position [°2Th.]:	1.0074
End Position [°2Th.]:	19.9794
Step Size [°2Th.]:	0.0170
Scan Step Time [s]:	41.0051
Scan Type:	Continuous
PSD Mode:	Scanning
PSD Length [°2Th.]:	2.12
Offset [°2Th.]:	0.0000
Divergence Slit Type:	Fixed
Divergence Slit Size [°]:	0.0315

Specimen Length [mm]:	10.00
Measurement Temperature [°C]:	25.00
Anode Material:	Cu
K-Alpha1 [Å]:	1.54060
K-Alpha2 [Å]:	1.54443
K-Beta [Å]:	1.39225
K-A2 / K-A1 Ratio:	0.50000
Generator Settings:	40 mA, 40 kV
Goniometer Radius [mm]:	240.00
Dist. Focus-Diverg. Slit [mm]:	91.00
Incident Beam Monochromator:	No
Spinning:	No

Peak List

Pos.[°2Th.]	Height[cts]	FWHM[°2Th.]	d-spacing[Å]	Rel.Int.[%]
4.1316	1285.97	0.2342	21.38692	49.86
5.8046	2579.26	0.4015	15.22595	100.00
7.5255	589.38	0.1338	11.74759	22.85
12.7729	64.36	0.2676	6.93076	2.50
14.9144	103.36	0.2007	5.94006	4.01
16.0628	32.08	0.4015	5.51789	1.24

Supporting References

S1. C. D. Gutsche, M. Iqbal, D. Stewart, *J. Org. Chem.* **51**, 742 (1986).

S2. C. D. Gutsche, L. G. Lin, *Tetrahedron* **42**, 1633 (1986).

Supporting Figures

Fig. S1. Chloroform gel from mixing of **1a** and *D*-**2** (left), and Clear chloroform solution from mixing of **1a** and *L*-**2** (right).

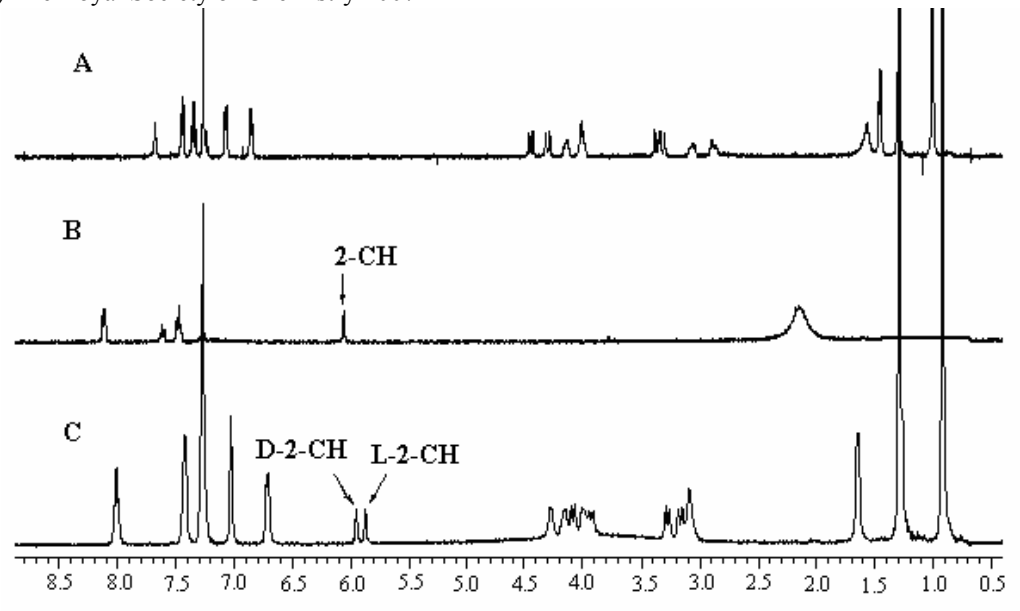

Fig. S2. ^{1}H NMR spectra. (**A**) from chiral calixarene **1b** (5 mM in $CDCl_3$, similarly hereinafter). (**B**) from racemic 2,3-dibenzoyltartaric acid **2** (5 mM). (**C**) from mixture of **1b** (3.3mM) and **2** (3.3 mM).

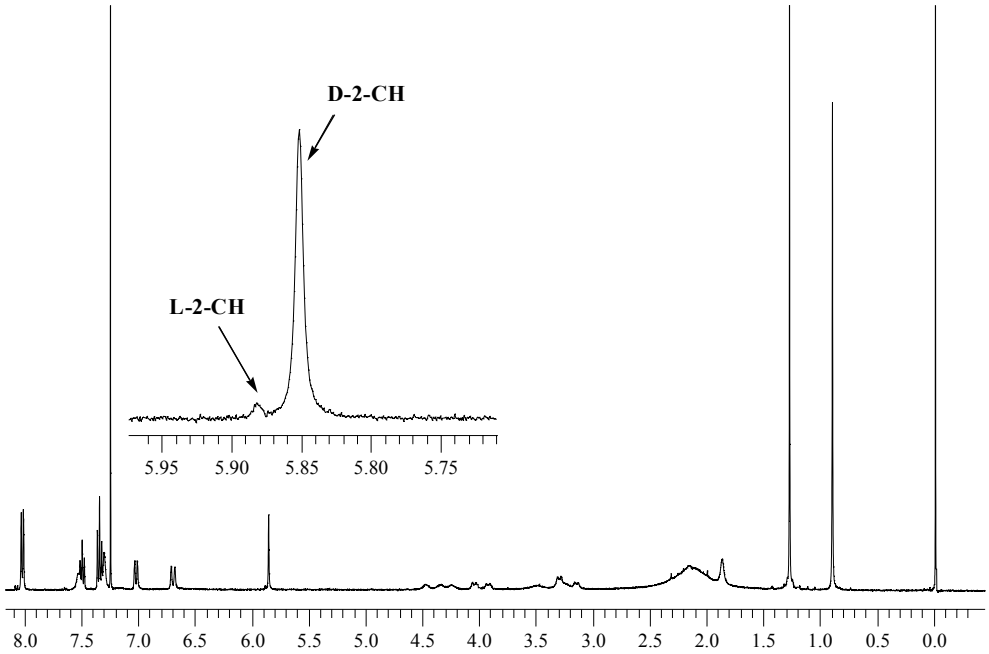

Fig. S3. ^{1}H NMR spectra of isolated gel of **1a** and racemic **2** in $CDCl_3$. The isolated gel was obtained by filtration of chloroform-gel formed by mixing **1a** (5 mM) and racemic **2** (10 mM) in chloroform through Teflon membrane having 0.22 μm pores.

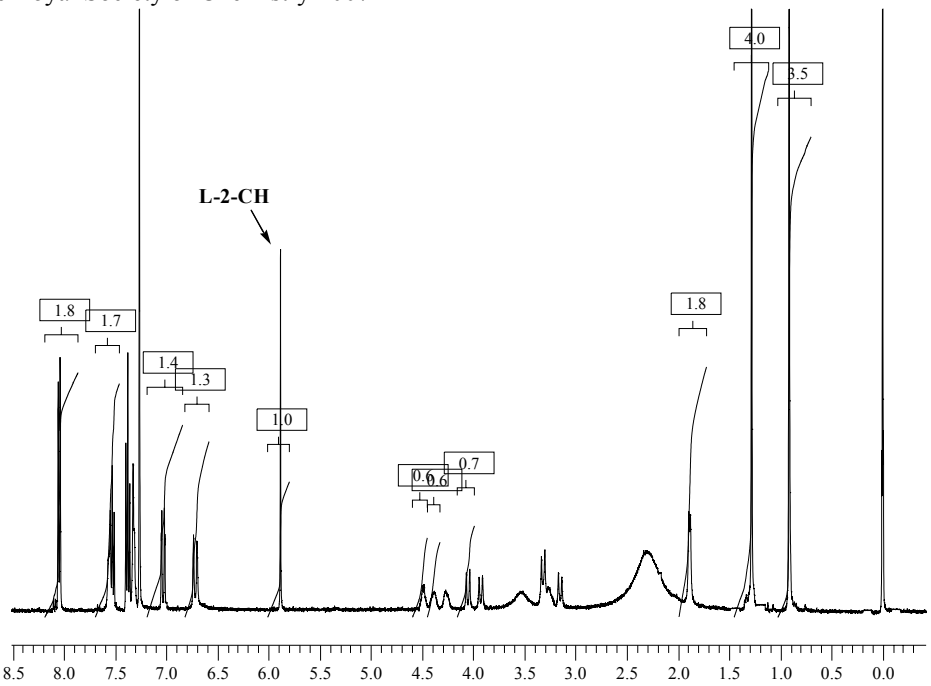

Fig. S4. ^{1}H NMR spectra of isolated gel of **1b** and *L*-**2** in $CDCl_3$. The isolated gel was obtained by following procedure: The first isolated gel was got by filtration of chloroform-gel formed by mixing **1b** (5 mM) and *L*-**2** (10 mM) in chloroform through Teflon membrane having 0.22 μm pores. The first isolated gel was dissolved in chloroform to form again gel, and then filtered.

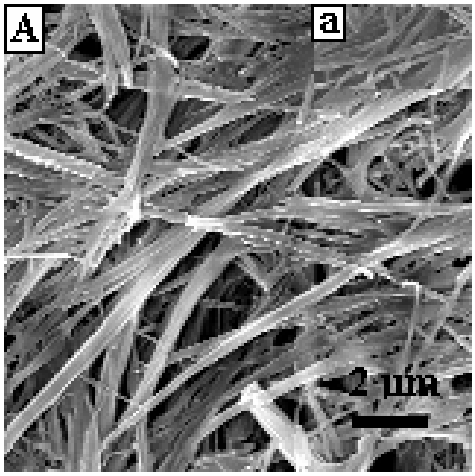

Fig. S5. FE-SEM images of chloroform xerogel from mixture of **1a** (5 mM) and *D*-**2** (10 mM) in chloroform, in which some single-stranded helical coils could be sighed. The sample was prepared by casting the gel onto a glass slide and let it air dry. (Inset **a**) A double-stranded helix.

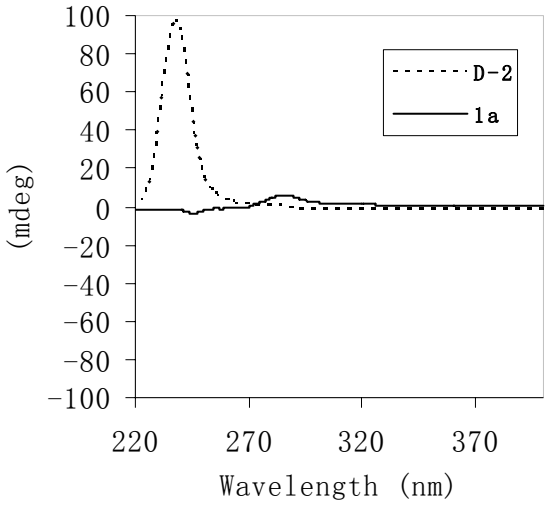

Fig. S6. CD spectra of pure **1a** (0.83 mM) and pure *D*-**2** (1.67 mM) in 1,2-dichloroethane at 20 °C

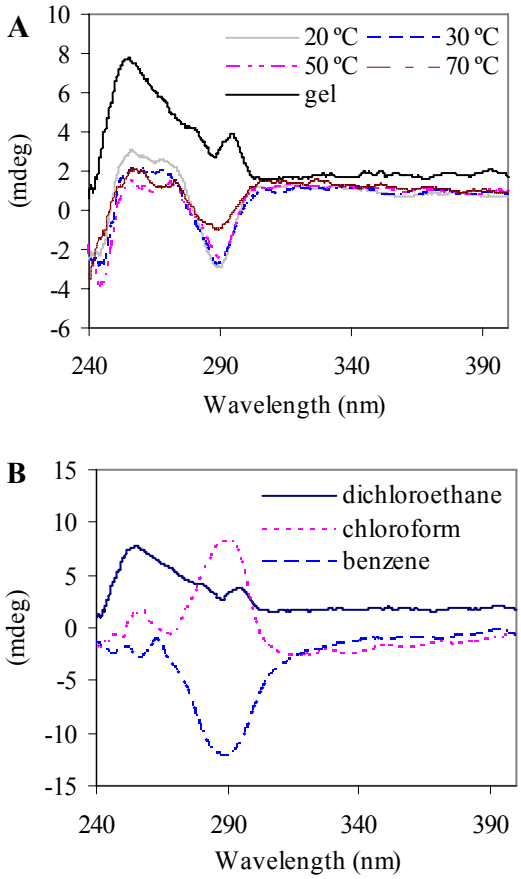

Fig. S7. CD spectra of mixture of **1a** and *D*-**2**. **A**) CD spectra of gel at 20 °C and solution of **1a** (0.83 M) and *D*-**2** (1.67 M) in 1,2-dichloroethane at varied temperature; **B**) CD spectra of gels of **1a** (0.83 M) and *D*-**2** (1.67 M) in different solvents at 20 °C.

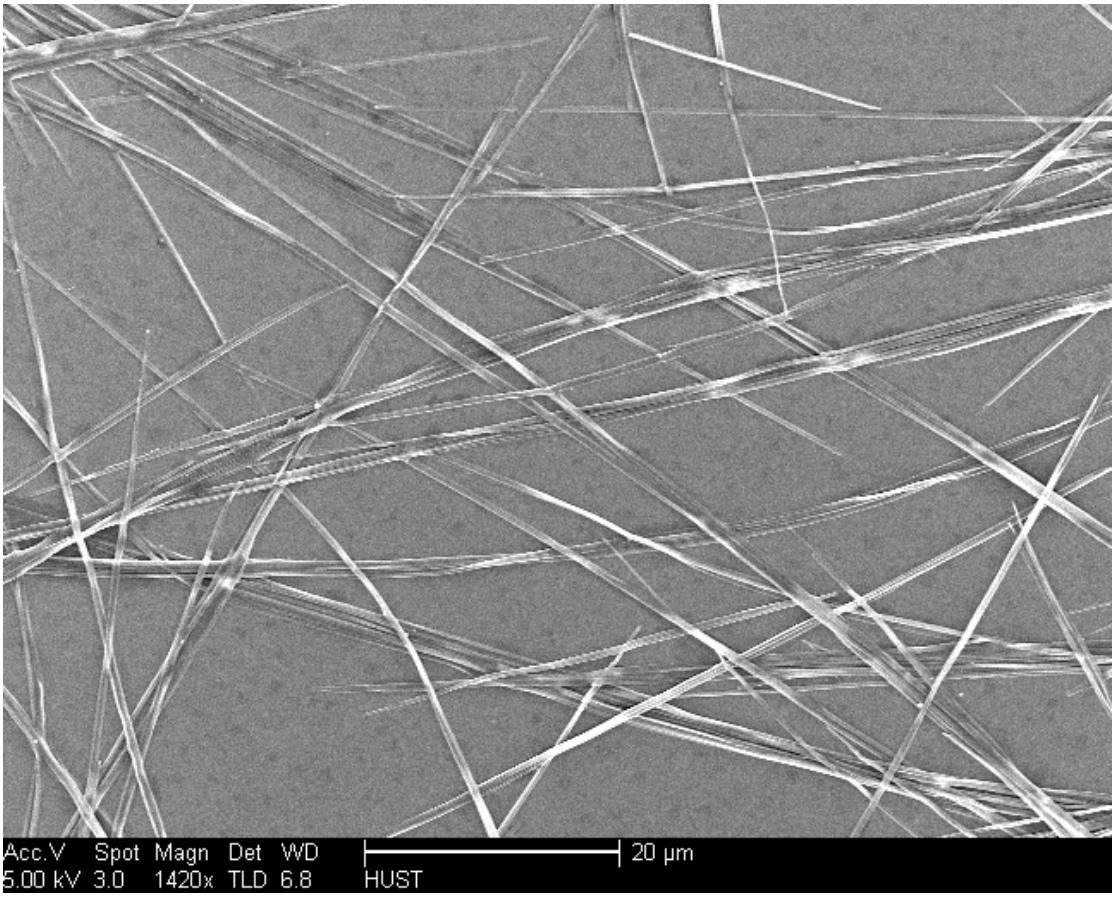

Fig. S8. FESEM images of gel obtained by interaction of **1a** (5 M) and *D*-**2** (10 M) in chloroform. The sample was prepared by casting the gel onto a glass slide and let it air dry.

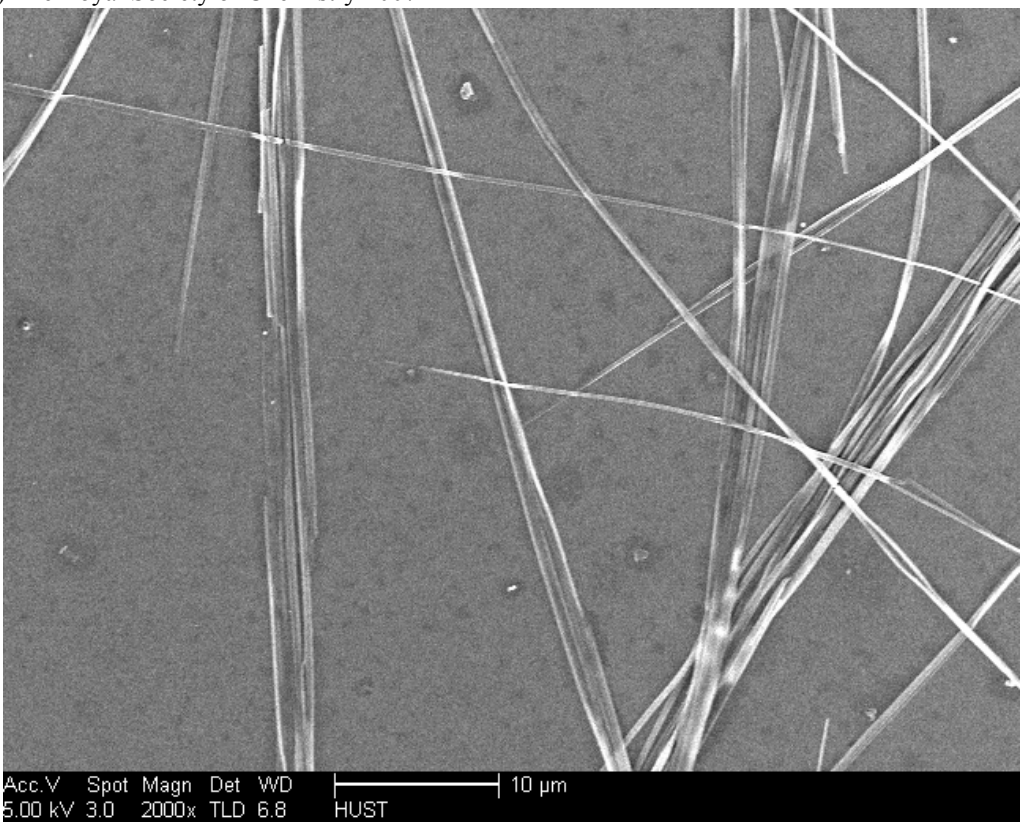

Fig. S9. FESEM images of gel obtained by interaction of **1a** (5 M) and *D*-**2** (10 M) in chloroform. The sample was prepared by casting the gel onto a glass slide and let it air dry.

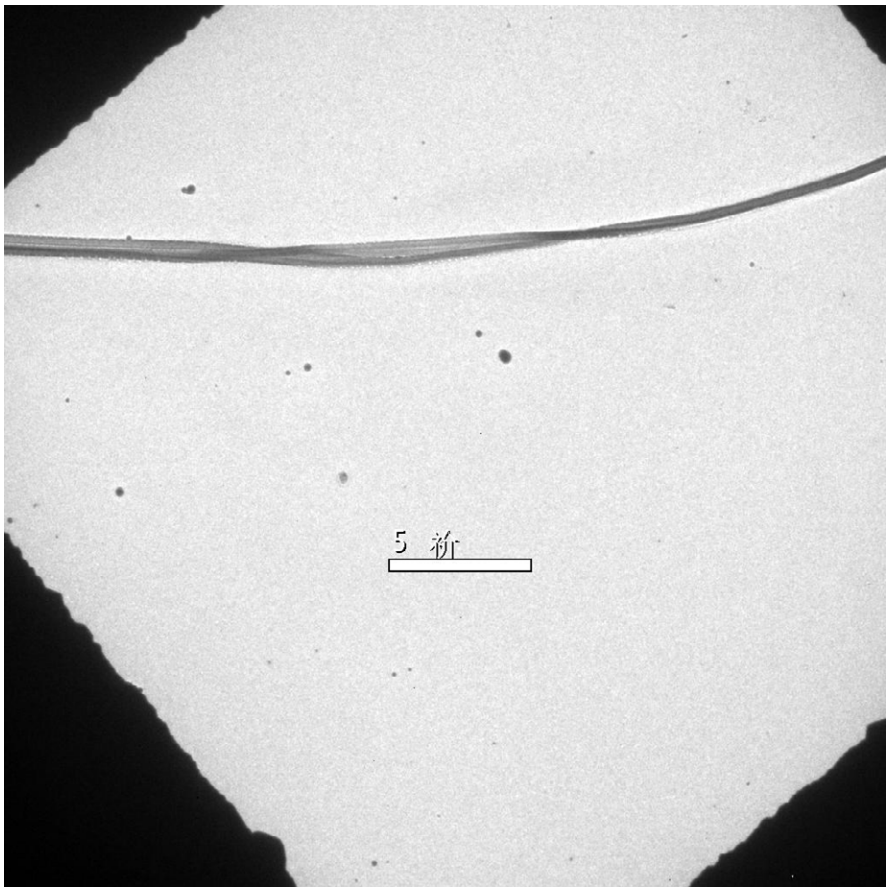

Fig. S10. TEM images of gel obtained by interaction of **1a** (5 M) and *D*-**2** (10 M) in chloroform. The gel was diluted 10 times with chloroform, and then a copper grid covered with a thin carbon film was put into the diluted gel and took it out at soon with a tweezers and air dried. Bar is 5 μm.

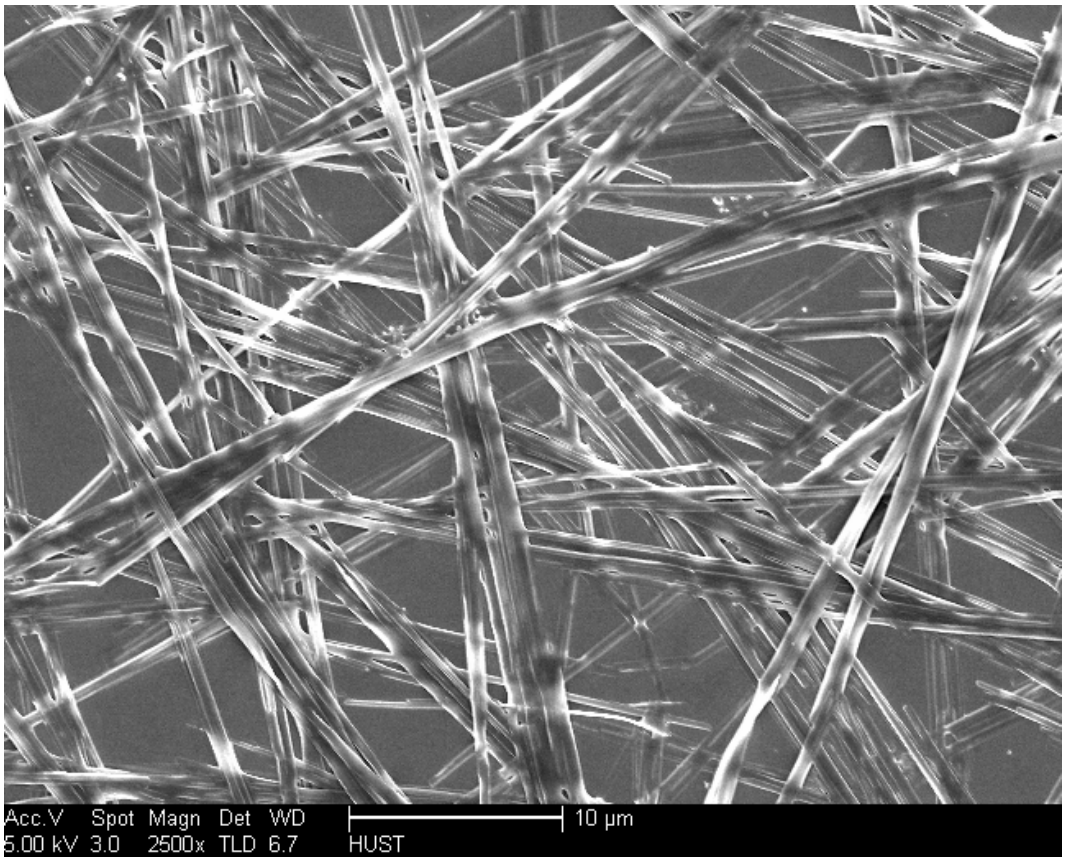

Fig. S11. FESEM images of gel obtained by interaction of **1a** (5 M) and *D*-**2** (10 M) in 1,2-dichloroethane. The sample was prepared by casting the gel onto a glass slide and let it air dry.

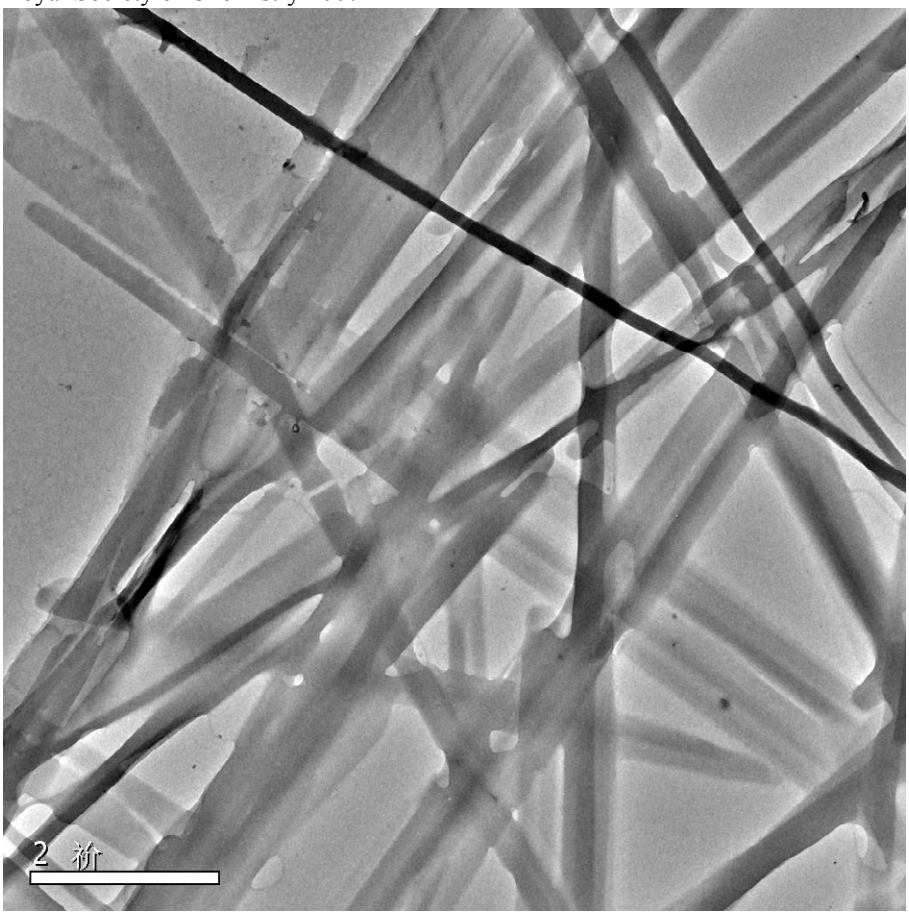

Fig. S12. TEM images of gel obtained by interaction of **1a** (5 M) and *D*-**2** (10 M) in 1,2-dichloroethane. The gel was diluted 10 times with 1,2-dichloroethane, and then a copper grid covered with a thin carbon film was put into the diluted gel and took it out at soon with a tweezers and air dried. Bar is 2 μm.

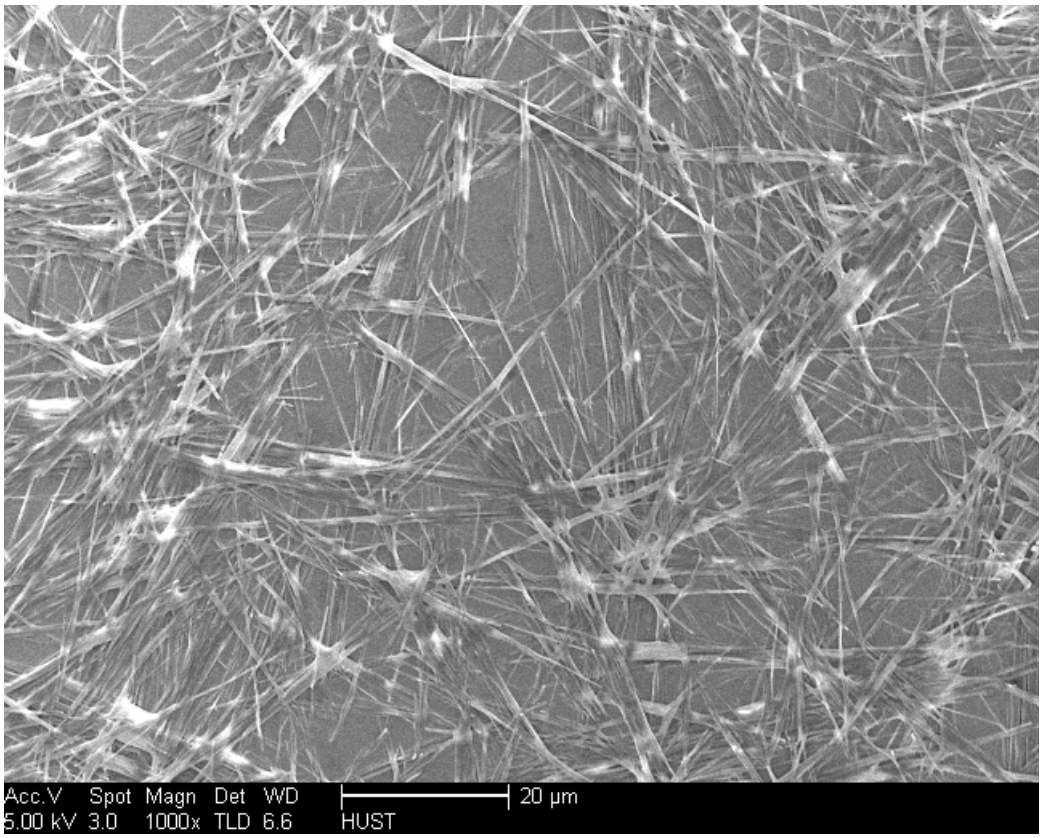

Fig. S13. FESEM images of gel obtained by interaction of **1a** (5 M) and *D*-**2** (10 M) in benzene. The sample was prepared by casting the gel onto a glass slide and let it air dry.

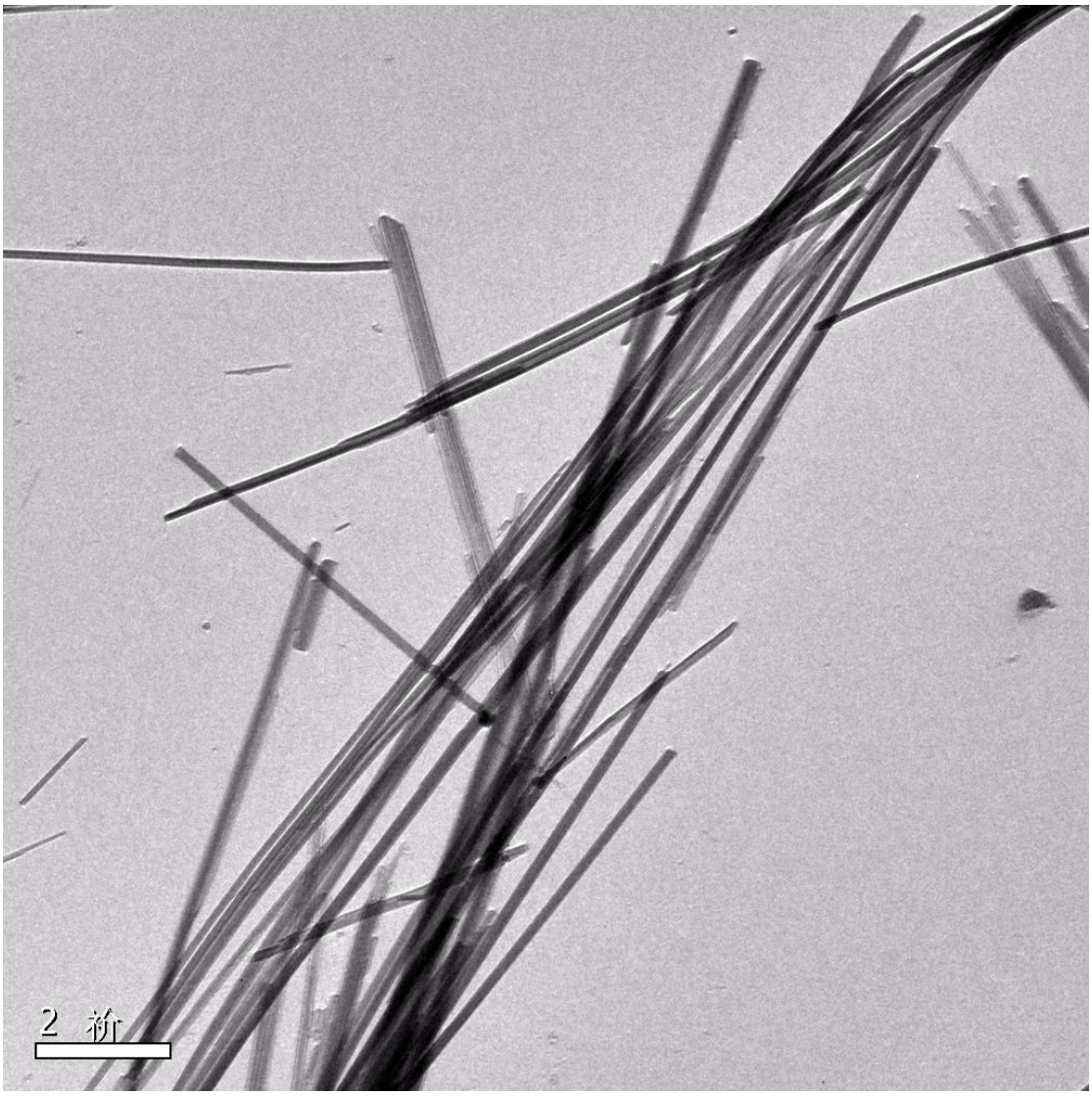

Fig. S14. TEM images of gel obtained by interaction of **1a** (5 M) and *D*-**2** (10 M) in benzene. The gel was diluted 10 times with benzene, and then a copper grid covered with a thin carbon film was put into the diluted gel and took it out at soon with a tweezers and air dried. Bar is 2 μm.

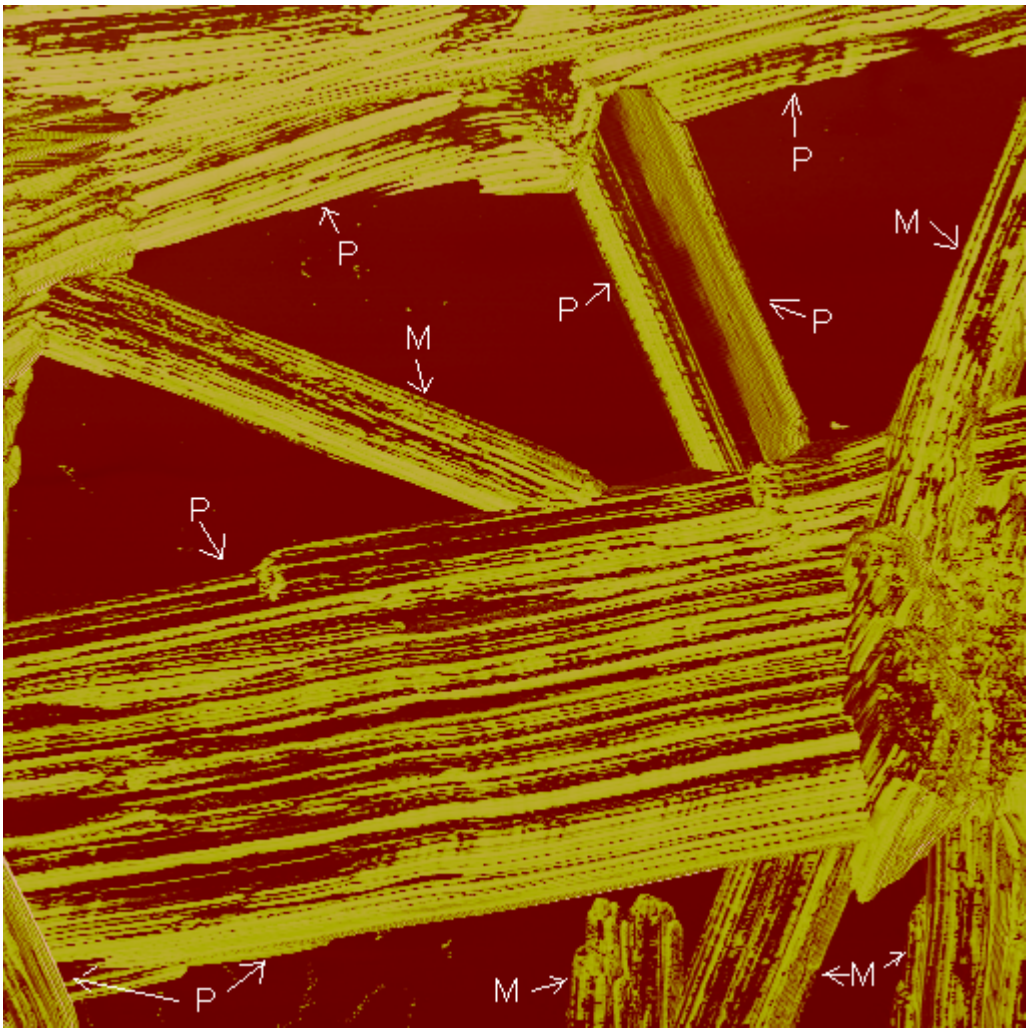

Fig. S15. AFM images of ribbon-like nanofibers of **1b** (5 mM) and *L*-**2** (10 mM) in chloroform, in which only the same handed coils stack together to form bigger fibers (7×7μm) The gel which was diluted 5 times with chloroform was dropped on the freshly broken mica and air dried.

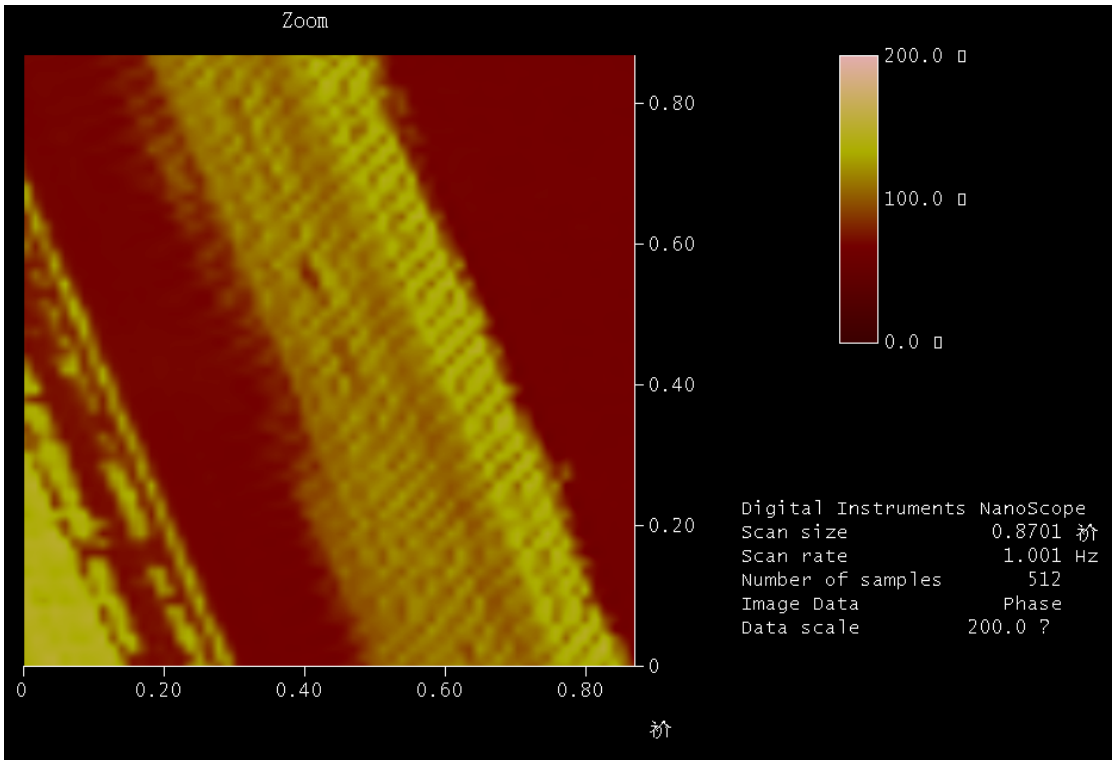

Fig. S16. AFM images of a ribbon-like nanofiber of **1b** (5 mM) and *L*-**2** (10 mM) in chloroform, which was formed by very orderly stack of coiled nanofibers having the same handedness (0.87×0.87μm). The gel which was diluted 5 times with chloroform was dropped on the freshly broken mica and air dried.

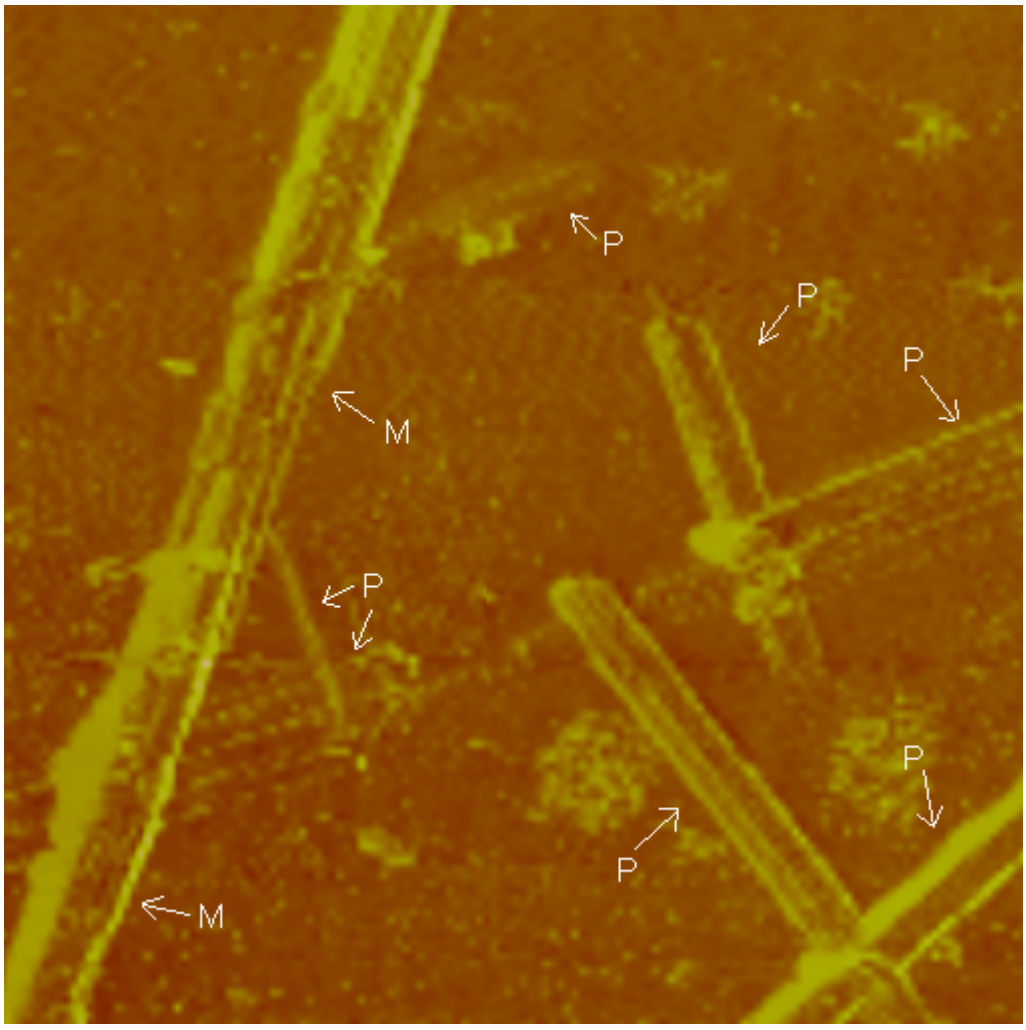

Fig. S17. AFM images of ribbon-like nanofibers of **1b** (5 mM) and *L*-**2** (10 mM) in 1,2-dichloroethane, in which only the same handed coils stack together to form bigger fibers (27×27μm). The gel which was not diluted was rubbed onto the freshly broken mica with capillary glass tube and air dried.

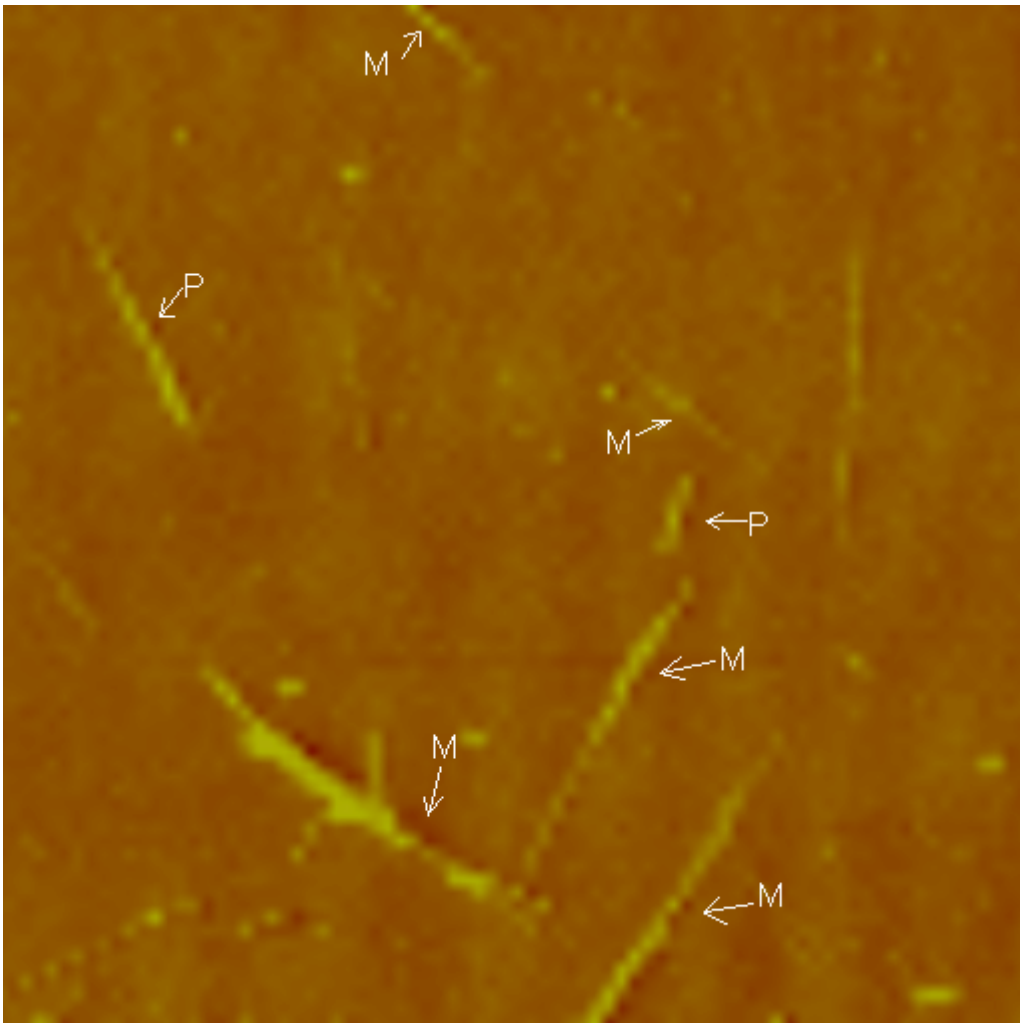

Fig. S18. AFM images of nanofibers of **1b** (5 mM) and *L*-**2** (10 mM) in benzene. Most of coils were left-handed (M) which was contrary to that of chloroform or 1,2-dichloroethane gel fibers (15.6×15.6 μm). The gel which was not diluted was rubbed onto the freshly broken mica with capillary glass tube and air dried.

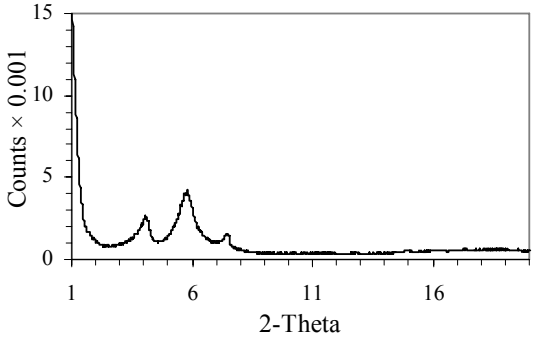

Fig. S19. Powder X-ray diffraction pattern of xerogel resulted from mixture of **1a** (5 mM) and *D*-**2** (10 mM) in chloroform.

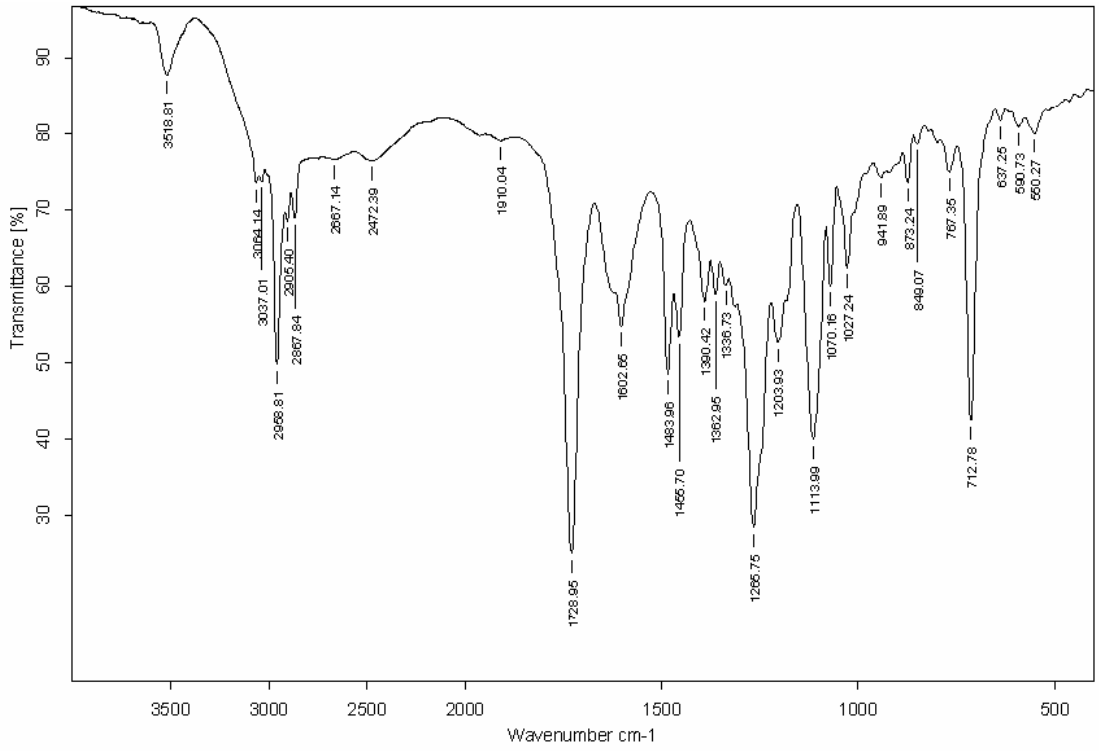

Fig. S20. IR spectrum of xerogel resulted from mixture of **1a** (5 mM) and *D*-**2** (10 mM) in chloroform.