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Electric Field-Assisted Alignment of Self-Assembled Fibers Composed of Hydrogen-Bonded Molecules Having Laterally Fluorinated Mesogens

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Abstract: Aligned fibrous aggregates of amide compounds having laterally fluorinated aromatic mesogens have been successfully obtained by the application of the alternating current electric field (1.0 V/μm, 1 kHz) in dodecylbenzene. In contrast, randomly entangled fibers are formed in the solvent without electric fields. For the analogous compounds without fluorine substituent, no aligned fibrous aggregates have been obtained under the electric fields. The electric field alignment of the fibers should be assisted by the fluorinated rod-shaped mesogens that exhibit negative dielectric anisotropy.

Introduction

Fibrous molecular aggregation in a variety of solvents through noncovalent interactions such as hydrogen bonding has attracted attention.[1] Normally, network structures of entangled fibrous aggregates are random in isotropic organic and aqueous solvents. If aligned fibrous aggregates are easily obtained by using external stimuli such as electric fields, the resulting oriented fibers would yield a variety of anisotropically functional materials such as electronic, optoelectronic, and photonic materials.[1,2] Our intension here is to control the growth and alignment of fibrous molecular aggregation by applying electric fields in isotropic organic solvents. Until now, the alignment of self-assembled fibers has been achieved by several approaches such as the use of liquid crystals as anisotropic templates[1h,1j,3] and the application of shear flow,[4] magnetic fields,[5] and electric fields.[2i,6] Among these approaches, the electric field alignment is of particular interest because of its possibility to fabricate oriented fibers in a desired small area. For self-assembled fibers aligned by electric fields, there are two examples of an oligothiophene derivative[2i] and a 1,3,5-triamide cyclohexane derivative.[6] These molecules have hydrogen-bonded moieties exhibiting a dipolar nature. However, the effects of the dipole moment on the formation of oriented fibrous aggregates under electric fields are unproved. Moreover, parallel alignment of fibers that bridge two electrodes on substrates has not yet been achieved by applied alternating current (AC) electric fields. It is of interest to examine the role of dipole moments.

Here we demonstrate that hydrogen-bonded molecules having laterally fluorinated rod-shaped mesogens form oriented fibers that bridge two gold electrodes under AC electric fields in an organic solvent. In comparison with the results of analogous compounds without the fluorine atoms, we have found that the

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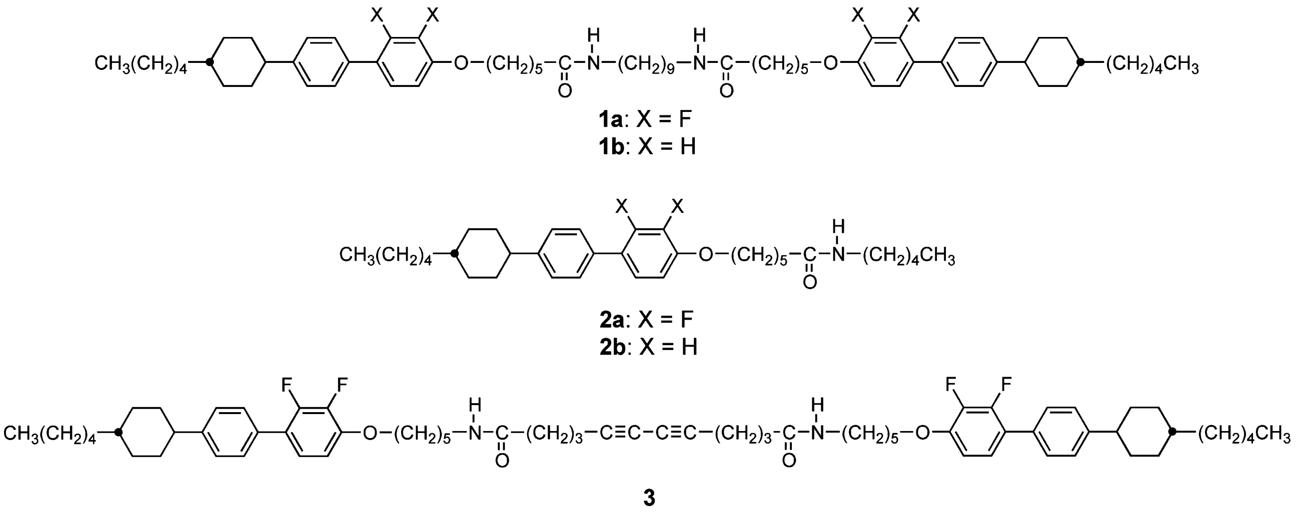

Figure 1. Molecular structures of amide compounds **1**−**3** having rod-shaped mesogens.

incorporation of fluorine atoms with a large dipole moment into the aromatic core plays a key role in the formation of aligned fibrous aggregates under electric fields.

Results and Discussion

Molecular Design and Synthesis of Amide Derivatives. Amide compounds **1**−**3** having one or two rod-shaped mesogens have been prepared (Figure 1). They were expected to form self-assembled fibers in organic solvents through hydrogen bonding. For compounds **1a**, **2a**, and **3**, lateral fluoro substituents are introduced to the rod-shaped aromatic mesogen to give negative dielectric anisotropy. To examine the effects of fluoro substituents on self-assembly of molecules under electric fields, compounds **1b** and **2b** without the fluorine atoms were prepared as references. Simpler diamide compounds such as didodecanoylamides of α,*ω*-dialkylidenediamines were found to form hydrogen-bonded fibrous aggregates in common organic solvents.[7] The fluoro-substituted rod-shaped aromatic compounds have been used for electric field-responsive liquid crystals as results of their low viscosities and low melting points.[8] For compound **3**, a diacetylene moiety was incorporated into the spacer to improve the mechanical stability of the fibers through photopolymerization.[9] Compounds **1**−**3** were synthesized by condensation of rod-shaped aromatic molecules having a carboxy or amino group at the end of a pentyloxy chain and 1,9-diaminononane, pentylamine, or 5,7-dodecadiynedioic acid (see Supporting Information).

Table 1. Phase Transition Behavior of Compounds **1**−**3**

compound	thermal properties^a				
1a	Cr_1	140 (12)	Cr_2	195 (66)	Iso
1b	Cr	234 (91)	Iso		
2a	Cr	139 (41)	SmA	143 (6.0)	Iso
2b	Cr	158 (20)	SmA	177 (8.8)	Iso
3	Cr_1	91 (6.3)	Cr_2	208 (32)	Iso

[a] Cr: crystal; SmA: smectic A liquid crystal; Iso: isotropic liquid. Temperatures in °C are given as the onset of the peaks detected by DSC measurements on the second heating run at a scanning rate of 10 °C min^{-1}. The enthalpy changes (kJ mol^{-1}) are in parentheses.

Liquid-Crystalline Properties. The phase transition behavior of compounds **1**−**3** is summarized in Table 1. No mesomorphic behavior was observed for compounds **1a**, **1b**, and **3**, which have dimeric mesogenic molecular structures. In contrast, compounds **2a** and **2b** exhibited enantiotropic smectic A liquid-crystalline (SmA) phases. Compound **2a** with the fluoro substituents showed lower melting and clearing points than those of **2b**. The X-ray diffraction patterns of **2a** at 130 °C and **2b** at 170 °C revealed that the layer spacings are 3.8 and 3.6 nm, respectively. The molecular length of **2a** and **2b** was estimated to be 3.4 nm by molecular modeling. On the basis of these results, it is assumed that compounds **2a** and **2b** with the extended molecular conformation form monolayer structures in the SmA phase.

Formation of Physical Gels. The results of the gelation test of amide derivatives **1**−**3** for organic solvents are shown in Table 2. The gelation abilities of compounds **1a** and **2a** with fluoro substituents are higher than those of compounds **1b** and **2b**.

The morphology of the xerogel of **1a** was studied by scanning electron microscopy (SEM). The SEM image (Figure 2) reveals that compound **1a** forms entangled fibrous networks in dodecylbenzene. The approximate diameter of fibrous aggregates is 60 nm.

Self-Assembled Structure of 1a in the Fibrous Aggregate. To examine the assembled molecular structure of **1a** in the fibrous aggregate, X-ray diffraction measurements were conducted for the dodecylbenzene gel (Figure 3a). The X-ray diffraction pattern gave three weak peaks of 3.3, 1.7, and 1.1 nm with the reciprocal spacing ratio of 1:2:3, which is attributed to the formation of a layer

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Table 2. Gelation Properties of Compounds **1**−**3**[a]

solvent	1a	1b	2a	2b	3
acetone	P	I	P	I	I
chloroform	G (23)	P	S	S	G (27)
tetrahydrofuran	G (23)	P	S	S	I
ethyl acetate	G (5)	P	G (46)	I	I
ethanol	P	P	P	I	I
benzene	G (5)	P	S	P	I
dimethylsulfoxide	G (23)	P	P	P	P
hexane	I	I	G (13)	I	I
dodecylbenzene	G (13)	P	G (13)	G (13)	G (8)

[a] G: gel; I: insoluble; P: precipitation; S: soluble. Minimum gel concentrations are in parentheses (given in mM).

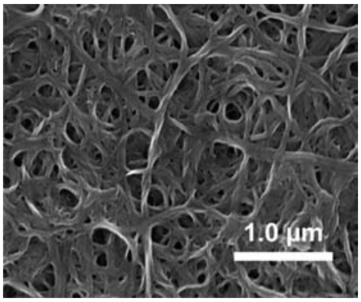

Figure 2. SEM image of fibrous aggregates of **1a** formed in dodecylbenzene.

structure. In addition, compound **1a** showed broad peaks in the wide angle region due to the short-range order of the self-asssembled structure. The length of the molecular long axis of **1a** was estimated to be 6.4 nm by molecular modeling (Figure 3b). On the basis of these results, it is assumed that compound **1a** with an extended molecular conformation forms an interdigitated layer structure[10] (Figure 3c).

Fibrous Self-Assembly of Molecules 1−3 under Electric Fields in Dodecylbenzene. For the experiments using electric fields, dodecylbenzene was selected as an isotropic solvent because of its electrochemical stability and low polarity. Moreover, dodecylbenzene shows a high boiling point over 235 °C, which is higher than those of the gels. This high temperature stability makes the processing of the fibrous assemblies easy. The dodecylbenzene gels of **1a**, **2a**, **2b**, and **3** were obtained at the concentration of 47 mM for **1a**, 32 mM for **2a**, 32 mM for **2b**, and 8 mM for **3**. These gels showed thermoreversible sol−gel transitions due to the association and dissociation of intermolecular hydrogen bonding among the amide moieties of the molecules.[7]

Comb-shaped gold electrodes deposited on a glass substrate were used for the alignment of fibers by applying electric fields. The dodecylbenzene solutions of **1**−**3** obtained by heating above their sol−gel transition temperatures (160 °C for **1a**, 80 °C for **2a**, 110 °C for **2b**, and 190 °C for **3**) were filled between the electrodes. Then, the solutions were cooled below the sol−gel transition temperatures at the rate of 1 °C/s under the application of electric fields.

We have found that aligned fibrous aggregates of **1a** that bridge the two gold electrodes (Figure 4a) were formed by the application of an AC electric field (1.0 V/μm, 1 kHz), while randomly entangled fibers were obtained without electric fields (Figure 4b). The formation of fibers occurred in seconds. No birefringence is observed for these fibers between crossed polarizers. The parallel alignment of the fibers of **3** having a diacetylene moiety was also induced in the direction of the AC electric field. In contrast, for compound **2a** plate-like aggregates were formed in the solvent, and no aligned fibrous aggregates of **2a** were obtained by applying the AC electric field. In addition, compound **2b** without the fluoro substituents formed randomly entangled fibers under the AC electric field (Figure 5). Considering the results of **1a** and **2b**, it is assumed that the dipole moment of fluoro substituents contributes to the formation of aligned fibrous aggregates under the electric field.

The direction of fiber growth was greatly influenced by the magnitude of applied AC voltage and its frequency. When the magnitude of the applied electric field was more than 0.5 V/μm at the frequency of 1 kHz, the aligned fibers of **1a** were formed (Figure 6). The magnitude of the estimated dipole moment for **1a** was 5.3 D to the direction of the molecular short axis. The strength of the AC electric fields required for the alignment might be determined by the degree of orientation of mesogenic moieties with the dipole moment in the solvent and the viscosity of the system. In the frequency range between 100 Hz and 10 kHz under the applied electric field of 1.0 V/μm, the macroscopic alignment of fibers of **1a** was achieved. In contrast, when a direct current (DC) electric field of 1.0 V/μm was applied between the electrodes, no oriented fibers that bridge the two electrodes were obtained. Randomly entangled fibers were seen at the vicinity of a negatively charged electrode (Figure 7). The adsorption of the fibers to the negatively charged gold electrode can be explained by poor affinity of the hydrophobic fibers and hydrophilic surface of a positively charged electrode that is partially oxidized.[6]

The structures of oriented fibers of **1a** were examined by polarizing infrared spectroscopy. The aligned fibers were prepared by the application of an AC electric field (0.7 V/μm, 1 kHz) on the glass substrate with the gold electrodes covered with a KBr cover plate. Then, a part of the oriented fibers was transferred onto a KBr substrate (Figure 8a) by peeling off the KBr plate from the glass substrate. Figure 8b shows the polar plot of the absorbance for the C=O stretching band of the amide group (1638 cm^{-1}) of **1a**. The absorbance of the C=O band showed the maximum value in its intensities when the polarized light was parallel to the direction of applied electric field. The dichroic ratio (the ratio of the intensities for the absorbance in the directions parallel and perpendicular to the direction of electric field) of the C=O stretching band was 2.1. This observation suggests that the short axes of the molecule of **1a** are oriented in the direction parallel to the applied electric field. In contrast, no dichroism was observed for the randomly entangled fibers of **1a** and **2b**.

The mechanism of the formation of fibrous aggregates has been examined by Meijer and co-workers.[11] A solvent-assisted nucleation process occurs in the self-assembly of π-conjugated molecules into helical fibrous structures.[11] It was concluded that the solvent structures play a key role in rigidifying the aggregates and guiding them toward further assembly into fibers. In the present study, the formation of aligned fibrous aggregates may result from the nucleation and growth of the liquid crystal-like aggregates composed of aligned molecules having the fluorinated mesogens in the viscous solvent, then the aggregates solidified into fibrous solids.

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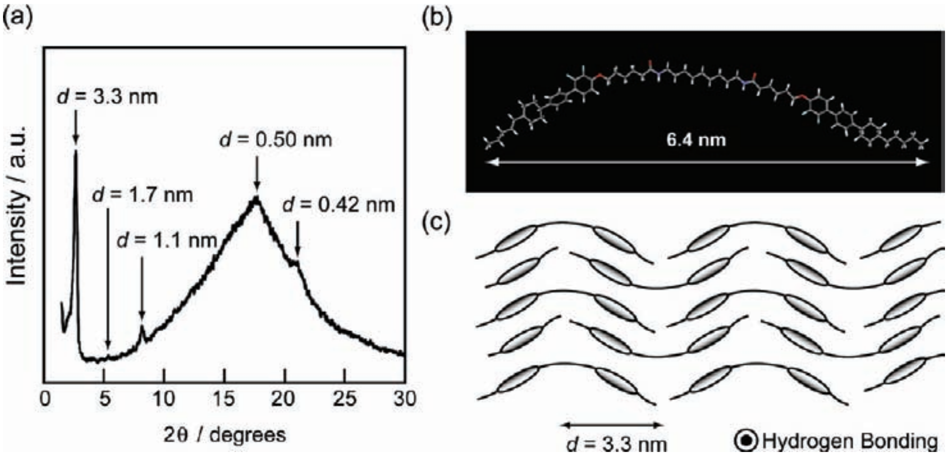

Figure 3. (a) X-ray diffraction pattern of the dodecylbenzene gel of **1a** (51 mM) at 25 °C. (b) Energy minimized molecular structure of **1a**. (c) Possible layer structure in the fibrous aggregate for **1a**.

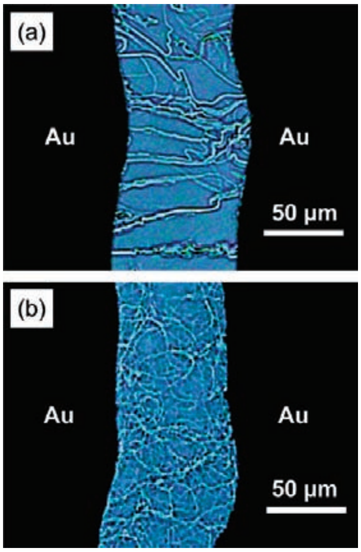

Figure 4. Optical micrographs of the fibers of **1a** formed between gold electrodes: (a) under an AC electric field (1.0 V/μm, 1 kHz) and (b) without an electric field.

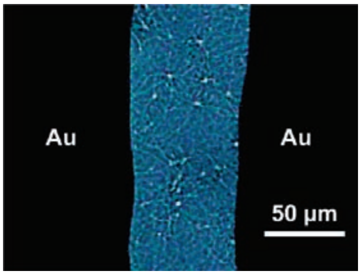

Figure 5. Optical micrograph of the entangled fibers of **2b** formed between gold electrodes under an AC electric field (1.0 V/μm, 1 kHz).

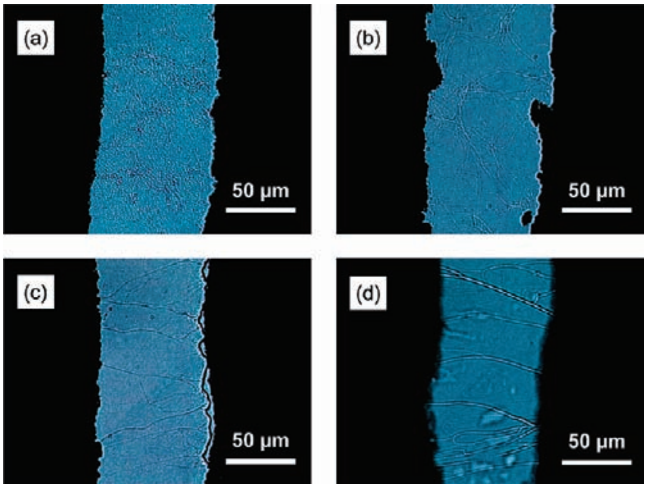

Figure 6. Optical microscopic images of the fibrous aggregates of **1a** formed in dodecylbenzene under AC electric fields: (a) 0.1 V/μm; (b) 0.3 V/μm; (c) 0.5 V/μm; (d) 1.0 V/μm.

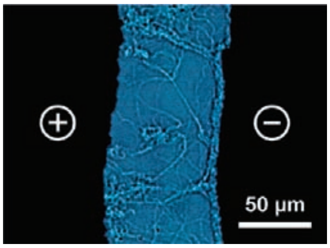

Figure 7. Optical photomicrograph of self-assembled fibers of **1a** in dodecylbenzene by applying a DC electric field (1.0 V/μm).

On the other hand, for nematic liquid crystals, it is known that the alignment of liquid crystals is influenced by the transit of charged species as impurities through the media under external electric fields.[12] In order to examine the effects of ionic impurities on the alignment of fibers, the transit current was measured for the dodecylbenzene mixture of **1a** in the sol and gel states. The values of current ranged from 40 to 140 nA. The values of current density were 0.25−0.88 μA cm^{-2}. For nematic liquid crystals, the current density was estimated to be 7 μA cm^{-2}.[12b] The amount of charged species in the dodecylbenzene solution of **1a** is smaller than that in the nematic liquid crystals. It is considered that the transit current through the samples is not essential for the alignment of fibers.

Formation of Aligned Polydiacetylene Fibers. Aligned fibrous aggregates of **3** that bridge the two gold electrodes were formed by the application of an AC electric field (1.0 V/μm, 1 kHz) in dodecylbenzene (Figure 9a,c), while randomly entangled fibers were formed without electric fields (Figure 9b,c). Photopolymerization of these fibrous aggregates was carried out by UV light irradiation (254 nm, 10 mW cm^{-2}) using a high pressure mercury lamp. The macroscopic orientation of the aligned fibers of **3** was successfully preserved by in situ photopolymerization of **3** (Figure 9d−f). The color of the fibers changed from translucent white to

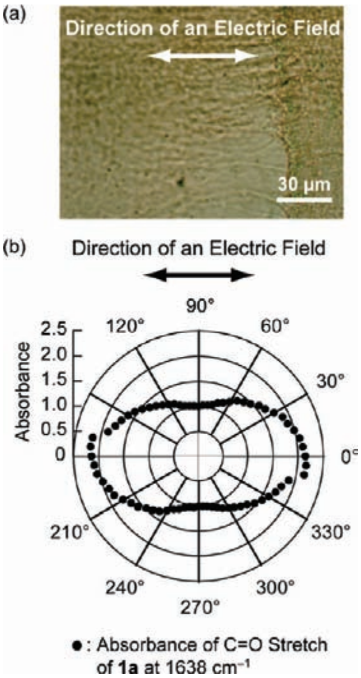

Figure 8. (a) Optical photomicrograph of the fibrous aggregates of **1a** transferred on a KBr substrate. The oriented fibers were formed in dodecylbenzene by the application of an AC electric field (0.7 V/μm, 1 kHz). (b) Polar plot of the IR band of C=O stretching at 1638 cm^{-1} for **1a** at room temperature.

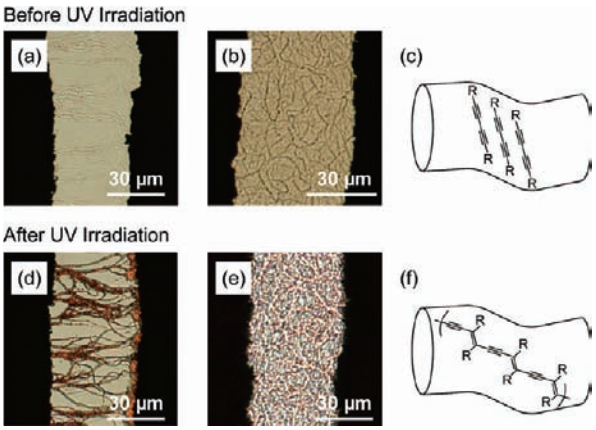

Figure 9. Optical micrographs and illustrations of the fibrous aggregates observed for the dodecylbenzene gel of **3** (8 mM): (a−c) before UV irradiation; (d−f) after UV irradiation; (a) aligned fibers formed under an AC electric field; (b) randomly entangled fibers without electric fields; (d) polymerized aligned fibers; (e) polymerized randomly entangled fibers.

red after UV irradiation for 30 min at room temperature. This change was monitored by UV−vis spectroscopy (see Supporting Information). An absorption band with a peak at 520 nm was observed after photoirradiation, which is attributed to the formation of π-conjugated poly(diacetylene)s.[9] Interestingly, a scanning electron microscopic (SEM) image of the polymerized sample of **3** showed left-handed and right-handed helical fibers of 300 nm in diameter and about 50 μm in length with a helical pitch of approximately 1 μm (Figure 10).

Conclusion

New hydrogen-bonded molecules bearing rod-shaped fluorinated mesogens were designed and synthesized as fiber-forming molecules. We succeeded in controlling the alignment of self-assembled fibers formed from the isotropic solution states by the application of AC electric fields. These self-assembled fibers may be useful for the development of future electronic and optoelectronic molecular materials.

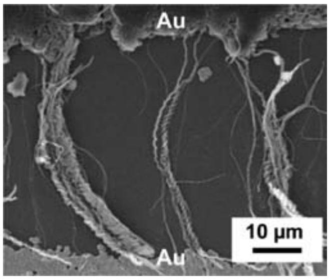

Figure 10. SEM image of the photopolymerized fibers of **3**.

Experimental Section

Gelation Test. An organic solvent (100 μL) was added to a weighted sample (5.0 mg) in a test tube. The tube was sealed and heated until a clear solution was obtained, and then the resultant mixture was cooled to room temperature. When the tube could be inverted without any flow, it was determined to be a "gel". When the compound was not dissolved completely, it was determined to be "insoluble". In case that reprecipitation occurred by cooling, it was defined as "precipitation".

Alignment of Fibrous Aggregates Using Electric Fields. Comb-shaped gold electrodes (thickness: 0.8 μm; gap: about 60 μm) deposited on a glass substrate were used for the experiment on the alignment of fibrous aggregates using electric fields. Alternating current (AC) electric fields (NF Corporation WF1943A as a power source and Echo Electronics Corporation ENP-3012 as an amplifier) were applied to the cells on cooling.

Measurement of Transit Current. The mixtures of **1a** and dodecylbenzene were introduced between two pieces of glass with indium tin oxide electrodes on their surfaces. The area of these cells was 0.16 cm^2. The thickness of the cell was 9.0 μm. A DC voltage of 9 V was applied for these cells. Transit currents were observed through a series load resistor (1 kΩ) by means of a digital oscilloscope.

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Supporting Information Available: Syntheses of **1**−**3**, IR spectra of the mixture of **1a** and dodecylbenzene, self-assembly of **2a** in dodecylbenzene with and without electric fields, XRD pattern and proposed self-assembled structures of **3** in the fibrous aggregates, UV−vis absorption spectra of **3**, polarized microscopic images of **2a** and **2b** aligned in electric fields. This material is available free of charge via the Internet at http://pubs.acs.org.

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