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Banana-shaped molecules with 4,6-dichlorinated central core: effect of lateral substituents and terminal chains on the formation of antiferroelectric smectic mesophases

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Six banana-shaped compounds with a central core based on a 4,6-dichloro-1,3-phenylene group were synthesized by varying the terminal chains $(R=OC_{10}H_{21} \text{ or } OC_{11}H_{21})$ and the lateral substituents (X=H, F or Cl). Their mesophases were characterized by a combination of differential scanning calorimetry, polarizing optical microscopy, triangular wave method, and X-ray diffractometry. Mesomorphic properties of the banana-shaped mesogens with an olefinic group (R=OC₁₁H₂₁) as a terminal chain are sensitive to lateral halogen substituents as much as those of the analogues with a saturated group (R=OC₁₀H₂₁). The compounds with X=F showed an antiferroelectric switchable smectic phase, which has been designated a B₂ phase. The compounds without a lateral halogen substituent only formed a nematic phase, while the compounds with X=Cl did not exhibit a mesophase in the melt.

1. Introduction

In 1996, Niori et al. [1] were first to report a non-linear liquid crystalline compound consisting of a bent aromatic mesogen with ester and imino linking groups and two octyl end groups ($R=C_8H_{17}$), 1,3-phenylene bis[4-(4-n-octylphenyliminomethyl)benzoate], and found that the achiral banana-shaped molecule could produce a ferroelectric mesophase. One year later, Link et al. [2] investigated the same $(R=C_8H_{17})$ and analogous ($R=OC_9H_{19}$) molecules, and proposed that the layers are chiral even though the molecules are achiral, by showing the layer structures of the SmCP phase based on the layer, tilt and polar planes. Since 1996, many studies on modifying the parent series of banana-shaped molecules, 1,3-phenylene bis[4-(4-nalkylphenyliminomethyl)benzoates] and 1,3-phenylene bis[4-(4-n-alkyloxyphenyliminomethyl)benzoates], have been made by a number of research groups [3–16]. It should be noted that some members of the alkyloxy series were already reported by Akutagawa *et al.* [17] but without investigation of the B-phase.

In 1999, Pelzl et al. [3] surveyed about banana-shaped liquid crystals of different molecular structures reported in the literature, which included the liquid crystalline behaviour of banana-shaped molecules with a central core based on a 4,6-dichloro-1,3-phenylene group [4]; the homologous series of 4,6-dichloro-1,3-phenylene bis[4-(4-n-alkyloxyphenyliminomethyl)benzoate]s showed the frustrated smectic C phase when the carbon number of the alkyloxy end group was larger than nine. In the same year, Lee and Chien [5] reported the first observation of a double-twisted helical structure in a banana-shaped achiral molecule with a chlorine atom as a lateral substituent (X=Cl) on the terminal rings, 1,3-phenylene bis[4-(3-chloro-4-n-octyloxyphenyliminomethyl)benzoate]. Later, we reported work on the mesomorphic and electrooptical properties of the same (X=CI) and analogous (X=F) molecules [10, 11]. Their mesomorphic and electro-optical properties were greatly influenced by the introduction of the halogen substituent: the

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compound with X=Cl showed a ferroelectric B_7 phase, and the compound with X=F exhibited an antiferroelectric B_2 phase.

In an earlier paper [12], we reported synthesis of derivatives of 4,6-dichloro-1,3-phenylene bis[4-(4-nalkyloxyphenyliminomethyl)benzoate] containing lateral halogen substituents in the terminal rings, and found that as the carbon number of the alkyloxy end group is eight, the molecules could not form the B-phase but showed nematic and smectic phases even on varying the lateral substituent on the terminal rings. We also have investigated analogous molecules introducing an unsaturated $(R = OC_{11}H_{21})$ as well as saturated $(R=OC_{10}H_{21})$ alkyloxy group, longer than the octyloxy group ($R = OC_8 H_{17}$), into the terminal chains. While we were conducting this investigation, Weissflog and coworkers investigated the same structures with saturated alkyloxy groups and found that the bent-core mesogens could form antiferroelectric smectic CP phases when the carbon number of the terminal chain was larger than eight [14, 15]. They also reported that the introduction of lateral substituents on the central ring (F, Cl, CN, NO₂, CH₃) and/or on the terminal rings (Cl, Br, CH₃) can significantly change the mesophase behaviour [16].

In this paper, we report the synthesis of three new banana-shaped compounds containing a 4,6-dichlorinated central core and an olefinic terminal chain, characterization of their mesophases, and the effect of the lateral halogen (X=H, F or Cl) on the formation of antiferroelectric switchable mesophase.

2. Experimental

2.1. Synthesis

The synthetic route followed to obtain the bananashaped molecules used a modified literature procedure [12, 18] and is shown in figure 1. Since the synthetic procedures for the six compounds were essentially similar, one representative synthesis for 4,6-dichloro-1,3-phenylene bis{4-[4-n-(10-undecenyloxy)-phenyliminomethyl]benzoate} is described. First, 4-nitro-1-(10undecenyloxy)benzene was prepared using a substitution reaction of 4-nitrophenol and 11-bromo-1-undecene. Then, 4-(10-undecenyloxy) aniline was obtained by hydrogenation of 4-nitro-1-(10-undecenyloxy)benzene with iron in a ternary mixture of CH₂Cl₂/MeOH/ H₂O (1/1/1, v/v). Next, 4,6-dichloro-1,3-phenylene bis(4-formyl benzoate) was prepared by reaction of



Figure 1. Synthetic route to compounds 1 and 2.

4,6-dichloro-resorcinol and 4-formyl benzovl chloride in THF with triethylamine at 0°C. The final product was prepared through a condensation reaction between the aniline and the dialdehyde. It was purified by chromatography on silica gel, and recrystallized several times from a mixture of ethanol and dimethylforamide (20/1, v/v). The yield after purification was about 20-30%. ¹H NMR (CDCl₃): δ in ppm=1.0–1.6 [m, 24H, -(CH₂)₆-], 2.0 (m, 4H, -OCH₂CH₂-), 2.1 (m, 4H, $-CH_2CH=$), 4.0 (t, 4H, $-OCH_2-$), 5.0 (q, 4H, $=CH_2$), 5.8 (m, 2H, -CH=), 7.0-8.3 (m, 18H, Ar-H), 8.5 (s, 2H, -CH=N-); EIMS (m/z): 930 (M). HRMS (E.I.): calcd for $C_{56}H_{63}ClN_2O_6$ (M⁺) 928.3985; found 928.3992. The other banana-shaped compounds were prepared and confirmed by the same method as for the 4,6-dichloro-1,3-phenylene bis{4-[4-n-(10-undecenyloxy)phenyliminomethyl]benzoate}.

2.2. Characterization

NMR spectra were obtained using a Brücker DRX NMR spectrometer (500 MHz). Mass spectra were measured on a JEOL JMS-700 spectrometer. The transition behaviours were characterized by differential scanning calorimetry (Perkin-Elmer DSC7) and polarizing optical microscopy (Nikon Eclipse E400 POL). DSC measurements were performed in a N₂ atmosphere with heating and cooling rates of 5° C min⁻¹. Optical texture observation was carried out using the polarizing microscope with a hot stage. The switching current was examined by the triangular wave method [19]. The sample cell was mounted in a microfurnace to measure spontaneous polarization at varying temperatures. The temperature fluctuations inherent to the furnace were approximately 0.1°C. For direct measurement of the polarization, we used the triangular wave method for ease of subtracting the background current. The polarization current, converted into a voltage signal through an amplifier, was measured using a digitizing oscilloscope and fed into a computer for data analysis.

SAXS measurements were performed in transmission mode with synchrotron. Two dimensional scattering patterns were collected on a CCD (charge coupled device) detector (Roper Scientific) with a wavelength of 1.608 Å. The observed intensity was corrected for background scattering and absorption by the sample. Since the samples exhibited typical isotropic scattering patterns in the absence of an external field, the intensity data were normalized to one-dimensional scattering patterns by integration through circular averaging to improve the data resolution, and reduce the measurement time.

Table 1. Phase transition temperatures (°C) and corresponding enthalpy changes (kJ mol⁻¹, in parentheses) of compounds 1 and 2



Compound	R/X	Transition temperature ^a
1a	OC ₁₀ H ₂₁ /H	Cr 114(14.4) N 147(1.4) I
		I 145(1.1) N 92(2.1) Cr
1b	$OC_{10}H_{21}/F$	Cr 79(17.1) ^b , 107(23.3) SmA 137(4.0) I
		I 131(4.2) SmA 110(^c) SmCP _A 54 (4.1) Cr
1c	$OC_{10}H_{21}/Cl$	Cr 97(13.8) ^b , 117(7.8) I
		I 106(3.1) Cr
2a	$OC_{11}H_{21}/H$	Cr 107(8.1) N 136(2.2) I
		I 135(1.3) N 77(1.4) Cr
2b	$OC_{11}H_{21}/F$	Cr 102(8.0) SmA 128(3.6) I
		I 126(7.5) SmA 95(°) SmCP _A C 55(4.1) Cr
2c	$OC_{11}H_{21}/Cl$	Cr 99 (21.6) I
		I 90(2.2) Cr

^aThe phase sequence in the lower line is observed on cooling; $SmCP_A$ =antiferroelectric polar smectic C phase. ^bSolid-solid transition temperature. ^cThe calorimetric peaks of the SmA-SmCP_A transition could not be resolved.



Figure 2. Optical micrographs of compound 1 on cooling from the isotropic liquid. (a) Nematic droplets of compound 1a at 145.1°C; (b) homeotropically aligned texture of 1a at 143.5°C; (c) smectic granular pattern of compound 1b at 129.6°C; (d) broken-fan texture of 1b at 120.2°C.

WAXS measurements were conducted with an apparatus consisting of an 18kW rotating anode Xray generator (Rigaku Co.) operated at 50 kV and 20 mA, mirror optics with point focusing, and a onedimensional position sensitive detector (M. Braun Co.). The CuK_{α} radiation (λ =1.542 Å) from a 0.1 × 1 mm² microfocus cathode was used as a light source. A beam path was maintained under vacuum to reduce air scattering, and tungsten foil (50 µm) was used for primary beam protection. The data are presented as a function of $q=4\pi\sin\theta/\lambda$, where θ is the scattering angle and λ the X-ray wavelength. In order to investigate structural changes caused by heating, the samples were held in an aluminum sample holder, sealed with a window of 7 µm thick Kapton film on both sides. The sample was heated with two cartridge heaters and its temperature monitored by a thermocouple placed close to the sample.

3. Results and discussion

3.1. Synthesis and mesomorphic behavior

The synthetic route for the banana-shaped compound is quite straight forward and each reaction step is relatively well known. The compounds obtained were characterized by NMR and mass spectroscopy, which provided data in accordance with expected formulae. As shown in table 1, phase transition temperatures of the compounds depend on the variation of the terminal flexible group (R) and lateral halogen atom (X) at the outer phenylene unit. The compounds with X=F, such as **1b** and **2b**, showed a smectic phase with switching properties, while compounds without a halogen substituent, such as **1a** and **2a**, showed only the nematic phase.

Recently, it was reported that the introduction of a polar group into the 3-position of the *p*-alkoxyaniline Schiff's base moiety could affect the mesophase formation



Figure 3. Optical micrographs of compound **2** on cooling from the isotropic liquid. (a) Nematic droplets of compound **2a** at 133.4° C; (b) marble texture of **2a** at 125.0° C; (c) smectic germ-like structure of compound **2b** at 124.2° C; (d) unknown texture of **2b** at 100.2° C.

in two ways depending on polarity, namely, the Hammett Substituent Constant (σ) value [20, 21]: an electron-donating group could enhance electrostatic repulsion in the direction of the dipoles, and an electron-withdrawing group is expected to decrease electrostatic repulsion in the direction of the dipoles. Thus, banana-shaped molecules containing a halogen substituent with positive σ value can have decreased intralamellar electrostatic repulsion in the direction of the longitudinal dipoles, often with a more pronounced tendency to form a smectic phase than their analogues without halogen substituent. In addition, the introduction of a lateral halogen substituent into the 3-position of an outer 1,4-phenylene unit results in a decrease of the melting temperature over the molecules without halogen substituent. This is because the presence of the lateral halogen substituent together with the bulky dichloro substituent in the central phenylene unit prevents the regular stacking of the banana-shaped molecules in the solid state. Moreover, for compounds

1c and **2c**, the lateral Cl substituent, which is less polar but larger in size then the lateral F substituent, prevents regular stacking of the bent molecules along the direction of the longitudinal dipoles in the fluid state so that they can not form a mesophase.

Although compound 2 with an olefinic side chain has no more pronounced tendency to form a nematic mesophase than their saturated analogues, compound 1, generally compound 2 showed lower transition temperatures for melting and isotropization than compound 1. Comparison between compounds 1 and 2 shows that the lateral fluorination is clearly effective in forming an antiferroelectric smectic C phase, whereas the dependence of mesomorphic properties on alkyloxy end group is not simple. Interestingly, as the carbon number increased from C_8 to C_{10} [12] to obtain the smectic C phase, the compound with X=Cl could not even form a mesophase. On the other hand, replacing the saturated C_{10} with olefinic C_{11} did not affect their mesophase property.



Figure 4. Switching current curves obtained by applying a triangular voltage wave at 70° C and 80° C for (a) **1b** and (b) **2b**.

3.2. Microscopic textures

Using an optical microscope with crossed polarizer, on cooling the isotropic liquid we could identify every phase transition. When the isotropic liquid of compound **1a** is cooled slowly, at the clearing point the optical texture of the nematic phase appeared as a drop-like four-brush structure, figure 2(a), and on further cooling it developed into a homeotropically aligned texture with schlieren remnants, figure 2(b). The smectic phase for compound **1b** initially shows a striped

granular pattern at the clearing point, figure 2(c), which forms a broken fan-shaped texture on further cooling, figure 2(d). The nematic phase for compound **2a** initially appears as a drop-like two-brush structure at the clearing point, figure 3(a), and then forms a marble texture with disclinations on further cooling, figure 3(b). The optical texture of the smectic phase for compound **2b** initially appears as a germ-like structure at the clearing point, figure 3(c), and then forms an unknown texture with thread remnants on further cooling, figure 3(d).



Figure 5. Temperature dependence of spontaneous polarization on cooling the isotropic liquid for (a) 1b and (b) 2b.

3.3. Electro-optical studies

The electro-optical properties of the smectic mesophase of compounds **1b** and **2b** (X=F) were investigated. For this measurement, the samples were prepared in a cell consisting of conductive indium tin oxide-coated glasses treated with rubbed polyimide for alignment. The cell gap was maintained by patterned organic spacers of 1.5 µm thickness. The spontaneous polarization of the smectic mesophase was measured by applying a triangular shape voltage, and the switching was also observed by using the polarizing microscope. Figure 4 shows the switching current response for compounds **1b** and **2b** measured at 70°C and 80°C (within the smectic phase forming region). By applying a sufficiently high triangular voltage, two reversal current peaks per half period were recorded on the oscilloscope screen. This clearly indicates that the smectic phases of these compounds are antiferroelectric with the tip of the bent molecule orienting along the electric field and reversing its orientation on the polarity of the field. Figure 5 shows the temperature dependence of spontaneous



Figure 6. X-ray diffraction patterns of compound **2a**: upon heating as-prepared solid sample, measured at (*a*) room temperature and (*b*) 150° C; upon cooling the isotropic liquid of the same sample, measured at (*c*) 90° C and (*d*) 60° C.

polarization for compounds **1b** and **2b** in the cooling experiment. In figure 5, when the isotropic liquid is cooled from the starting temperature of a switchable smectic phase, the spontaneous polarizations increase with decreasing temperature and then exhibit a maximum of 355 and $245 \,\mathrm{nC \, cm^{-2}}$, respectively. Further lowering the temperature below $50^{\circ}\mathrm{C}$ caused the polarization to vanish because of crystallization of the bent-core mesogen.

3.4. X-ray studies

Figures 6–8 display the X-ray diffraction patterns obtained at a given temperature. The nematic meso-phases of compounds **1a** and **2a**, identified by observation of their optical textures, were confirmed by X-ray



Figure 7. X-ray diffraction patterns of compound **1b** on heating as-prepared solid sample, measured at (*a*) room temperature, (*b*) 90°C, (*c*) 120°C, and (*d*) 150°C.

experiments. As shown in figure 6, first, a crystalline sample of compound 2a was heated to 150°C to obtain the isotropic phase; it was then cooled to obtain the typical X-ray pattern for a nematic mesophase, showing broad but distinctively strong peak around а $q=1.476 \text{ nm}^{-1}$ at 90°C. On further cooling the mesophase, a crystalline pattern was obtained at 60°C. Previously, we reported that in cases where bananashaped molecules form the nematic phase, molecules could self-assemble into a rod-like cylinder consisting of smectic columnar structure of bent molecules, which orient like a nematic mesogen [20]. In the same manner, the large distance of about 40 Å in the nematic mesophase of these compounds may be originated from interference among these self-assembled cylinders.



Figure 8. X-ray diffraction patterns of compound 1c on heating as-prepared solid sample, measured at (a) room temperature and (b) 160° C.

In figure 7, when a crystalline sample of compound 1b was heated to 90°C, the diffraction pattern of the small angle region changed, but that of the wide angle region did not disappear, indicating a second solid state. When this solid state was further heated to 120°C (within the temperature range of mesophase formation) these compounds showed smectic characteristics in their diffraction patterns: relatively sharp peaks appear in a small angle region, and no crystalline peaks are found in the wide angle region. On further heating to 150°C, the obtained diffraction pattern indicates the isotropic liquid phase. Compound 2b showed similar X-ray diffraction patterns to those of **1b**, except for the absence of the solid-solid transition on heating (see table 2). The smectic phases of compounds 1b and 2b showed two reflections with a 1:2 ratio of peak scattering vectors in their small angle regions. The layer spacing (d) was calculated to be 4.71 and

Table 2. Comparison of the molecular length (L), layer spacing (d) and tilt angle (θ)

Compound	<i>L</i> /nm	<i>d</i> /nm	$ heta I^\circ$
1b	54	4.71	29
2b	58	4.84	33

4.84 nm from the peak at q=1.333 and 1.297 nm^{-1} , respectively. The molecular length (*L*) for compounds **1b** and **2b** was measured as 54 and 58 nm by assuming the alkyl chain to be fully extended in the all*trans*-conformation, and the tilt angle (θ) for the molecules was estimated to be about 29° and 33°, respectively. Compounds **1c** and **2c** showed crystalline diffraction patterns with $q=1.333 \text{ nm}^{-1}$ at room temperature, but these compounds showed only diffraction patterns for isotropic liquid in the melt (see figure 8).

4. Conclusions

We synthesized six halogen-substituted banana-shaped molecules with n-decyloxy or 10-n-undecenyloxy end groups. The parent compounds with no lateral halogen substituent exhibit only nematic phases in the melt. On the other hand, we could obtain B₂-phases by introducing a fluorine substituent in the 3-position of the palkoxyaniline Schiff's base moiety of the banana-shaped molecules; unexpectedly, the banana-shaped molecules with X=Cl do not form a liquid crystal phase. Thus we found that the lateral halogen substituent plays an important role in mesomorphic behaviour for this system of centrally dichloro-substituted bananashaped molecules, whereas the olefinic side chain $(R=OC_{11}H_{21})$, known often to lead to lower rotational viscosities, shows no stronger influence in forming a nematic mesophase than the saturated analogue $(R = OC_{10}H_{21}).$

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