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Synthesis and mesomorphic properties of fluoro-substituted chiral liquid crystals derived from (*S*)-lactic acid with alkoxyethanols

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A homologous series of fluoro-substituted chiral liquid crystals derived from (*S*)-lactic acid and alkoxyethanols were prepared for investigation. Mesophases and their corresponding transition temperatures were identified by polarized optical microscopy and differential scanning calorimetry. The compounds exhibit a broad temperature range in the ferroelectric chiral smectic C (SmC*) phase; in particular, compounds with shorter alkyl chain lengths have an SmC* phase at ambient temperature. The physical properties of the ferroelectric SmC* phase, such as switching current, spontaneous polarization, optical tilt angle and electro-optical response, were also measured. The effects of fluoro substituent on the mesophases and physical properties of the chiral liquid crystals are discussed.

1. Introduction

Optically active lactic acid is commonly used as a chiral precursor for the preparation of chiral liquid crystals [1–14]. In general, the mesophase properties of the lactic acid derivatives display a broad ferroelectric chiral smectic C (SmC*) phase and, in a few cases, an antiferroelectric SmC_A* phase [11–14]. A precious study on the chiral liquid crystals, *nEmBCPCP* (see scheme 1), derived from (*S*)-lactic acid and alkoxyethanols showed that the compounds have a broad ferroelectric phase; in particular, some compounds exhibit a ferroelectric SmC* phase close to room temperature [15]. In order to explore further, structurally similar compounds, *nEFmBCPCP* (see scheme 1) with a fluoro substituent in the 2-position of phenyl ring that is closed to the chiral group, were synthesized for the investigation of mesomorphic phases and physical properties.

Fluorine has been designed as the lateral substituent on mesogenic core of liquid crystal molecules [16–26]. Incorporation of a fluoro lateral substituent in the mesogenic core of a molecule causes some degree of steric asymmetry when molecules are packed together. Therefore, it reduces the occurrence of higher order smectic phase, and decreases the melting point in most of cases [23]. Some other reports on liquid crystals also indicate that introduction of a fluoro lateral substituent into the mesogenic core can enhance the formation of a titled smectic C phase [24–26].

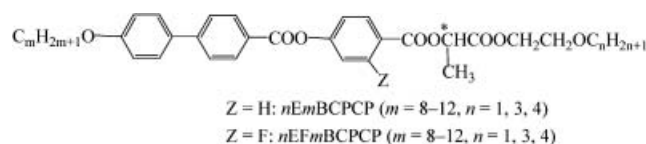
2. Experimental

2.1. Characterization of the materials

The chemical structures of intermediates and target compounds were identified by ¹H NMR spectroscopy using a JEOL-500 FT-NMR spectrometer. The purity of the final compounds was checked by thin layer chromatography (TLC) and further confirmed by elemental analyses using a Perkin-Elmer 2400 spectrometer.

The phase transition temperatures and corresponding phase transition enthalpies of the compounds were determined by differential scanning calorimetry (DSC) using a Perkin-Elmer DSC 7 calorimeter at a scanning rate of 5°C min⁻¹. Mesophases were principally identified by Nikon Microphot-FXA optical microscopy in conjunction with hot stage Mettler FP82-HT or INSTEC HS1 using clean, untreated microscope slides and cover slips. All transition sequences were recorded on cooling from the isotropic (I) liquid at a cooling rate of 5°C min⁻¹.

The ferroelectricity of the compounds was characterized by observing switching behavior and optical response in 5 μm parallel-aligned cells that were



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Scheme 1. Chemical structure of compounds investigated.

purchased from E.H.C. Co, Japan. The magnitude of spontaneous polarization (P_s) was measured by a triangular wave method [27]. The optical setup for transmittance measurements involved using a He-Ne laser (5 mW, 632.8 nm) as the probe beam [28, 29]. The transmittance of the probe beam through the cell between the crossed polarizers, the axes of which were parallel and perpendicular to the smectic layer normal, was detected by a photodiode. The optical tilt angle was measured using a cell placed in the hot stage and the temperature dependence of the tilt angle determined. A digital oscilloscope (HP54502A) monitored the signals. The voltage applied to the cell was produced by an arbitrary waveform generator (AG1200) and amplified by a homemade power preamplifier.

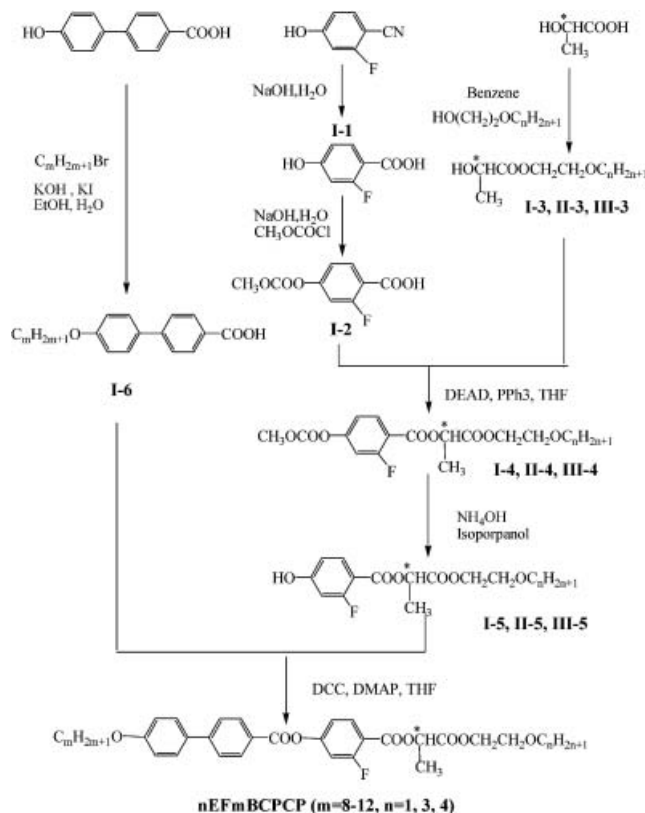
2.2. Preparation of materials

The starting chiral compound, (*S*)-lactic acid, was purchased from Fluka Co. Chem., Japan, with optical purity greater than 99%. 2-Methoxyethanol, 2-propoxyethanol and 2-butoxyethanol were also purchased from Fluka Co. Chem., Japan. TLC was performed with sheets coated with silica; spots were detected by UV irradiation. Silica gel (MN kieselgel 60, 70–230 mesh) was used for column chromatography. Anhydrous organic solvents, dichloromethane (CH_2Cl_2) and tetrahydrofuran (THF), were purified by treatment with CaH_2 and LiAlH_4 , respectively, and distilled before use.

The synthetic processes of the target compounds were carried out as outlined in scheme 2. Some intermediates were prepared according to the synthetic procedures described in the literature [12, 30, 31]. Other new intermediates and target compounds were prepared according to the process description below.

2.2.1. Synthesis of 2-alkoxyethyl (*S*)-2-[2'-fluoro-4-(methoxycarbonyloxy)phenylcarbonyloxy]propionate, I-4, II-4, III-4 [32].

A solution of diethyl azodicarboxylate (DEAD, 1.92 g, 11 mmol) and compound I-2 (2.35 g, 11 mmol) in 10 ml anhydrous THF was added dropwise to a solution of triphenyl phosphine (Ph_3P , 3.41 g, 13 mmol) and compound I-3, II-3 or III-3 (10 mmol) in 10 ml anhydrous THF at room temperature with vigorous stirring. The reaction soon started. After the mixture had been kept standing overnight at room temperature, the triphenylphosphine oxide was removed by filtration. THF was removed in vacuo. After work-up procedure, the products were isolated by column chromatography over silica gel (70–230 mesh) using ethyl acetate/hexane (1/4) as eluent to give colourless liquids. Yields of 50–70% for compounds I-4, II-4 and III-4 were obtained. For I-4,



Scheme 2. Synthetic procedures for the target compounds *n*EF*m*BCPCP ($m=8-12$, $n=1, 3, 4$).

^1H NMR (500 MHz, CDCl_3): δ (ppm) 8.02–8.06 (t, 1H, ArH), 7.08–7.10 (m, 2H, ArH), 5.35–5.36 (q, 1H, $-\text{COOCH}-$), 4.32–4.35 (t, 2H, $-\text{COOCH}_2\text{CH}_2\text{O}-$), 3.94 (s, 3H, $\text{CH}_3\text{OCOO}-$), 3.60–3.62 (t, 2H, $-\text{COOCH}_2\text{CH}_2\text{O}-$), 3.37–3.41 (s, 3H, $-\text{OCH}_3$), 1.64–1.67 (d, 3H, $-\text{CHCH}_3$). For compound II-4, ^1H NMR (500 MHz, CDCl_3): δ (ppm) 8.02–8.05 (t, 1H, ArH), 7.07–7.10 (m, 2H, ArH), 5.34–5.38 (q, 1H, $-\text{COOCH}-$), 4.30–4.36 (t, 2H, $-\text{COOCH}_2\text{CH}_2\text{O}-$), 3.94 (s, 3H, $\text{CH}_3\text{OCOO}-$), 3.63–3.65 (t, 2H, $-\text{COOCH}_2\text{CH}_2\text{O}-$), 3.39–3.42 (t, 2H, $-\text{OCH}_2\text{CH}_2\text{CH}_3$), 1.64–1.65 (d, 3H, $-\text{CHCH}_3$). For compound III-4, ^1H NMR (500 MHz, CDCl_3): δ (ppm) 8.02–8.06 (t, 1H, ArH), 7.08–7.10 (m, 2H, ArH), 5.33–5.38 (q, 1H, $-\text{COOCH}-$), 4.31–4.34 (t, 2H, $-\text{COOCH}_2\text{CH}_2\text{O}-$), 3.94 (s, 3H, $\text{CH}_3\text{OCOO}-$), 3.63–3.65 (t, 2H, $-\text{COOCH}_2\text{CH}_2\text{O}-$), 3.43–3.46 (t, 2H, $-\text{OCH}_2\text{CH}_2\text{CH}_2-$), 1.62–1.64 (d, 3H, $-\text{CHCH}_3$).

2.2.2. Synthesis of 2-alkoxyethyl (*S*)-2-(2'-fluoro-4-hydroxyphenylcarbonyloxy)-propionates, I-5, II-5, III-5 [32].

Compounds I-4, II-4 or III-4 (3 mmol) were stirred in a mixture of isopropanol (90 ml) and ammonia (28%, 30 ml) at room temperature for 60 min (TLC analysis revealed a complete reaction) and then

poured into water (40 ml) while stirring. The product was extracted using dichloromethane (3 × 30 ml). The combined ethereal extracts were washed with brine (50 ml), dried (MgSO₄), filtered and evaporated to give colourless oils. The oils were purified by flash column chromatography over silica gel (70–230 mesh) using dichloromethane; the resulting oil was then dried in vacuo. Yields of 90–92% for compounds **I-5**, **II-5** and **III-5** were obtained.

2.2.3. 2-Alkoxyethyl (*S*)-2-[2'-fluoro-4-(4'-alkoxybiphenylcarbonyloxy)phenylcarbonyloxy]propionates, *n*EFmBCPCP (*m*=8–12, *n*=1, 3, 4) [32]. A mixture of 4-(4'-alkoxybiphenyl)benzoic acid (3.9 mmol), *N,N'*-dicyclohexylcarbodiimide (0.79 g, 3.9 mmol), compound **I-5**, **II-5** or **III-5** (3.5 mmol), 4-dimethylaminopyridine (0.04 g, 0.35 mmol) and dry THF (15 ml) was stirred at room temperature for three days. The precipitates were filtered and washed with 5% acetate acid solution (3 × 50 ml), 5% saturated aqueous sodium hydrogen carbonate (3 × 50 ml) and water (3 × 50 ml), dried over anhydrous magnesium sulfate (MgSO₄) and concentrated in vacuum. The residue was purified by column chromatography over silica gel (70–230 mesh) using ethyl acetate/hexane (1/2) as eluent. After purification by recrystallization from absolute ethanol, yields of 40–50% final products were obtained. For *n*EFmBCPCP (*m*=10, *n*=1) as an example, ¹H NMR (500 MHz, CDCl₃): δ (ppm) 8.21–8.22 (d, 2H, –ArH), 8.08–8.09 (t, 1H, –ArH), 7.70–7.72 (d, 2H, –ArH), 7.60–7.61 (d, 2H, –ArH), 7.14–7.16 (m, 2H, –ArH), 7.00–7.01 (d, 2H, –ArH), 5.37–5.38 (q, 1H, –COOCHCH₃), 4.34–4.35 (t, 2H, –COOCH₂CH₂–), 4.01–4.03 (t, 2H, –CH₂CH₂O–), 3.61–3.64 (t, 2H,

–COOCH₂CH₂–), 3.38 (s, 3H, –OCH₃), 1.65–1.67 (d, 3H, –COOCHCH₃). Elemental analysis results for the target compounds are listed in table 1.

3. Results and discussion

3.1. Mesomorphic properties

The mesophases and their corresponding phase transition temperatures were mainly determined by use of polarizing optical microscopy and DSC. The chiral smectic A (SmA*) phase was characterized by the formation of a focal-conic texture and the SmC* phase by the formation of striated focal-conic texture. Detailed data of phase transition temperatures and corresponding enthalpies of the transitions are shown in table 2. All compounds exhibit enantiotropic SmA* and SmC* phases. An additional unidentified higher ordered SmX* phase occurred at longer alkyl chain lengths (*m*=11, 12) for three series of compounds.

The previous study on non-fluoro-substituted compounds revealed the occurrence of a ferroelectric SmC* phase close to room temperature. The present study on fluoro-substituted compounds shows that the corresponding I–SmA* and SmA*–SmC* transition temperatures and melting points are decreased due to the effect of the fluoro substituent and, consequently, these compounds exhibit a ferroelectric SmC* phase at ambient temperature for shorter alkyl chain members: i.e. *n*EFmBCPCP (*m*=8–10, *n*=1), *n*EFmBCPCP (*m*=8–9, *n*=3) and *n*EFmBCPCP (*m*=8–10, *n*=4).

3.2. Switching behaviour

The physical properties of the compounds were measured in 5 μm homogenous aligned cells. The

Table 1. Elemental analysis results for the final compounds *n*EFmBCPCP (*m*=8–12, *n*=1, 3, 4).

Compound	Elemental analysis			
	C/%	H/%	C/%	H/%
1EF8BCPCP	68.67	6.61	68.71	6.66
1EF9BCPCP	69.06	6.79	69.14	6.82
1EF10BCPCP	69.44	6.96	69.51	6.98
1EF11BCPCP	69.79	7.12	70.03	7.17
1EF12BCPCP	70.13	7.28	70.10	7.34
3EF8BCPCP	69.44	6.96	69.54	7.01
3EF9BCPCP	69.79	7.12	70.05	7.05
3EF10BCPCP	70.13	7.28	70.16	7.28
3EF11BCPCP	70.46	7.43	70.46	7.42
3EF12BCPCP	70.77	7.57	70.73	7.61
4EF8BCPCP	69.79	7.12	69.92	7.13
4EF9BCPCP	70.13	7.28	7.03	7.35
4EF10BCPCP	70.46	7.43	70.48	7.50
4EF11BCPCP	70.77	7.57	70.75	7.60
4EF12BCPCP	71.08	7.71	71.08	7.78

Table 2. Transition temperatures and associated enthalpy data for the chiral materials $nEFmBCPCP$ ($m=8-12$, $n=1, 3, 4$).

Transition temperatures/ $^{\circ}C^a$											
m	n	I	SmA*	SmC*	SmX*	Cr ^b	m.p. ^c				
8	1	•	137.75 [4.17] ^e	•	86 ^d _f	•	–	–10.43 [2.04]	•	24.46 [8.80]	
9	1	•	134.14 [4.83]	•	92 _f	•	–	–1.96 [12.64]	•	15.92 [12.53]	
10	1	•	132.34 [3.95]	•	97 _f	•	–	5.28 [13.48]	•	24.03 [15.77]	
11	1	•	127.48 [4.02]	•	99 _f	•	40.6 [8.57]	•	21.23 [14.03]	•	39.01 [14.26]
12	1	•	124.65 [7.72]	•	89 _f	•	50.93 [8.67]	•	26.41 [12.09]	•	36.5 [12.92]
8	3	•	129.3 [6.13] ^e	•	80 ^d _f	•	–	<–30 _f	•	11.75 [12.49]	
9	3	•	123.45 [3.74]	•	81 _f	•	–	–7.83 [8.33]	•	24.66 [13.24]	
10	3	•	123.17 [4.22]	•	84 _f	•	–	1.27 [12.18]	•	27.22 [16.45]	
11	3	•	120.16 [2.99]	•	86 _f	•	22.7 [3.74]	•	8.30 [10.69]	•	33.23 [16.85]
12	3	•	117.83 [4.00]	•	88 _f	•	27.43 [2.4]	•	16.33 [14.26]	•	37.41 [21.87]
8	4	•	124.74 [3.15] ^e	•	71 _f	•	–	<–30 _f	•	5.89 [7.09]	
9	4	•	120.41 [3.08]	•	79 _f	•	–	–16.79 [6.81]	•	21.98 [15.70]	
10	4	•	119.43 [3.53]	•	84 _f	•	–	–11.91 [9.33]	•	21.1 [18.51]	
11	4	•	116.9 [5.19]	•	88 _f	•	26.69 [6.44]	•	6.82 [12.03]	•	31.62 [16.42]
12	4	•	116.25 [2.77]	•	88 _f	•	34.89 [2.53]	•	16.09 [8.94]	•	36.13 [10.60]

^aRecorded by DSC at cooling rates of $5^{\circ}C\ min^{-1}$. ^bCr=crystal. ^cm.p.=melting point taken from DSC thermograms recorded at heating rates of $5^{\circ}C\ min^{-1}$. ^dThe SmA*–SmC* phase transition temperature was determined by optical microscopy. ^eFigures in square parenthesis denote enthalpies in $kJ\ mol^{-1}$. ^fThe enthalpy of transition was too small to be determined by DSC.

electrical switching response was measured under a triangular wave voltage with field frequency 20 Hz and amplitude 5 V. A typical example of the electrical switching responses obtained for $nEFmBCPCP$ ($m=8$, $n=3$) is shown in figure 1. The switching exhibits one current peak in the range of temperatures similar to behaviour reported for the SmC* phase [33]. The strength of these current peaks increases with decreasing temperature, and the relative position of the peaks is slightly shifted to higher values.

3.3. Spontaneous polarization

The spontaneous polarization (P_s) of the compounds in the SmC* phase was measured in $5\ \mu m$ LC cells made of ITO-coated glass, the surface of which had been treated with a parallel aligned polyimide, at 25 Hz applied frequency and $5\ V\ \mu m^{-1}$ applied voltage. The P_s values were measured after slowly cooling the compound to the SmC* phase. Figure 2 shows the spontaneous

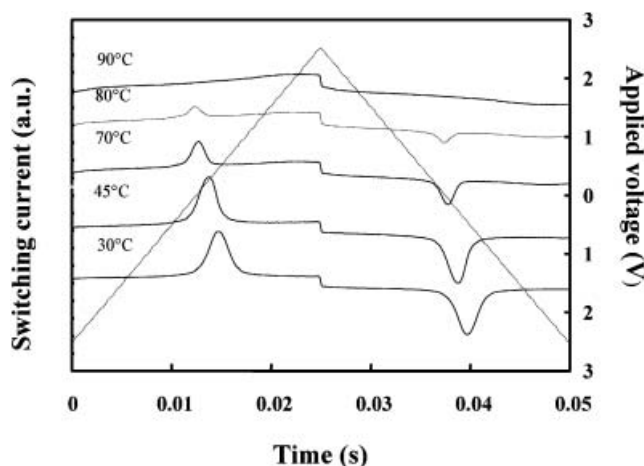


Figure 1. Switching current behaviour of $nEFmPBPP$ ($m=8$, $n=3$) in the SmA* phase at $90^{\circ}C$ and SmC* phase at $80^{\circ}C$, $70^{\circ}C$, $45^{\circ}C$ and $30^{\circ}C$ in a $5\ \mu m$ homogeneously aligned cell.

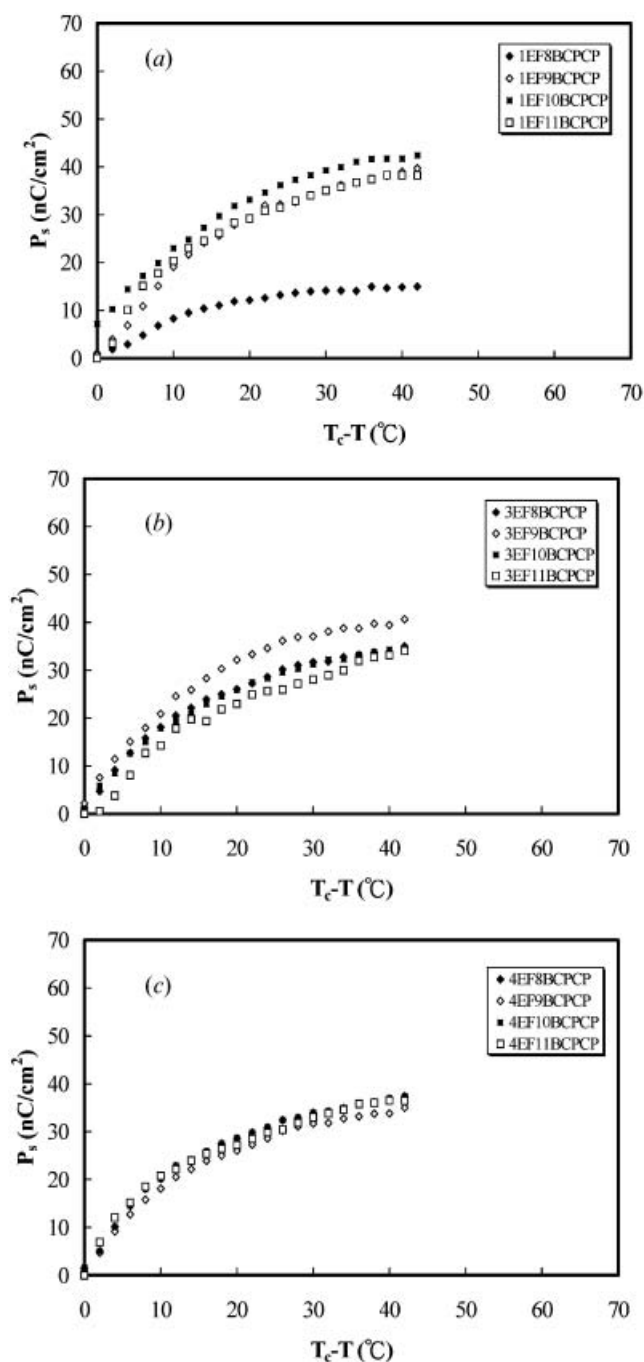


Figure 2. Spontaneous polarization (P_s) plotted as a function of temperature for compounds (a) $nEFmBCPCP$ ($m=8-11$, $n=1$), (b) $nEFmBCPCP$ ($m=8-11$, $n=3$) and (c) $nEFmBCPCP$ ($m=8-11$, $n=4$). T_c is the temperature of SmA*–SmC* transition.

polarization plotted as a function of temperature for compounds $nEFmBCPCP$ ($m=8-11$, $n=1$), $nEFmBCPCP$ ($m=8-11$, $n=3$) and $nEFmBCPCP$ ($m=8-11$, $n=4$). In general, the P_s values depend on the temperature and exhibit maxima. The first increase

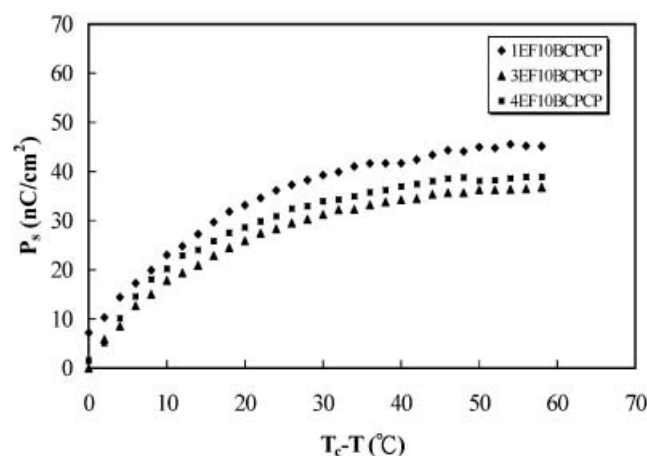


Figure 3. The measured P_s values for compounds $nEFmBCPCP$ ($m=10$, $n=1, 3, 4$), enabling comparison of P_s values with variation of the alkyl chain in the chiral tail of the compounds.

in P_s value at lower temperatures, below T_c , can be attributed to decreased thermal motion at the lower temperature. After the maximum P_s is reached, a gradual decrease in the temperature results in a decreasing P_s , which might be due to crystallization.

The measured P_s values for compounds $nEFmBCPCP$ ($m=10$, $n=1, 3, 4$) are shown in figure 3 to enable comparison of P_s values for different extent of the alkyl chain in the chiral tail of the compounds. The P_s values for three compounds are in the order of $nEFmBCPCP$ ($m=10$, $n=1$) > $nEFmBCPCP$ ($m=10$, $n=4$) > $nEFmBCPCP$ ($m=10$, $n=3$) at any temperature below the Curie point. It appears that compounds with lower alkyl chain length, n , have higher P_s values at any temperature below the Curie point. The maximum P_s values of $nEFmBCPCP$ ($m=8-12$, $n=1, 3, 4$) are somewhat lower than those of the non-substituted compounds, $nEmBCPCP$ ($m=8-12$, $n=1, 3, 4$) due to the transverse dipole effect of the fluoro substituent in the 2-position of the phenyl ring that is closed to the chiral group.

3.4. The optical tilt angle

The optical tilt angles for all compounds in the SmC* phase were measured as a function of temperature on cooling in $2\ \mu\text{m}$ homogeneously aligned cells. Figure 4 shows the optical tilt angle as a function of temperature for the SmC* phase for compounds $nEFmBCPCP$ ($m=8-11$, $n=1$), $nEFmBCPCP$ ($m=8-11$, $n=3$) and $nEFmBCPCP$ ($m=8-11$, $n=4$). In general, the optical tilt angles increase with decreasing temperature and exhibit maxima.

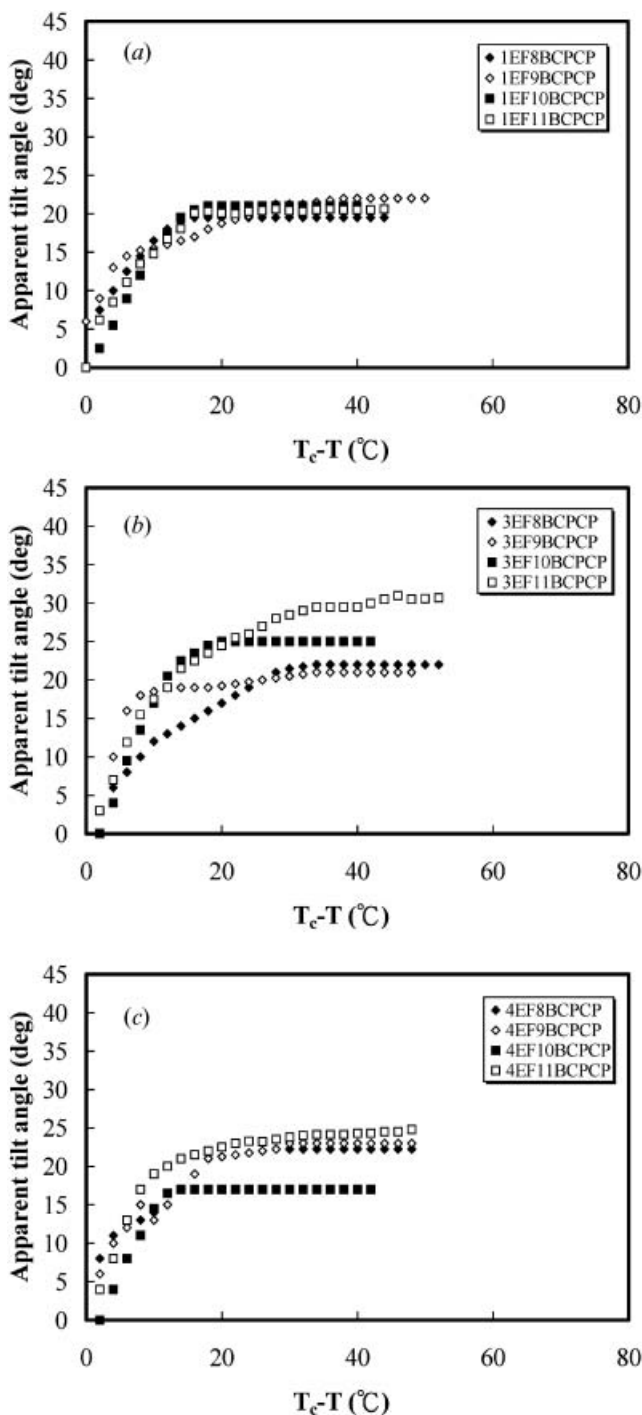


Figure 4. Optical tilt angles as a function of temperature for compounds (a) $nEFmBCPCP$ ($m=8-11$, $n=1$), (b) $nEFmBCPCP$ ($m=8-11$, $n=3$) and (c) $nEFmBCPCP$ ($m=8-11$, $n=4$). T_c is the temperature of SmA^*-SmC^* transition.

The measured optical tilt angles as a function of temperature for compounds $nEFmBCPCP$ ($m=10$, $n=1, 3, 4$) shown in figure 5 enable comparison of optical tilt angles with different extent of the alkyl chain in the

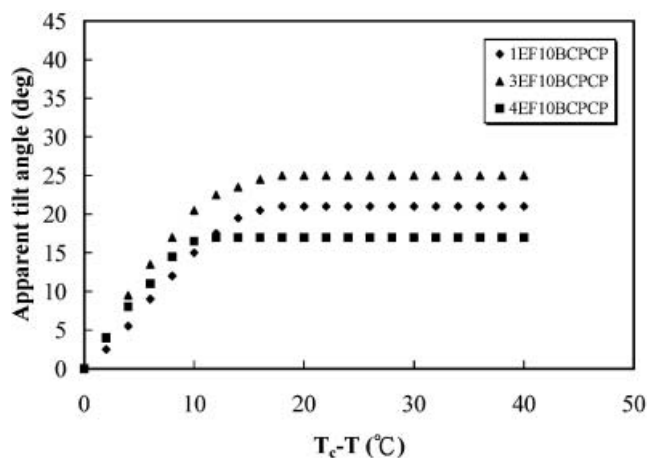


Figure 5. The measured optical tilt angles as a function of temperature for compounds $nEFmBCPCP$ ($m=10$, $n=1, 3, 4$), enabling comparison of optical tilt angles with variation of the alkyl chain in the chiral tail of the compounds. T_c is the temperature of SmA^*-SmC^* transition.

chiral tail of the compounds. The maximum optical angles for the three compounds are in the order of $nEFmBCPCP$ ($m=10$, $n=3$) $>$ $nEFmBCPCP$ ($m=10$, $n=1$) $>$ $nEFmBCPCP$ ($m=10$, $n=4$) at any temperature below the Curie point.

3.5. The electro-optical responses

Electro-optical responses of the SmC^* phase for the compounds were measured under crossed polarizers where the axes of polarizer and analyzer were parallel and perpendicular, respectively, to the smectic layer normal in $2\ \mu\text{m}$ homogeneously aligned cells. In general, the responses are critically dependent on temperature and frequency, and display a normal ferroelectric hysteresis curve. A thresholdless V-shaped switching curve was also obtained in the SmC^* phase of the compounds at certain temperatures and applied frequencies. Typical examples of the V-shaped switching curves in the SmC^* phase of $nEFmBCPCP$ ($m=10$, $n=1, 3, 4$) are shown in figure 6. These results are similar to our previous observations in non-substituted compounds, $nEmBCPCP$ ($m=8-12$, $n=1, 3, 4$).

4. Conclusion

We have demonstrated that fluoro-substituted chiral liquid crystal compounds derived from (*S*)-lactic acid and alkoxyethanols display wide temperature ranges in the ferroelectric SmC^* phase. In particular, in compounds with shorter alkyl chain lengths the ferroelectric SmC^* phase appears at ambient temperature. In contrast, the fluoro substituent at the 2-position of the phenyl ring suppresses the magnitude of the

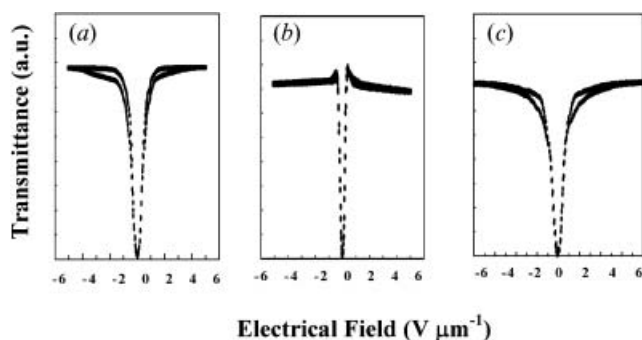


Figure 6. Transmittance versus electrical field obtained in the SmC* phase of compounds (a) *nEFmBCPCP* ($m=9$, $n=1$) at 46°C for a 5 Hz triangular wave, (b) *nEFmBCPCP* ($m=10$, $n=3$) at 80°C for a 1 Hz triangular wave and (c) *nEFmBCPCP* ($m=10$, $n=4$) at 74°C for a 1 Hz triangular wave.

spontaneous polarization. A thresholdless, V-shaped switching property appears in the SmC* phase of the fluoro-substituted compounds, similar to previous observations in non-substituted compounds.

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