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V-shaped switching in ferroelectric liquid crystal mixtures induced by an achiral swallow-tailed material

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V-shaped switching in ferroelectric liquid crystal mixtures induced by an achiral swallow-tailed material

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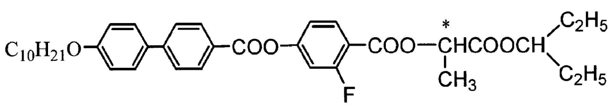
A new ferroelectric liquid crystal, 1-ethylpropyl (*S*)-2-[2-fluoro-4-(4′-decyloxybiphenylcarbonyloxy)benzoyloxy]propanoate, **F**, was synthesized and mixed with an achiral swallow-tailed material, 2-propylpentyl 4-(4′-nonyloxybiphenyl-4-carbonyloxy)benzoate, **P**, for the preparation of binary mixtures for the study. The binary mixtures gave a phase sequence SmA*–SmC*–SmX*. The electro-optic response of the mixtures in the ferroelectric SmC* phase was investigated. V-shaped switching was observed as the amount of the achiral swallow-tailed material became greater than 20 wt %. This result suggests that thresholdless, V-shaped switching in ferroelectric liquid crystal mixtures can be achieved by mixing a ferroelectric liquid crystal with an achiral swallow-tailed compound.

1. Introduction

Thresholdless, V-shaped switching in chiral smectic liquid crystals [1] is of great interest, since this type of material is very promising for display applications. Two antiferroelectric mixtures (Inui and Mitsui mixtures) showing V-shaped switching have been extensively studied [1–8], and some empirical models to account for the switching have been proposed [1, 3, 8, 9]. These two mixtures are composed of molecules with chiral tails possessing the highly polar trifluoromethyl substituent attached to the chiral centre. Consequently, the mixtures possess high polarization (the maximum $\mathbf{P}_s$ for the Inui mixture is 170 nC cm^{-2}) [9].

Our previous results showed that binary mixtures of an achiral swallow-tailed compound with an antiferroelectric liquid crystal (*S*)-MHPOBC displayed V-shaped switching of electro-optical response in antiferroelectric liquid crystal mixtures [10]. Moreover, the V-shaped switching appeared at a relatively high amount of achiral swallowed-tailed compound, suggesting that this optical switching can take place at a relatively low polarization. In this study, therefore, we decided to investigate binary mixtures of a ferroelectric liquid crystal (FLC) and an achiral swallow-tailed compound, in order to explore whether V-shaped switching in electro-optical response could be induced. A new FLC, 1-ethylpropyl (*S*)-2-[2-fluoro-4-(4′-decyloxybiphenylcarbonyloxybenzoyl)-propanoate, **F**, was designed and synthesized from L-lactic acid for mixing with the achiral swallow-tailed compound, 2-propylpentyl 4-(4′-nonyloxybiphenyl-4-carbonyloxy)-benzoate, **P**, for the preparation of FLC mixtures. The structural formulae for these compounds are shown below:

F



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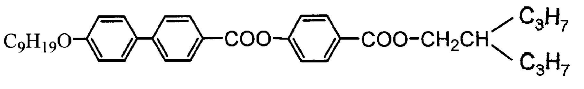

2. Experimental

2.1. *Characterization of the materials*

The chemical structures for intermediates and target materials were characterized by NMR spectroscopy using a Jeol EX-400 FTNMR spectrometer. The purity was checked by thin layer chromatography and further confirmed by elemental analysis using a Perkin-Elmer 2400 spectrometer. Transition temperatures and enthalpies of compounds were determined by differential scanning calorimetry using a Perkin-Elmer DSC7 calorimeter at running rates of 1 to 20°C min^{-1}. Mesophases were identified principally by microscopic texture of the materials sandwiched between two glass plates under a polarizing optical microscope using a Nikon Microphot-FXA in conjunction with Instec HS1 hot stages.

The physical properties of ferroelectric phases were measured in homogeneously aligned cells, purchased from E. H. C. Co., Japan. The spontaneous polarization $\mathbf{P}_s$ was measured by a triangular wave method [11]. A triangular wave was applied to the sample from a Yogaw AG1200 arbitrary wave-form generator; the induced

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current was displayed by measuring the voltage across a wire-wound resistor using a Hewlet-Packard HP54502A digital storage oscilloscope.

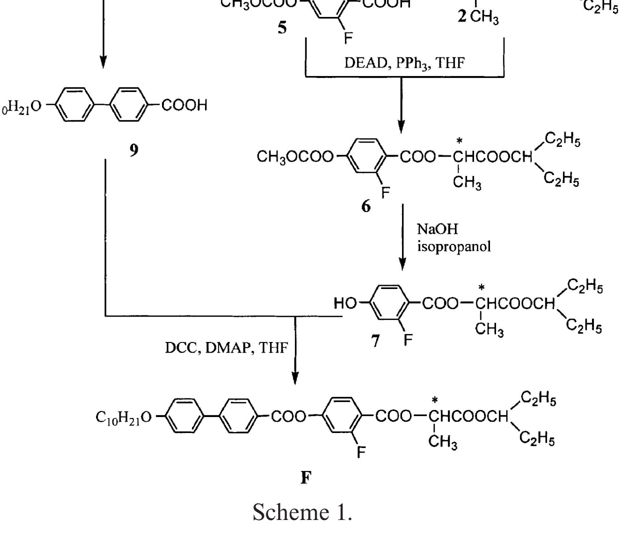
The measurement of optical transmittance versus applied electric field used a He-Ne laser (5 mW, 632.8 nm) as a probe beam [12, 13]. The optical transmittance of the probe beam passing though the cell between crossed polarizers, with axes parallel and perpendicular to the smectic layer normal, was detected by a photodiode; signals were detected using HP54502A digital oscilloscope. The voltage applied to the cell was produced by an arbitrary wave-form generator (AG1200) and amplified by a home-made power preamplifier.

2.2. *Preparation of materials*

Achiral swallow-tailed liquid crystal **P** was synthesized as described before [10]. Ferroelectric liquid crystal **F** was prepared according to the synthetic procedures described in the scheme 1. The detailed procedures are described below.

2.2.1. *2-Propylpentyl 4-(4′-nonyloxybiphenyl-4-carbonyloxy)benzoate, **P***

Yield 80%. Elemental analysis for $C_{37}H_{48}O_5$: calc. C 77.62, H 8.39; found C 77.38, H 8.36%. ^{1}H NMR ($CDCl_3$): δ (ppm) 0.87–1.57 (m, 32H, $RC\mathbf{H}_2C\mathbf{H}_3$), 1.77–1.80 (m, 1H, $OCH_2\mathbf{CH}$), 4.00–4.02 (t, 2H, $ArO\mathbf{CH}_2$), 4.18–4.20 (d, 2H, $COO\mathbf{CH}_2$), 6.98–8.23 (m, 12H, ArH).



F

Scheme 1.

2.2.2. *2-Ethylpropyl (S)-lactate, **2*** [14]

(L)-Lactic acid (12 g, 0.13 mmol) and 2-ethylpropanol (8.8 g, 0.1 mmol) were dissolved in dry benzene (30 ml) and heated under reflux overnight using a Dean–Stark trap to remove water. After removing the benzene solvent, the residue was distilled under vacuum to yield 30% of compound **2**, which was collected at 85°C 30 mm Hg. ^{1}H NMR (400 MHz, $CDCl_3$): δ (ppm) 0.76–0.84 (m, 6**H**), 1.31–1.36 (d, 3**H**), 1.38–1.55 (m, 4**H**), 3.84 (s, 1**H**), 4.21–4.15 (m, 1**H**), 4.76–4.70 (m, 1**H**).

2.2.3. *2-Fluoro-4-hydroxybenzoic acid, **4*** [15]

4-Cyano-2-fluorophenol (3 g, 22 mmol) was hydrolyzed by heating under refluxing with sodium hydroxide (3.12 g) in water (30 ml) for 2 h. When cool, the mixture was acidified with concentrated hydrochloric acid, and the solution extracted with diethyl ether was removed and the product crystallized from ethyl acetate/hexane to afford a white crystalline solid, m.p. 200–202°C; yield 91.7%. ^{1}H NMR (400 MHz, DMSO): δ (ppm) 10.90–10.67 (s, 2H, –O**H**), 7.46–6.56 (m, 3H, –Ar**H**).

2.2.4. *2-Fluoro-4-(methoxycarbonyloxy)benzoic acid, **5*** [16]

To a solution of sodium hydroxide (2.9 g, 0.073 mmol) in water (40 ml) maintained at 0°C, compound **4** (3 g, 20 mmol) was added with vigorous stirring. Methyl chloroformate (4.4 g, 47 mmol) was then added slowly to the resulting suspension which was maintained at 0–5°C. The resulting slurry was stirred for a further 4 h and brought to PH = 5 by addition of conc. hydrochloric acid and water (1 : 1). The voluminous precipitate was filtered off and recrystallized from ethanol to give a white solid; yield 75% of compound **5**, m.p. 178–180°C. ^{1}H NMR (400 MHz, $CDCl_3$): δ (ppm) 0.91–0.81 (m, 6H, $-(CH_2\mathbf{CH}_3)_2$), 1.50–1.47 (d, 3H, $-CH\mathbf{CH}_3$), 1.62–1.58 (m, 12H, $-CH(\mathbf{CH}_2\mathbf{CH}_2\mathbf{CH}_2CH_3)_2$), 3.90 (s, 3H, $-COO\mathbf{CH}_3$), 4.84–4.80 (q, 1H, $-COO\mathbf{CH}(CH_2)_2-$), 5.31–5.28 (m 1H, $-COO\mathbf{CH}CH_3-$), Ar**H**, 8.10–7.24 (d, 4H).

2.2.5. *1-Ethylpropyl (S)-2-[2-fluoro-4-(methoxycarbonyloxy)benzoyloxy]propanoate, **6*** [17]

A solution of diethyl azodicarboxylate (DEAD, 1.72 g, 9.9 mmol) and compound **5** (2.12 g, 9.9 mmol) in 10 ml anhydrous THF was added dropwise to a solution of triphenylphosphine (Ph_3P, 2.6 g, 9.9 mmol) and compound **2** (1.3 g, 8.3 mmol) in anhydrous THF (10 ml) at room temperature with vigorous stirring. The reaction soon started. After standing overnight at room temperature, triphenylphosphine oxide was removed by filtration; THF was then removed under vacuum. After the work-up procedure, the product was isolated by column chromatography over silica gel (70–230 mesh) using ethyl acetate/hexane (1/11) as eluent, to give compound **6** as a colourless liquid; yield 77.9%. ^{1}H NMR (400 MHz, $\mathrm{CDCl_3}$): δ (ppm) 8.19–7.25 (m, 3H, $-\mathrm{Ar}\mathbf{H}$), 5.34–5.26 (q, 1H, $-\mathrm{COOC}\mathbf{H}\mathrm{CH_3}-$), 4.85–4.83 (m, 1H, $-\mathrm{COOC}\mathbf{H}(\mathrm{CH_2})_2-$), 3.95 (s, 3H, $-\mathrm{COOC}\mathbf{H_3}$), 1.69–1.62 (m, 4H, $-\mathrm{CH}(\mathbf{CH_2}\mathrm{CH_3})_2$), 1.58–1.57 (d, 3H, $-\mathrm{CH}\mathbf{CH_3}-$), 0.94–0.85 (m, 6H, $-(\mathrm{CH_2}\mathbf{CH_3})_2$).

2.2.6. *1-Ethylpropyl (S)-2-[(2-fluoro-4-hydroxyphenylcarbonyloxy)propanoate, 7*

Compound **6** (2 g, 5.3 mmol) was stirred in a mixture of isopropanol (100 ml) and ammonia (28%, 40 ml) at room temperature for 50 min (TLC analysis revealed a complete reaction) and then poured into water (50 ml) with stirring. The product was extracted with dichloromethane (3 × 50 ml). The combined extracts were washed with brine (50 ml), dried ($\mathrm{MgSO_4}$), filtered and evaporated to give a colourless oil. The oil was purified by column chromatography over silica gel (70–230 mesh) using dichloromethane; it was then dried *in vacuo* to give 80.6% yield of compound **7**. ^{1}H NMR (400 MHz, $\mathrm{CDCl_3}$): δ (ppm) 8.12–6.91 (m, 3H, $-\mathrm{Ar}\mathbf{H}$), 6.85 (s, 1**H**), 5.25–5.20 (q, 1H, $-\mathrm{COOC}\mathbf{H}\mathrm{CH_3}-$), 4.82–4.77 (m, 1H, $-\mathrm{COOC}\mathbf{H}(\mathrm{CH_2})_2-$), 1.68–1.60 (m, 4H, $-\mathrm{CH}(\mathbf{CH_2}\mathrm{CH_3})_2$), 1.57–1.55 (d, 3H, $-\mathrm{CH}\mathbf{CH_3}-$), 0.91–0.83 (m, 6H, $-(\mathrm{CH_2}\mathbf{CH_3})_2$).

2.2.7. *4-(4′-Decyloxyphenyl)benzoic acid, 9*

A solution of 4′-hydroxybiphenyl-4-carboxylic acid (25 mmol) in ethanol (200 ml) was treated by dropwise addition of a solution of potassium hydroxide (2.8 g, 50 mmol) and potassium iodide (KI) (0.6 g, 3.6 mmol) in water (50 ml). The mixture was heated under reflux for 1 h. 1-Bromodecane (75 mmol) was added to the mixture and reflux continued for a further 12 h. Aqueous potassium hydroxide (100 ml, 10%) was added, and after a further 2 h reflux the mixture was acidified with 5% aqueous HCl and filtered. The crude product was washed with cold water and recrystallized from glacial acetic acid and absolute ethanol; a 60% yield of compound **9** was obtained.

2.2.8. *1-Ethylpropyl (S)-2-[2-fluoro-4-(4′-decyloxybiphenylcarbonyloxy)benzoyloxy]propanoate, F*

A mixture of compound **9** (1 g, 2.8 mmol), compound **7** (0.8 g, 3.1 mmol), *N*,*N*′-dicyclohexylcarbodiimide (0.35 g, 2.8 mmol), 4-dimethylaminopyridine (0.06 g, 0.28 mmol) and dry THF (15 ml) was stirred at room temperature for three days. The precipitates were filtered off and the filtrate washed with 5% acetic acid solution (3 × 50 ml), 5% saturated aqueous sodium hydrogen carbonate (3 × 50 ml) and water (3 × 50 ml); it was dried ($\mathrm{MgSO_4}$) and concentrated in vacuum. The residue was purified by column chromatography over silica gel (70–230 mesh) using ethyl acetate/hexane (1/1) as eluent. After purification by crystallization from absolute ethanol a 40% yield of final product was obtained. Elemental analysis: calc. for $\mathrm{C_{36}H_{43}O_7F}$, C 71.90, H 7.46; found C 71.89, H 7.51%. ^{1}H NMR (400 MHz, $\mathrm{CDCl_3}$): δ (ppm) 8.24–7.00 (m, 12H, $\mathrm{Ar}\mathbf{H}$) 5.36–5.32 (q, 1H, $\mathrm{COOC}\mathbf{H}\mathrm{CH_3COO}$) 4.87–4.85 (m, 1H, $\mathrm{COOC}\mathbf{H}$) 4.04–4.01 (t, 2H, $\mathrm{ArOC}\mathbf{H_2}$) 1.84–0.88 (m, 28H, $\mathrm{R}\mathbf{CH_2}\mathbf{CH_3}$).

2.2.9. *Preparation of binary ferroelectric liquid crystal mixtures, **P**/**F***

The mixtures were prepared by weight % and mixed thoroughly with the addition of anhydrous dichloromethane. The dichloromethane was then evaporated and the mixtures further dried under vacuum. The binary mixtures obtained are described, for example, as follows: 20**P**/80**F** refers to the mixture of 20% **P** and 80% **F**, while 80**P**/20**F** refers to the mixture of 80% **P** and 20% **F**.

3. Results and discussion

Observation of mesophases and their transition temperatures for compounds **F** and **P**, and for ferroelectric mixtures, was carried out by optical microscopy texture observation and DSC calorimetry. SmA* phases appeared as focal-conic textures, whereas SmC* phases displayed a broken focal-conic texture. Detailed phase transition data are shown in the table. Melting points and I–SmA* and SmA*–SmC* transition temperatures decrease as the amount of chiral material **F** increases.

Physical properties of the compounds were measured in 5 μm homogeneous cells. In order to obtain a unit domain of surface-stabilized geometry for the ferroelectric compound **F** and mixtures in the cells, a variable frequency a.c. electric field ($f = 0.5$ Hz–2.5 kHz, $E = 10$–20 $\mathrm{V_{p\text{-}p}}$ wave form: triangular wave) was applied to the cells during cooling.

Detailed results of the switching current behaviour in this series are presented in figure 1. The switching currents measured for compound **F** and each mixture display only one peak, supporting the assignment of a ferroelectric SmC* phase. The strength of these current peaks slightly decrease with increasing amount of **P**, but the relative position of the peaks is almost unchanged. Figure 2 shows some representative results of spontaneous polarization $\mathbf{P}_\mathrm{s}$ as a function of temperature measured for **F** and binary mixtures. The maximum $\mathbf{P}_\mathrm{s}$ value for compound **F** is approximate 82 nC cm^{-1}. The maximum $\mathbf{P}_\mathrm{s}$ value decreases as the amount of achiral material **P** increases.

Table 1. The transition temperatures (°C) of achiral swallow-tailed and binary mixtures.

Mixture	I		SmA*		SmCalt/SmC*		SmX*		Cr	m.p.[b]
100**P**/0**F**	•	139.4	•	119.2	•	—	—	61.6	•	97.6
80**P**/20**F**	•	133.8	•	113.5	•	33.2	•	7.3	•	87.6
60**P**/40**F**	•	129.2	•	108.7	•	7.5	•	−2.1	•	76.3
40**P**/60**F**	•	124.4	•	103.7	•	16.1	•	0.7	•	41.0
20**P**/80**F**	•	117.9	•	97.4	•	25.4	•	10.5	—	50.1
0**P**/100**F**	•	113.1	•	95.6	•	—	—	14.3	•	34.7

[a] Recorded by DSC thermograms at cooling rates of 5°C min^{-1}.
[b] Melting point taken from DSC thermograms recorded at heating rates of 5°C min^{-1}.

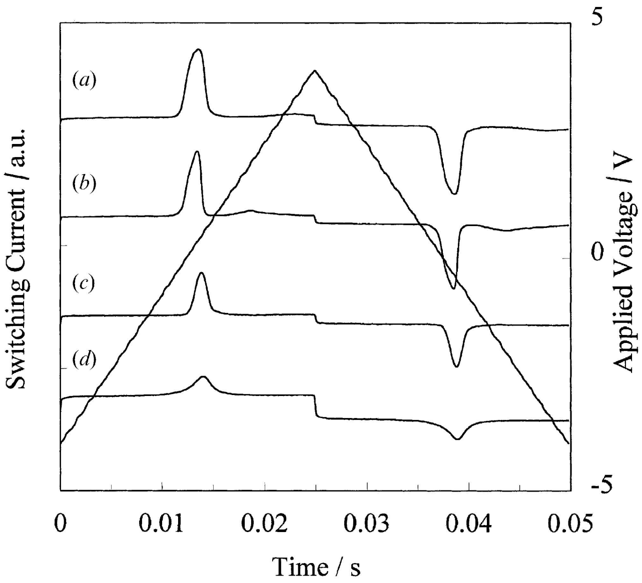


Figure 1. Switching behaviour in the SmC* phase of (*a*) 100**F**, (*b*) 80**F**/20**P**, (*c*) 40**F**/60**P**, (*d*) 20**F**/80**P** measured at 30°C below curie point in homogeneously aligned cells of 5 μm thickness.

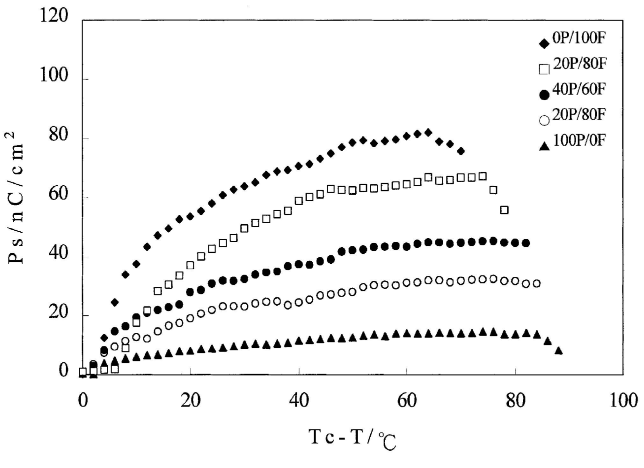


Figure 2. Spontaneous polarization plotted as a function of temperature for binary mixtures of compounds **F** and **P**.

Electro-optical responses were obtained under crossed polarizers where the axes of polarizer and analyser were parallel or perpendicular to the smectic layer normal in 5 μm homogeneously aligned cells. Figure 3 illustrates the response of the transmittance versus electric field on applying a field of triangular waveform, measured in the

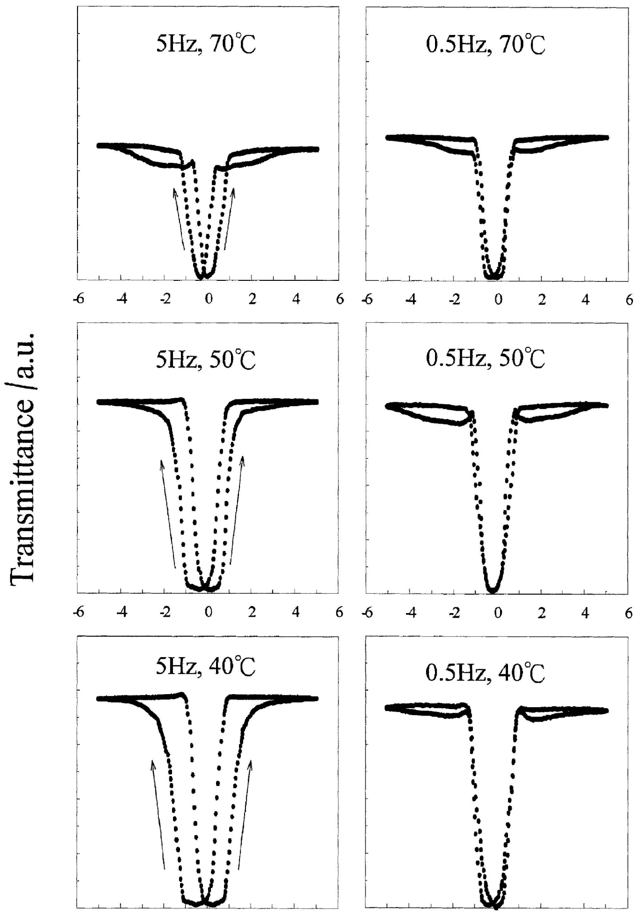


Figure 3. Electro-optical response of transmittance versus electric field for compound **F** in the SmC* phase at several temperatures and frequencies of applied triangular wave.

SmC* phase for compound **F**. The responses critically depend on temperature and frequency. As the temperature decreases, the maximum transmittance values increase due to the increase of the tilt angle [8]. At 5 Hz applied frequency, the characteristic ferroelectric hysteresis loop appears in the temperature region of the SmC* phase. However, as the applied frequency is lowered to 0.5 Hz, hysteresis-free, U-shaped switching is seen, as shown in the switching response at 0.5 Hz and 50°C. The optical responses in the SmC* phase essentially depend on the applied frequency.

Figure 4 shows the electro-optic responses obtained for two mixtures: 20**P**/80**F** and 40**P**/60**F**, at 0.5 Hz applied frequency and various temperatures in 5 μm homogeneous cells. In the mixture 20**P**/80**F**, the hysteresis loop appears different from that observed in a normal ferroelectric SmC* phase. As can be seen from the hysteresis loop at 80°C, the optical response appears W-shaped near the minimum (arrow 1), while the rest of the hysteresis loop retains its ferroelectric nature (arrow 2). However, as the temperature is cooled to 60°C, a typical W-shaped switching appears. Further cooling to 40°C gives a thresholdless, V-shaped switching. On increasing the amount of achiral swallow-tailed compound **P**, as in the mixture 40**P**/60**F**, V-shaped switching is observed at 80 and 60°C in SmC* phase. A W-shaped feature occurs at 40°C, which can alter to V-shaped switching on changing the applied frequency or the cell thickness. The mixtures containing higher amount of **P**, gave similar results to those of 40**P**/60**F**. These optical phenomena suggest that V-shaped switching in ferroelectric mixtures can be achieved by mixing a FLC with an achiral swallow-tailed material. The increasing amount of **P** in the ferroelectric mixtures results in a decrease of polarization, also implying that low polarization of the ferroelectric mixtures leads to thresholdless, V-shaped switching. This phenomenon is in agreement with our previous observation of the appearance of V-shaped switching in an antiferroelectric liquid crystal (*S*)-EP10PBNP with low polarization (maximum $\mathbf{P}_s = 30\,\mathrm{nC\,cm^{-2}}$) [10, 18], and in antiferroelectric mixtures of (*S*)-MHPOBC with an achiral swallow-tailed material [10].

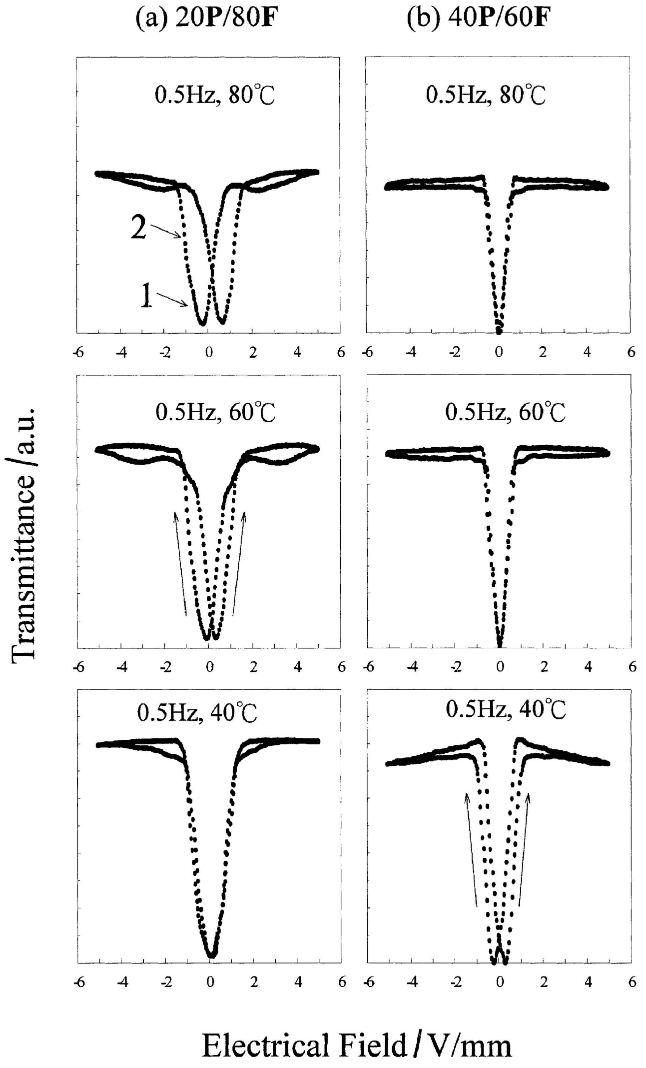


Figure 4. Electro-optical response of transmittance versus electric field for the mixtures (*a*) 20**P**/80**F** and (*b*) 40**P**/60**F** in the SmC* phase at several temperatures and frequencies of applied triangular wave.

In general, the electro-optical response of liquid crystals strongly depends on the interaction between the liquid crystal molecules and the surface of the substrate in a homogeneously aligned cell. The occurrence of thresholdless, V-shaped switching has been suggested to be a result of the substrate interface which induces tilting randomization due to weak inter-layer correlation [2]. In our case, beside the effect of substrate interface, the mixing of two structurally different compounds might play an important role in the emergence of V-shaped switching in ferroelectric mixtures. However, the reasons why V-shaped switching in the mixtures is affected by addition of an achiral swallow-tailed compound, or by the reducing polarization of the mixture (or both), are not clear at present.

4. Conclusion

Thresholdless, V-shaped switching in ferroelectric mixtures was induced by the addition of an achiral swallow-tailed compound; whether this is due to the lowering of polarization of the mixtures or to the increasing content of achiral swallow-tailed compound, is not yet certain. However, our results strongly suggested that thresholdless, V-shaped switching could be achieved in FLCs by mixing an achiral swallow-tailed material with the FLCs.

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