



Molecular Crystals and Liquid Crystals

Publication details, including instructions for authors and subscription information:

<http://www.tandfonline.com/loi/gmcl20>

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Version of record first published: 22 Sep 2010

To cite this article: Jae-Cheol Park, Dong-Ju Park, Kyung-Chul Son & Yong-Bae Kim (2007): The Properties of Homeotropic Alignment Materials as the Side Chain Molecular Structure in Polyimides, *Molecular Crystals and Liquid Crystals*, 479:1, 191/ [1229]-203/[1241]

To link to this article: <http://dx.doi.org/10.1080/15421400701734056>

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The Properties of Homeotropic Alignment Materials as the Side Chain Molecular Structure in Polyimides

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LCD (Liquid Crystal Display) screens are today's most commonly used displays and are available in a wide range of shapes, size, levels of quality, and models. VA-LCD is good to TV applications from among other LCD modes, owing to wide viewing angle, high contrast ratio and fast switching time. To apply for VA-LCDs, we have synthesized novel homeotropic alignment materials, which all generated high pretilt angles ($>88^\circ$) with and without rubbing. They showed good homeotropic alignment state too. In addition, response time was very fast. In this paper, we discussed the synthesis and alignment properties of new homeotropic alignment materials.

Keywords: homeotropic alignment materials; pretilt angle; response time

INTRODUCTION

Two kinds of liquid-crystalline shape diamines, 2,3-Difluoro-4-(4-propyl-cyclohexyl)-biphenyl-4'-propyl 3,5-diamino benzoate (negative liquid crystalline shape diamine, SP type) and 4'-(4-Pentylcyclohexyl)-biphenyl-3,5-diamine (positive liquid crystalline shape diamine, R5CPP type), were polymerized with 4,4-oxydianilin (ODA) and

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three aromatic dianhydrides, 3,3',4,4'-benzophenone tetracarboxylic dianhydride (BTDA), 4,4'-Oxydiphthalic anhydride (ODPA) and 3,3',4,4'-Biphenyltetracarboxylic dianhydride (BPDA), in *N*-methyl-2-pyrrolidinone (NMP), and the resulting poly(amic acid)s were thermally cyclodehydrated to polyimides.

These polyimides possess high pretilt angle and high electro-optical properties. They can generate greater pretilt angle than 88°. They showed good homeotropic alignment state too. In addition, response time of SP type polyimide was very fast.

So these negative liquid crystalline shape side chain polyimides (novel homeotropic alignment materials), which we synthesized, are possible to use for VA-LCD application.

EXPERIMENTAL

Materials

Diamine monomer, 2,3-Difluoro-4-(4-propyl-cyclohexyl)-biphenyl-4'-propyl 3,5-diamino benzoate was synthesized as following the Figure 2 and 4'-(4-Pentylcyclohexyl)biphenyl-3,5-diamine was synthesized as following the Figure 3. Poly(amic acid) precursors [1] were prepared by copolymerization from mixing dianhydride, ODA(4,4'-oxydianiline) and synthesized diamine with side chain. The three dianhydride monomers, 3,3,4,4-Benzophenonetetracarboxylic dianhydride (BTDA), 4,4'-Oxydiphthalic anhydride (ODPA), 3,3',4,4'-Biphenyltetracarboxylic dianhydride (BPDA) were purchased from Aldrich and purified by recrystallization from THF and MEK.

Measurement

The chemical structures of products were confirmed by ¹H NMR spectroscopy (Bruker Advance 400 MHz Spectroscope) and GC/MS spectroscope (Hewlett Packard 5972 MSD). Voltage holding ratio (VHR) was measured with 6254 Instruments from TOYO Inc.Co. Pretilt angles were measured by using crystal rotation method [2] with the self set up optic system. Figure 1. indicates the principle block diagram of the measuring system of pretilt angle. Response time of LC was measured with the self set up system using a waveform generator and oscilloscope. The condition of alignment of LC was observed by the polarized microscope of Olympus Co. BX-51.

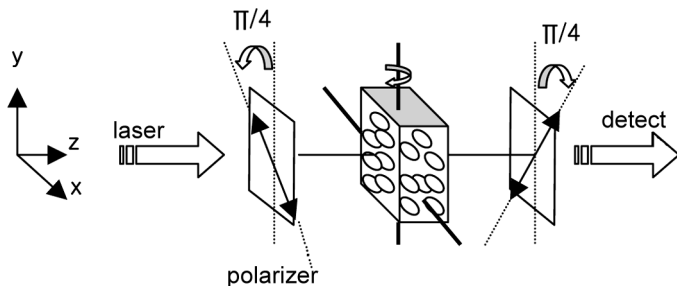


FIGURE 1 Configuration of polarizer and cell.

SYNTHESIS

Synthesis of Diamines

1-(2,3-difluoro-phenyl)-4-propyl-cyclohexanol (3)

To the 1,3-difluoro-benzene in super dried THF was added 2.5 M Butyl lithium at -78°C , stirred for 3 hr at -78°C and then added the 4-propylcyclohexanone in super dried THF at -78°C . The temperature of mixture was raised until -10° , added 5% hydrochloric acid in water and then extracted with ether, washed with 5% sodium chloride and water. The organic layer was dried over anhydrous magnesium sulfate, evaporated *in vacuo*, chromatographed on silica gel to give compound (3).

Mass: 254(M^+), 236, 207, 179, 169, 156, 141, 127, 113, 55

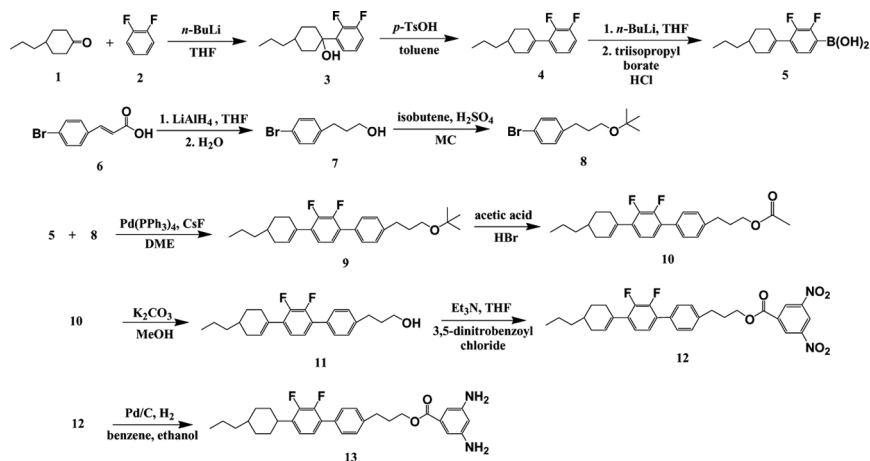


FIGURE 2 Synthetic route of SP diamine.

1,2-difluoro-3-(4-propyl-cyclohex-1-enyl)-benzene (4)

To the solution (3) in toluene was added catalytic amount of *p*-toluenesulfonic acid, refluxed for 4 hr and then extracted with ether, washed with 5% sodium bicarbonate and water. The organic layer was dried over anhydrous magnesium sulfate, evaporated *in vacuo*, chromatographed on silica gel to give compound (4).

Mass: 236(M⁺) 193, 179, 127, 81, 68, 55

2,3-difluoro-4-(4-propyl-cyclohex-1-enyl)-benzyl boronic acid (5)

To the solution (4) in super dried tetrahydrofuran was added 2.5 M Butyl lithium at -80°C , stirred for 3 hr and then added triisopropyl borate at -78°C . The temperature of mixture was raised until room temperature, stirred continuously. The mixture was added 10% hydrochloric acid in water, stirred for 1 hr, and then extracted ether.

The organic layer was dried over anhydrous magnesium sulfate, evaporated *in vacuo*, chromatographed on silica gel to give compound (5).

^1H NMR (400 MHz, CDCl_3): δ 7.1 (2H, d), 5.87 (1H, s), 2.57 (4H, s), 2.24 (2H, m), 1.87 (2H, m), 1.65 (1H, m), 1.55 (2H, m), 1.39 (2H, m), 1.32 (2H, m), 0.94 (3H, t)

3-(4-Bromo-phenyl)-propan-1-ol (7)

To the super dried tetrahydrofuran was added LiAlH_4 , stirred for 30 min and then added 4-Bromocinnamic acid in super dried THF. The mixture was refluxed for 2 hr and cooled until room temperature. The mixture was added water, stirred for 1 hr. The mixture was added 10% hydrochloric acid in water, stirred for 1 hr, and then extracted ether. The organic layer was dried over anhydrous magnesium sulfate, evaporated *in vacuo*, chromatographed on silica gel to give compound (7) [3].

Mass: 214(M⁺), 196, 169, 117, 104, 91, 77, 63, 51

1-Bromo-4-(3-tert-butoxy-propyl)-benzene (8)

MC was cooled until -50°C , 2-methyl propene was dissolved in MC. 3-(4-Bromo-phenyl)-propan-1-ol in MC was added, and then H_2SO_4 1 drop was added. The mixture was stirred for 10 hr at 20°C , and then extracted ether. The organic layer was dried over anhydrous magnesium sulfate, evaporated *in vacuo*, chromatographed on silica gel to give compound (8) [3].

Mass: 270(M⁺), 214, 196, 169, 117, 104, 90, 77, 57

4'-(3-tert-Butoxy-propyl)-2,3-difluoro-4-(4-propyl-cyclohex-1-enyl)-biphenyl (9)

1-Bromo-4-(3-tert-butoxy-propyl)-benzene and 1,2-Difluoro-3-(4-propyl-cyclohex-1-enyl)-benzene boronic acid were dissolved in DME. The mixture was added CsF and Pd(PPh₃)₄ and then refluxed for 3 hr, and then extracted ether. The organic layer was dried over anhydrous magnesium sulfate, evaporated *in vacuo*, recrystallized with EA and ethanol to give compound (9) [4,5].

Mass: 426(M⁺), 370, 339, 325, 255, 229, 134, 81, 57

2,3-Difluoro-4-(4-propyl-cyclohex-1-enyl)-biphenyl-4'-propyl acetate (10)

4'-(3-tert-Butoxy-propyl)-2,3-difluoro-4-(4-propyl-cyclohex-1-enyl)-biphenyl was dissolved in acetic acid and HBr, stirred for 1 hr at 50°C. The mixture was extracted ether. The organic layer was dried over anhydrous magnesium sulfate, evaporated *in vacuo*, chromatographed on silica gel to give compound (10) [6,7].

Mass: 412(M⁺), 352, 282, 256, 243, 201, 55

2,3-Difluoro-4-(4-propyl-cyclohex-1-enyl)-biphenyl-4'-propyl alcohol (11)

2,3-Difluoro-4-(4-propyl-cyclohex-1-enyl)-biphenyl-4'-propyl acetate was dissolved in methanol. The mixture was added 3.5 M K₂CO₃ and refluxed for 3 hr. The mixture was extracted ether. The organic layer was dried over anhydrous magnesium sulfate, evaporated *in vacuo*, chromatographed on silica gel to give compound (11) [6,7].

Mass : 370(M⁺), 352, 313, 274, 255, 201, 91, 55

¹H NMR (400 MHz, CDCl₃): δ 7.47 (2H, d), 7.28 (2H, d), 7.10 (1H, m), 7.03 (1H, m), 6.01 (1H, s), 3.71 (2H, t), 2.76 (2H, t), 2.41 (1H, m), 1.90 (4H, m), 1.62 (2H, m), 1.35 (7H, m), 0.93 (3H, t)

2,3-Difluoro-4-(4-propyl-cyclohex-1-enyl)-biphenyl-4'-propyl 3,5-dinitro benzoate (12)

2,3-Difluoro-4-(4-propyl-cyclohex-1-enyl)-biphenyl-4'-propyl alcohol in super dried THF was added triethyl amine. The mixture was added 3,5-dinitro benzoyl chloride in super dried THF, refluxed for 3 hr and then extracted ether. The organic layer was dried over anhydrous magnesium sulfate, evaporated *in vacuo*, recrystallized with EA and ethanol to give compound (12) [8].

¹H NMR (400 MHz, CDCl₃): δ 9.19 (1H, t), 9.08 (2H, d), 7.46 (2H, d), 7.30 (2H, d), 7.03 (2H, m), 6.02 (1H, s), 4.53 (2H, t), 2.86 (2H, t), 2.36

(2H, m), 2.24 (2H, m), 1.87 (2H, m), 1.65 (1H, m), 1.55 (2H, m), 1.39 (2H, m), 1.32 (2H, m), 0.94 (3H, t)

2,3-Difluoro-4-(4-propyl-cyclohexyl)-biphenyl-4'-propyl 3,5-diamino benzoate (13)

2,3-Difluoro-4-(4-propyl-cyclohex-1-enyl)-biphenyl-4'-propyl 3,5-dinitro benzoate was dissolved in benzene and ethanol, and palladium 10 wt.% on activated carbon was added. The mixture was maintained for 12 hr under 4.5 kg/cm² pressure of H₂. The Pd/C was filtered off and residue was evaporated *in vacuo*. The product was recrystallized with ethanol to give compound (13).

¹H NMR (400 MHz, CDCl₃): δ 7.46 (2H, d), 7.28 (2H, d), 7.11 (1H, m), 7.02 (1H, m), 6.77 (2H, d), 6.19 (1H, m), 4.31 (2H, t), 3.68 (4H, s), 2.86 (1H, m), 2.81 (2H, t), 2.10 (2H, m), 1.89 (4H, m), 1.50 (1H, m), 1.35 (4H, m), 1.24 (2H, m), 1.09 (2H, m), 0.91 (3H, m)

4'-(4-Pentylcyclohexyl) biphenyl-3, 5-diamine (19) [9]

We synthesized diamine having side chain of 4'-(4-Pentylcyclohexyl) biphenyl-3, 5-diamine. The synthetic route was shown in Figure 3. 1-Bromo-3, 5- dinitrobenzene was synthesized by bromination of 1, 3-dinitrobenzene [10] and reacted with 4-(pentylcyclohexyl) benzyl boric acid through Suzuki reaction [11]. Diamine which 4'-(4-Pentylcyclohexyl) biphenyl-3, 5-diamine (R5CPP) was prepared by reduction of their coupling compound.

Synthesis of Polyamic Acid

Poly(amic acid)s were prepared from dianhydrides, ODA and the functional diamines. A typical polymerization procedure was shown in Figure 4. Dianhydride of three types, ODA and the functional diamine (mole ratio of 3:2:1) are dissolved in 1-methyl-2-pyrrolidinone (NMP) to 18 wt.% in a three-necked round-bottom flask with a mechanical stirrer in a nitrogen atmosphere. The reactants were stirred for 4 hrs at 0~5°C.

The prepared poly(amic acid)s were diluted to 6 wt.% in N-methyl-2-pyrrolidone (NMP) and 2-butoxyethanol. These solutions were coated on ITO glass substrates using spin coater and dried at 90°C for 5 min, and then cured at 220°C for 1 hr. Poly(amic acid)s resulting from co-polymerization is then converted to polyimide by thermal imidisation. The imidization ratio was measured by FT-IR.

Table 1 represents the names of polyimide synthesized, respectively.

TABLE 1 The Name of Polyimides

| Polyimide | Diamine with side chain | Diamine without side chain | Dianhydride |
|-----------|-------------------------|----------------------------|-------------|
| R5CPP-BT | | | |
| R5CPP-OD | | | |
| R5CPP-BP | | | |
| SP-BT | | | |
| SP-OD | | | |
| SP-BP | | | |

RESULTS AND DISCUSSION

One of the important parameters of liquid crystal alignment is the pretilt angle which influences the viewing angle and electro-optic properties of the LCDs. [12] To measure their pretilt angles, a test cell was located between a polarizer and an analyzer, and rotated from -50°C to $+50^{\circ}\text{C}$ as change of incident angle. We can calculate the

value of pretilt angle from symmetry angle at the transmittance curve by following Eq. (1).

$$\theta = \sin^{-1}\left(\frac{\sin \psi_x}{n_e + n_o}\right) \tag{1}$$

where, θ is pretilt angle and ψ_x is the symmetry angle. And n_e is the extraordinary refractive index, and n_o is the ordinary refractive index.

High pretilt angles were generated by the synthesis alignment materials as shown in Table 2.

The alignment characteristic was observed by BX-51 microscope with cross polarizer. The alignment textures were exhibited in Table 3. The polyimide films were rubbed by a rubbing machine under the following conditions: the translation speed of the sample was 13.3 mms^{-1} ; the rotation speed of the roller was 500 rpm; the pile impression depth P_{rub} was 0.1 mm. We used the nematic liquid crystal having negative dielectric anisotropy (MERCK, MJ961213).













The microscopic textures in off/on state for each cell represented as shown in Figure 3. In off state, no defect was observed for cells coated the synthesized polyimides. They represented white state without defect at the field-on state (a square-wave AC voltage of 5 V). A homoeotropic alignment of liquid crystal (LC) molecules obtains over wide areas on the polyimide.

Transmittance is one of the characteristic parameters decides active voltage of module. We measured V-T characteristics using electro-optic system after rubbing. Applied voltage was from 0 V to 10 V. Table 4. shows the threshold voltage and the transmittance versus applied voltage for the polyimides. SP type polyimides were showed high threshold and saturation voltage. The threshold voltage (V_{th}) usually has some connection with frequency, dielectric dispersion of LC materials, alignment [13]. We used same LC materials under the

TABLE 2 Pretilt Angle of Polyimides

| <div><div></div><div>Rubbing</div></div> <div>Polyimide</div> | Pretilt angle (°) | |
|---|-------------------|---------|
| | Non-rubbing | Rubbing |
| R5CPP-BT | 89.4 | 89.2 |
| R5CPP-OD | 89.3 | 88.6 |
| R5CPP-BP | 89.3 | 88.6 |
| SP-BT | 89.7 | 89.5 |
| SP-OD | 89.2 | 88.0 |
| SP-BP | 89.0 | 88.1 |

TABLE 3 Microscopic Textures of Polyimides (Rubbed Cell)

| Polyimides | Off | On |
|------------|---|---|
| R5CPP-BT |  |  |
| R5CPP-OD |  |  |
| R5CPP-BP |  |  |
| SP-BT |  |  |
| SP-OD |  |  |
| SP-BP |  |  |

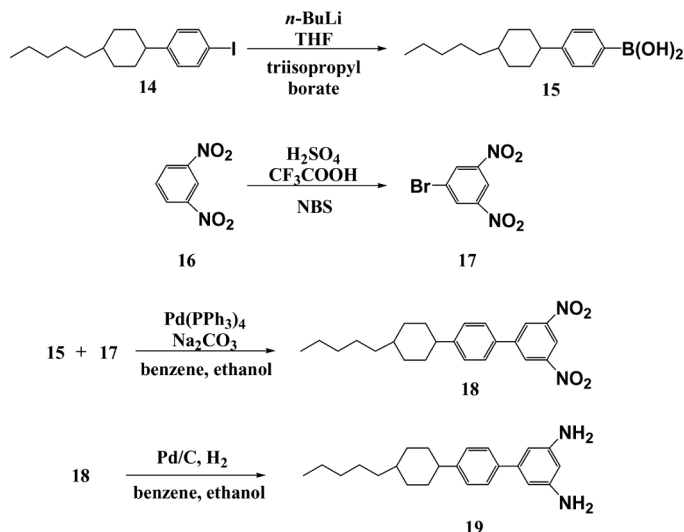
same frequency. So the reason due to increase the interaction between SP type polyimides and LC molecules than R5CPP type polyimides.

To drive TFT-LCD, voltage holding ratio (VHR) is also important to define the ability of LC pixel to keep the state activated by electric field during the frame time [14]. VHR of the polyimides were measured using 6254 instruments at 60 Hz with 5 V DC-voltage. The VHR values of polyimides were listed in Table 5.

Vertical Alignment (VA) mode is a matured technology of LCD field. The switching times can be described by the following formula [15–18],

$$\tau_{on} \propto \frac{\gamma_1 d^2}{\epsilon_0 \Delta \epsilon (V^2 - V_{th}^2)} \tag{2}$$

$$\tau_{off} \propto \frac{\gamma_1 d^2}{\epsilon_0 \Delta \epsilon V_{th}^2} \tag{3}$$

**FIGURE 3** Synthetic route of R5CPP diamine.

where, τ_{on} , τ_{off} are switching on and off times, V the applied voltage, γ_1 rotational viscosity, d cell gap, $\Delta\epsilon$ the dielectric anisotropy, and V_{th} is the voltage needed of 10% transmittance change from the off voltage state.

Numerous contributions have been made to understand the dynamic behavior of various LCDs. Also Eqs. (3) and (4) on even VA cells were proved that there exist a good linear relationship between threshold voltage V_{th} and switching time.

In order to study the relationship between response time and polyimide, switching time measurements were done by using anti-parallel

TABLE 4 Threshold Voltage for the Polyimides

| Polyimide | Host | |
|-----------|------------------|-------------------|
| | $V_{th}(V_{10})$ | $V_{sat}(V_{90})$ |
| R5CPP-BT | 2.1 | 3.8 |
| R5CPP-OD | 2.5 | 4.2 |
| R5CPP-BP | 2.3 | 4.1 |
| SP-BT | 2.5 | 4.5 |
| SP-OD | 2.5 | 4.4 |
| SP-BP | 2.3 | 4.4 |

TABLE 5 VHR of Polyimides (at 25°C)

| Polyimides | VHR (%) |
|------------|---------|
| R5CPP-BT | 98.89 |
| R5CPP-OD | 96.45 |
| R5CPP-BP | 97.36 |
| SP-BT | 97.47 |
| SP-OD | 96.06 |
| SP-BP | 98.79 |

rubbed cell with same cell gap (3.9 μm), driving voltage (a square-wave AC voltage of 5 V, 60 Hz), and LC (MERCK, MJ961213).

According to Table 6, the switching-off time of SP type polyimides was reduced. This phenomenon is because of side chain structure of polyimides. SP diamine has large dipole-moment perpendicularly as the following Figure 5 simulation (semi-empirical quantum chemical methods, AM1 Hamiltonian in the MOPAC 7.0 package). The side chain of SP type polyimides were switched with the electric field because of dipole-moment. And the electric field was cut-off, the side chain of SP type polyimides came back again because of elastic force as well as LC (Fig. 6). In consequence of this, we know that switching-off time of SP type polyimides was improved by the dipole-moment and elastic force of side chain.

CONCLUSION

We synthesized 2 types of polyimide with liquid crystalline shape side chain for fast response time VA mode LCD. One is positive liquid crystalline shape side chain type, the other is negative liquid crystalline shape side chain type. These novel homeotropic alignment materials

TABLE 6 Measurement of Response Time

| Polyimide | Host | | |
|-----------|-------------------------|--------------------------|--------------------------|
| | τ_{on} (ms) | τ_{off} (ms) | τ_{all} (ms) |
| R5CPP-BT | 8.0 | 12.1 | 20.1 |
| R5CPP-OD | 8.3 | 10.1 | 18.4 |
| R5CPP-BP | 7.7 | 12.8 | 20.5 |
| SP-BT | 6.8 | 8.8 | 15.6 |
| SP-OD | 7.2 | 9.3 | 16.5 |
| SP-BP | 6.3 | 10.7 | 17.0 |

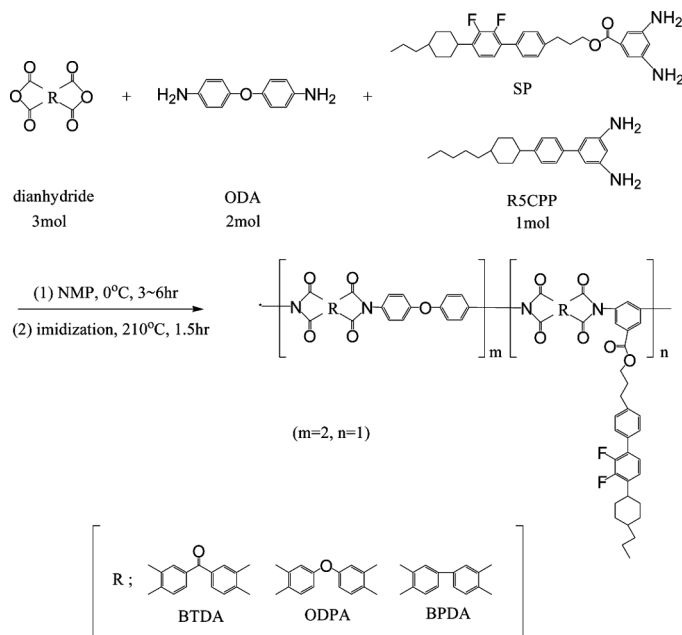
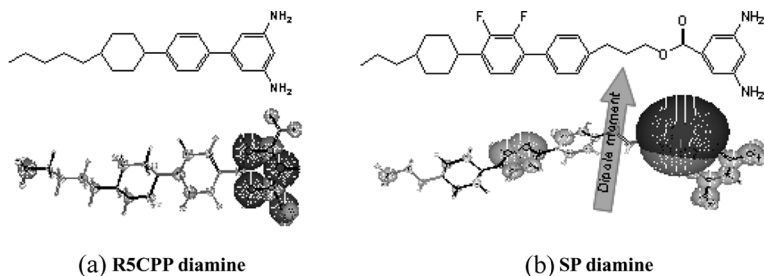


FIGURE 4 Synthetic route of polyimide.

which we have synthesized generated high pretilt angle above 88° . We could observe the LC cell coated the polyimides aligned vertically through the polarized microscope. In addition to negative liquid crystalline shape side chain type showed faster response time than



| | μ_x | μ_y | μ_z | μ | $\beta = \cos^{-1}(\mu_x/\mu)$ |
|---------------|---------|---------|---------|-------|--------------------------------|
| R5CPP diamine | 0.492 | -0.515 | -0.148 | 0.727 | 47.41 |
| SP diamine | 0.641 | -3.601 | -0.850 | 3.754 | 80.17 |

calculated by AM1 method with MOPAC

FIGURE 5 Simulations of the dipole moment (μ) and orientation angle (β).

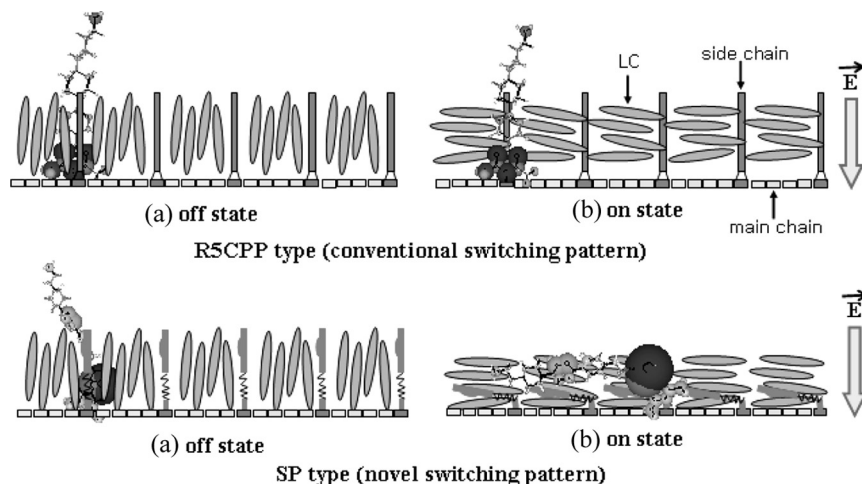


FIGURE 6 Principle of alignment and switching.

positive liquid crystalline shape side chain type because of its structure effect. Therefore, negative liquid crystalline shape side chain type polyimide is expected to use especially for fast response time VA mode LCD.

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