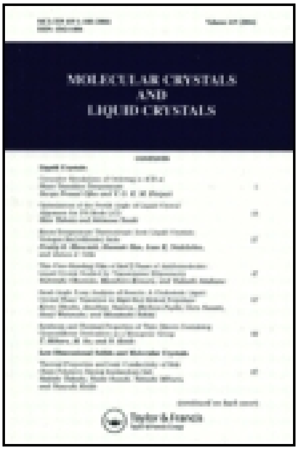

Molecular Crystals and Liquid Crystals

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Synthesis of Symmetric Liquid Crystal Dimers Based on 1,2-bis{-[-(4-alkoxybenzoyloxy)benzylidene]am and Investigation of Phase Behaviour by Varying Alkoxy Terminal Chain Length

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Synthesis of Symmetric Liquid Crystal Dimers Based on 1,2-bis{-[-(4-alkoxybenzoyloxy)benzylidene]amino)-phenoxy}ethane and Investigation of Phase Behaviour by Varying Alkoxy Terminal Chain Length

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With the objective to study the effect of alkoxy terminal chain length on mesomorphic properties of liquid crystals, We have synthesized ester and Schiff base dimers from diamine derivative containing 1,2-dioxyethylene as short spacer with aldehyde derivatives having different lengths of terminal alkoxy chains (n = 6–12). The chemical structure of the final products was investigated by fourier transform infrared (FT-IR) spectroscopy and proton nuclear magnetic resonance (^{1}H NMR) spectroscopy. The mesomorphic properties and optical textures of the resultant dimers were characterized by differential scanning calorimetry (DSC) and polarized optical microscopy (POM). It was found that all the dimers show mesomorphic properties and the increase in terminal alkoxy chain length has pronounced effect on the mesomorphic properties. The temperature range of smectic phase window widens and nematic-isotropic phase window narrows sharply with increasing alkoxy chain length.

Keywords: differential scanning calorimetry; ester and Schiff base; liquid crystal dimmers; optical microscopy

1. INTRODUCTION

Since the discovery of liquid crystal dimmers [1] and subsequent interest in these materials [2] many dimeric systems have been reported. Liquid crystal dimers are of interest because they act as models for main group

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Address correspondence to Kuk Ro Yoon, Organic and Polymer Synthesis Laboratory, Chemistry Department, Hannan University, Daejeon 305-811, Korea. E-mail: kryoon@hannam.ac.kr liquid crystalline polymers that allow the study of flexibility and properties of different mesogenic groups. Thus dimeric liquid crystals retain the crucial structural components of many thermotropic main group polymers. Furthermore, as a distinct class of compounds with unusual properties and potential application, dimeric liquid crystals is of interest in their own right. In typical dimers where two individual mesogenic entities are attached to each other by flexible polymethylene spacer units, the mesomorphic behavior of these compounds is basically dependent on its molecular architecture in which a slight change in the molecular geometry brings about considerable change in its mesomorphic properties. Dimmers containing identical mesogenic units are referred as symmetric dimmers, while the non-symmetric dimmers consist of two differing mesogenic groups. The structure of the mesophases can be modified through change in spacer length and terminal chain length.

Many studies of symmetric dimers have been reported [3,4]. For example, the symmetric Schiff base dimmers prepared by date *et al.* [3], namely the α, ω-bis(4-alkylanilinebenzylidene-4′-oxy) alkanes; these dimers were found to exhibit a rich smectic polymorphism. In 1969, Kelker and coworkers [5] reported for the first time that the Schiff base liquid crystal compound 4-methoxybenzylidene-4′-butylaniline (MBBA) which exhibits a room temperature nematic phase. Since then, Schiff bases, especially Schiff base esters have attracted much attention of liquid crystal researches. Over the past few decades, lot of low molar mass Schiff base esters have been synthesized and investigated extensively for liquid crystalline applications [6–11].

In our ongoing efforts to investigate the correlation between molecular structure and mesomorphic properties [12–15] of these novel materials, here we report the synthesis and the mesomorphic properties of symmetric ester and Schiff base dimers ($2ES_n$), which have terminal alkoxy chains ($-OC_nH_{2n+1}$; $n = 6–12$) and 1,2-dioxyethylene ($-OCH_2CH_2O-$) as a short spacer. The acronym used to refer to these dimmer is $2ES_n$, where 2 represents the number of mesogenic units, ES stands for ester-Schiff base and “n” refers the number of carbon atoms in the terminal alkoxy chain. The molecular structure of the title compounds were characterized by ^1H NMR and FT-IR techniques. The mesomorphic phase transition behaviour of these compounds was investigated by DSC and POM.

2. EXPERIMENTAL

2.1. Materials

1-Fluoro-4-nitrobenzene, ethylene glycol, hydrazine monohydrate, palladium on activated carbon (Pd/C, 10%), 4-hydroxybenzaldehyde, 4-alkoxybenzoic acid (n = 6–12), 1,3-dicyclohexylcarbodiimide (DCC), tetrahydrofuran (THF), p-toluenesulfonic acid (PTSA), anhydrous ethanol were purchased from Aldrich Chemical Co. and used as received. All other solvents and reagents were purchased commercially and used without further purification.

2.2. Synthesis of Ester and Schiff ($2ES_n$) Base Dimmers

The $2ES_n$ dimesogens were prepared as illustrated in Figure 1. The synthetic route used in the preparation of Ester and Schiff dimesogens ($2ES_n$) involved four steps: (1) reaction of 1-fluoro-4-nitrobenzene with ethylene glycol in presence of potassium carbonate to obtain 1,2-bis(4-nitrophenoxy)ethane (NPE), (2) then the reduction of nitro group with hydrazine monohydrate in the presence of palladium on activated carbon to obtain 1,2-bis(4-aminophenoxy)ethane (APE), (3) the 4-(4-alkoxy benzoyloxy) benzaldehyde was obtained by reacting 4-hydroxybenzaldehyde with 4-alkoxybenzoic acid, and (4) the final product 1,2-bis(4-(4-(4-alkoxybenzoyloxy) benzylidene amino)phenoxy)ethane was prepared by reacting 4-(4-alkoxy benzoyloxy) benzaldehyde (22mmol) (ABBn) [16,17] in anhydrous ethanol (50 mL) with

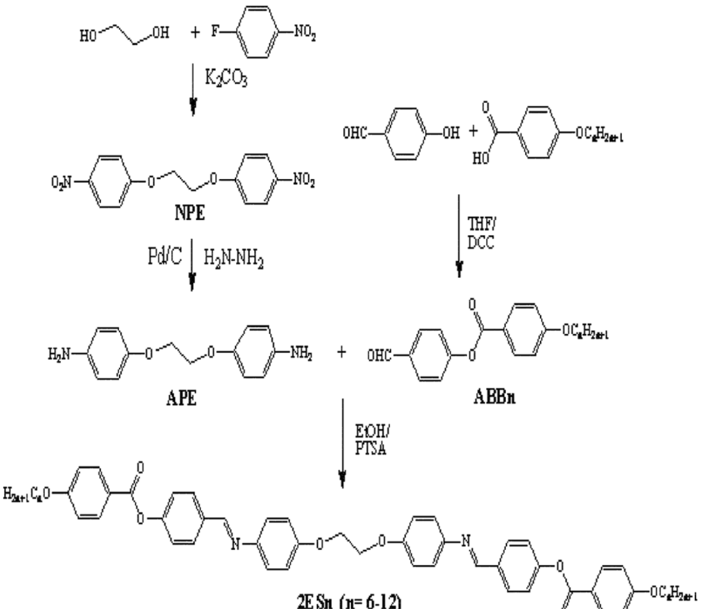

FIGURE 1 Synthetic pathyway for ester-Schiff base dimesogens ($2ES_n$).

1,2-bis(4-amino phenoxy)-ethane (11 mmol) in presence of catalytic amount of PTSA and then the reaction mixture was stirred for 16 h at the room temperature. The resulting precipitate was isolated by filtration and purified by the recrystallization of both chloroform and ethyl acetate solvents (off-white solid, 54–75%). The syntheses of final products were confirmed by FRT-IR and ^{1}H NMR.

2ES$_6$: Yield, 75%. IR (KBr pellet, cm^{-1}): 3070 (Ar−H), 2860, 2930 (aliphatic C−H), 1736 (C=O), 1604 (−CH=N−). ^{1}H NMR ($CDCl_3$, ppm): δ 0.85–0.94 (t, 6H, $-CH_3$), 1.24–1.50 (m, 12H, $-(CH_2)_3-$), 1.79–1.84 (m, 4H, $-\underline{CH_2}CH_2O-$), 4.02–4.07(t, 4H, $-CH_2\underline{CH_2}O-$), 4.37 (s, 4H, $-OCH_2CH_2O-$), 6.96–7.02 (t, 8H, Ar−H), 7.24–7.33 (d, 8H, Ar−H), 7.94–7.97 (d, 4H, Ar−H), 8.13–8.16 (d, 4H, Ar−H), 8.49 (s, 2H, −CH=N).

2ES$_7$: Yield, 56%. IR (KBr pellet, cm^{-1}): 3070 (Ar−H), 2860, 2930 (aliphatic C−H), 1740 (C=O), 1604 (−CH=N−). ^{1}H NMR ($CDCl_3$, ppm): δ 0.84–0.91 (t, 6H, $-CH_3$), 1.15–1.51 (m, 16H, $-(CH_2)_4-$), 1.79–1.88 (m, 4H, $-\underline{CH_2}CH_2O-$), 4.03–4.08 (t, 4H, $-CH_2\underline{CH_2}O-$), 4.38 (s, 4H, $-OCH_2CH_2O-$), 6.97–7.03 (t, 8H, Ar−H), 7.24–7.34 (d, 8H, Ar−H), 7.95–7.98 (d, 4H, Ar−H), 8.14–8.17 (d, 4H, Ar−H), 8.50 (s, 2H, −CH=N).

2ES$_8$: Yield, 75%. IR (KBr pellet, cm^{-1}): 3072 (Ar−H), 2850, 2922 (aliphatic C−H), 1736 (C=O), 1604 (−CH=N−). ^{1}H NMR ($CDCl_3$, ppm): δ 0.86–0.91 (t, 6H, $-CH_3$), 1.24–1.50 (m, 20H, $-(CH_2)_5-$), 1.77–1.84 (m, 4H, $-\underline{CH_2}CH_2O-$), 4.02–4.06 (t, 4H, $-CH_2\underline{CH_2}O-$), 4.37 (s, 4H, $-OCH_2CH_2O-$), 6.96–7.02 (t, 8H, Ar−H), 7.24–7.33 (d, 8H, Ar−H), 7.95–7.98 (d, 4H, Ar−H), 8.13–8.16 (d, 4H, Ar−H), 8.49 (s, 2H, −CH=N).

2ES$_9$: Yield, 58%. IR (KBr pellet, cm^{-1}): 3071 (Ar−H), 2850, 2922 (aliphatic C−H), 1736 (C=O), 1604 (−CH=N−). ^{1}H NMR ($CDCl_3$, ppm): δ 0.86–0.91 (t, 6H, $-CH_3$), 1.24–1.50 (m, 24H, $-(CH_2)_6-$), 1.77–1.84 (m, 4H, $-\underline{CH_2}CH_2O-$), 4.02–4.06 (t, 4H, $-CH_2\underline{CH_2}O-$), 4.37 (s, 4H, $-OCH_2CH_2O-$), 6.96–7.02 (t, 8H, Ar−H), 7.24–7.33 (d, 8H, Ar−H), 7.95–7.98 (d, 4H, Ar−H), 8.13–8.16 (d, 4H, Ar−H), 8.49 (s, 2H, −CH=N).

2ES$_{10}$: Yield, 73%. IR (KBr pellet, cm^{-1}): 3072 (Ar−H), 2850, 2922 (aliphatic C−H), 1736 (C=O), 1604 (−CH=N−). ^{1}H NMR ($CDCl_3$, ppm): δ 0.86–0.91 (t, 6H, $-CH_3$), 1.24–1.50 (m, 28H, $-(CH_2)_7-$), 1.77–1.84 (m, 4H, $-\underline{CH_2}CH_2O-$), 4.02–4.06 (t, 4H, $-CH_2\underline{CH_2}O-$), 4.37 (s, 4H, $-OCH_2CH_2O-$), 6.97–7.03 (t, 8H, Ar−H), 7.24–7.33 (d, 8H, Ar−H), 7.95–7.98 (d, 4H, Ar−H), 8.14–8.17 (d, 4H, Ar−H), 8.49 (s, 2H, −CH=N).

2ES$_{11}$: Yield, 54%. IR (KBr pellet, cm^{-1}): 3070 (Ar−H), 2850, 2922 (aliphatic C−H), 1736 (C=O), 1604 (−CH=N−). ^{1}H NMR ($CDCl_3$, ppm): δ 0.86–0.91 (t, 6H, $-CH_3$), 1.24–1.51 (m, 32H, $-(CH_2)_8-$), 1.77–1.84

(m, 4H, $-\underline{CH_2}CH_2O-$), 4.02–4.06 (t, 4H, $-CH_2\underline{CH_2}O-$), 4.37 (s, 4H, $-OCH_2CH_2O-$), 6.95–7.01 (t, 8H, Ar−H), 7.24–7.33 (d, 8H, Ar−H), 7.95–7.98 (d, 4H, Ar−H), 8.13–8.16 (d, 4H, Ar−H), 8.49 (s, 2H, −CH=N).

2ES$_{12}$: Yield, 71%. IR (KBr pellet, cm^{-1}): 3070 (Ar−H), 2850, 2922 (aliphatic C−H), 1736 (C=O), 1604 (−CH=N−). ^{1}H NMR ($CDCl_3$, ppm): δ 0.91–0.86 (t, 6H, $-CH_3$), 1.24–1.50 (m, 36H, $-(CH_2)_9-$), 1.77–1.84 (m, 4H, $-\underline{CH_2}CH_2O-$), 4.02–4.06 (t, 4H, $-CH_2\underline{CH_2}O-$), 4.37 (s, 4H, $-OCH_2CH_2O-$), 6.96–7.02 (t, 8H, Ar−H), 7.24–7.33 (d, 8H, Ar−H), 7.95–7.98 (d, 4H, Ar−H), 8.13–8.16 (d, 4H, Ar−H), 8.50 (s, 2H, −CH=N).

2.3. Measurements

IR spectra were recorded with a Perkin-Elmer 1000 FT-IR spectrophotometer on KBr pellets. ^{1}H NMR spectra were recorded by using a Varian Mercury 300 (300 MNz) NMR spectrometer. Differential scanning calorimetric measurements were performed using a TA instrument 910 S DSC apparatus under dry nitrogen flow at the scanning rate of 10°C/min. The crude product was repeatedly recrystallized until the transition temperatures were found to be constant and reproducible. The transition temperatures were taken at the maximum point of the peaks for each sample. The transition enthalpies were evaluated from the integrated area of the endothermic peaks using a reference indium sample as the standard. Optical microscopy observation was carried out using a Nikon Labophot-2 polarizing microscope fitted with a RTC-1 temperature controller (Instec Inc., Broom-field, Co.) and a mettler FP-82HT hot stage.

3. RESULTS AND DISCUSSION

3.1. Synthesis and Characterization

The synthetic routes used for the preparation of dimesogenic liquid crystals (2ES$_n$) containing −COO− and −CH=N− units are outlined in Figure 1. The structures of final products were confirmed by ^{1}H NMR (Fig. 2) and FT-IR (Fig. 3) spectroscopy. In ^{1}H NMR spectra, the synthesized 2ES$_n$ compounds showed CH=N peak at 8.49 ppm and the central spacer ($-OCH_2CH_2O-$) at 4.37 ppm. The IR spectral frequencies of the synthesized 2ES$_n$ compounds were obtained using KBr pellets. The absorption peak of ester group was observed at 1736–1740 cm^{-1} and CH=N− stretching frequencies occurred at 1604 cm along with aliphatic C−H stretching frequencies at

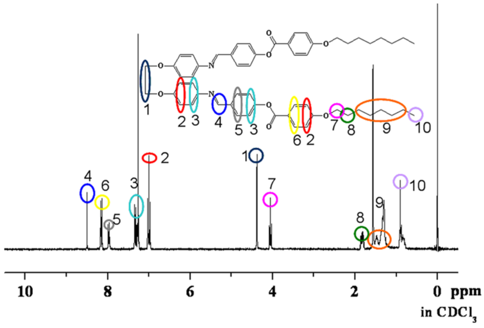

FIGURE 2 ^{1}H NMR spectra of $2ES_8$ compound.

2850–2860, 2922–2930 cm^{-1}. All seven compounds synthesized in the $2ES_n$ series were found to exhibit mesophases. The mesophase transitions exhibited by these symmetrical dimmers were investigated by means of DSC and POM.

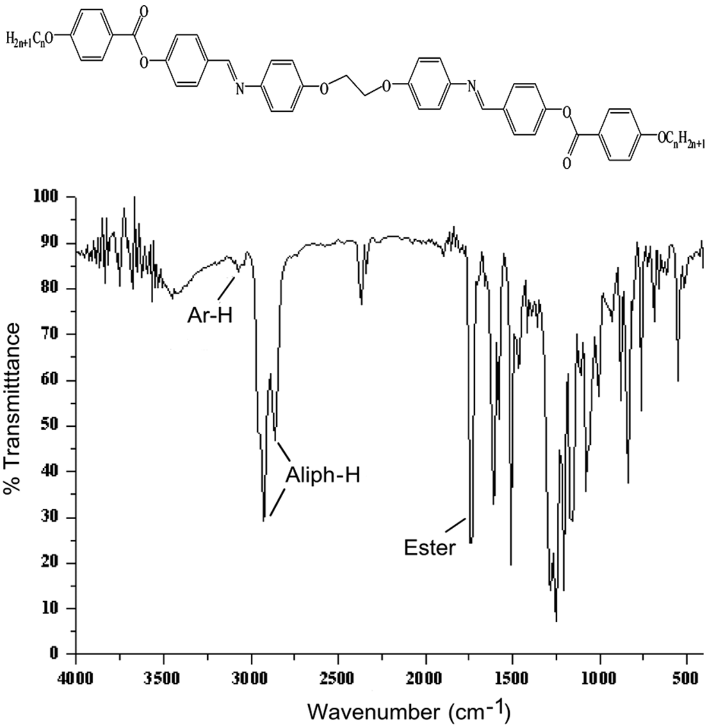

FIGURE 3 FT-IR spectra of $2ES_n$ series compounds.

3.2. The Dependence of Transitional Properties on the Length of Alkoxy Terminal Chains

Figure 4 presents the DSC heating traces of dimesogens ($2ES_n$, $n = 6$–12) and crystalline, smectic and nematic transitions. Table 1 summarizes the thermal transitions and associated enthalpy changes of $2ES_n$ dimesogens. With increase in alkoxy terminal chain length from 6 to 12, smectic to nematic transitions appears. With increase in chain length ($n = 6$–12), the smectic C transition increased from 229.8 to 311.8°C, however, the nematic transition decreased from 351.4 to 306.6°C, and smectic C-nematic transitions become closer. The existence of smectic C to nematic (SmC-N) transition was confirmed by the observation of schlieren textures in optical microscopy when compound were heated from smectic C phase. It can be seen from the Table 1, the crystal to smectic A phase transition at 167.4°C ($2ES_6$) decreased to 152.6°C ($2ES_{12}$) with increase in chain length indicating the reduction in crystallanity and increase in flexibility of the dimmers.

Figure 5 shows the dependence of the transition temperature on the terminal chain length (n). This figure clearly shows the increase in phase window of smectic A and smectic C phases, where as the nematic phase window narrows sharply with increasing alkyl chain length. The increasing terminal chain length is anticipated to increase significantly the length-to-breadth ratio in resulting in liquid crystal

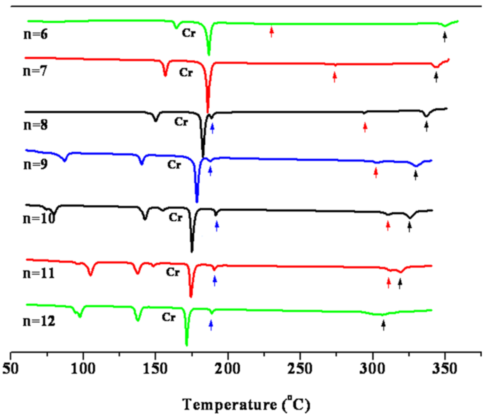

FIGURE 4 Differential scanning calorimetry thermograms for ester-Schiff base dimesogens ($2ES_n$; $n = 6$–12) in the heating scans (10°C/min). The arrows indicate the phase transitions observed in optical microscope.

TABLE 1 Thermal Transition Temperatures and Associated Enthalpy Changes of Ester-Schiff's Base Dimesogens ($2ES_n$)

	Phase transition temperatures (°C) and associated enthalpy change (ΔH, kJ/mol)			
Number of carbons in $2ES_n$ series	Crystalline phase	Smectic A phase	Smectic C phase	Nematic phase
6	167.4(26.6)	–	229.8(0.34)	351.4(10.5)
7	165.3(53.6)	–	273.9(0.98)	343.9(9.76)
8	162.2(49.7)	188.5(8.11)	293.6(1.40)	337.1(11.5)
9	161.0(47.0)	187.8(4.43)	302.2(1.70)	329.7(7.22)
10	157.4(46.0)	191.4(5.26)	310.5(1.93)	325.8(12.3)
11	156.2(44.4)	190.3(5.33)	311.8(1.93)	319.4(7.66)
12	152.6(43.8)	188.6(5.19)	–	306.6(25.2)

phases, and particularly smectic phases, being stabilized at higher temperatures for higher homologues. These results demonstrate that the tendency toward smectic mesomorphism and the thermal stability of smectic phase increases with increasing terminal alkoxy chain length. The effects of the terminal chain length on the transition temperatures and phases behavior observed in this series are in accordance with those observed for conventional low molar mass mesogens. However the odd-even effect observed [4] for similar compounds were not observed for these $2ES_n$ compounds.

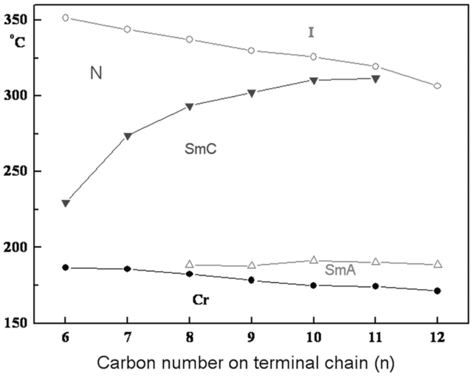

FIGURE 5 Dependence of transition temperatures on the increasing number of carbon atoms (n) in the terminal alkoxy chains for the $2ES_n$ series compounds.

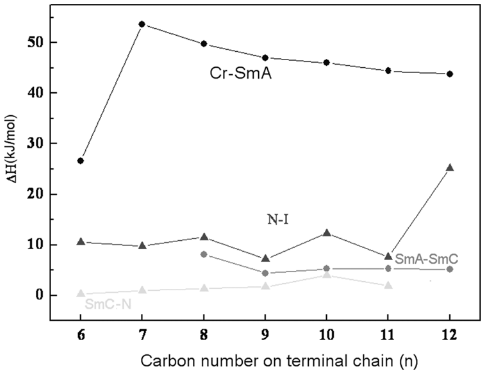

FIGURE 6 Dependence of enthalpy changes (ΔH) associated with transition temperatures on the increasing number of carbon atoms (n) in the terminal alkoxy chain for the $2ES_n$ series compounds.

Figure 6 shows the dependence of enthalpy changes associated with crystalline-smectic A (Cr-SmA), smectic A-smetic C (SmA-SmC), smectic C-nematic (SmC-N) and nematic-isotropic (N–I) phase transitions on carbon number (n) of the terminal chain. It is interesting to observe that the odd-even effect for mesophase transitions in general and nematic-isotropic (N–I) transition in particular. The N–I transition enthalpies for even numbers are in most instances much higher than those of the odd number terminal chains. However, the enthalpy changes associated with crystallization decreased from 53.6 to 43.8 kJ/mol indicating effect of chain length on the crystallization behaviour. Eventhough enthalpy changes does not identify the mesophases, it can give an indication about existence different type of mesophases and further enthalpy changes associated with the crystalline phase are generally higher than that observed for mesophase as observed here.

3.3. Optical Properties

DSC analysis reveals the existence of enantiotropic transitions and these transitions are more visible and sharp with increase in alkoxy terminal chain especially for ES (8–11) compounds. The optical textures of these selective compounds in the observed mesophase window are shown Figures 7–10 in the order. The properties of liquid crystalline phases were studied using POM with a hot stage. Optical micrographs of $2ES_8$ obtained during the heating cycle at different temperatures are shown in Figure 7 and optical micrographs of

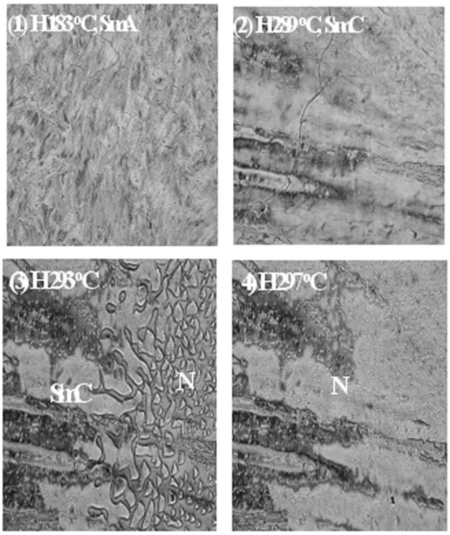

FIGURE 7 Optical textures of (1) SmA of $2ES_8$ (183°C); (2) SmC of $2ES_8$ (289°C); (3) SmC-N transition of $2ES_8$ (293°C); (4) N of $2ES_8$ (297°C).

$2ES_{10}$ are shown in Figure 8. The optical textures of these new types of compounds are tentatively identified based on DSC results and its resemblance. Schlieren optical texture of $2ES_8$ (Fig. 4) reveals the

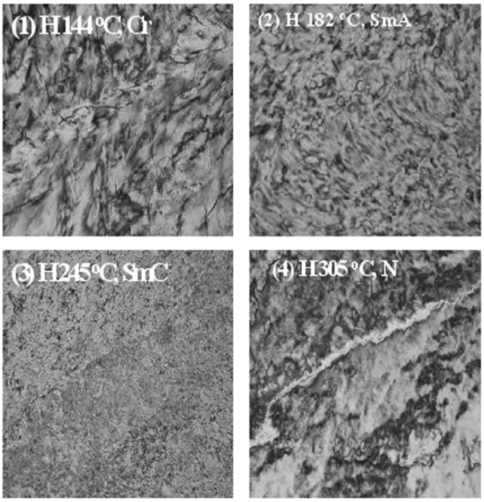

FIGURE 8 Optical textures of (1) Cr of $2ES_9$ (144°C); (2) SmA of $2ES_9$ (182°C); (3) SmC of $2ES_9$ (245°C); (4) N of $2ES_9$ (305°C).

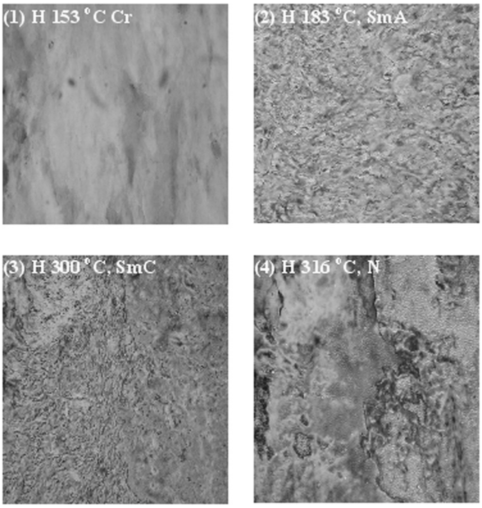

FIGURE 9 Optical textures of (1) Cr of 2ES$_{10}$ (153°C); (2) SmA of 2ES$_{10}$ (183°C); (3) SmC of 2ES$_{10}$ (300°C); (4) N of 2ES$_{10}$ (316°C).

transformation of smectic phase into nematic (N) transitions on heating. Smectic A phase of 2ES$_8$ at 183°C is shown in Figure 4(1), smectic C phase of 2ES$_8$ at 289°C is shown in Figure 4(2), smectic

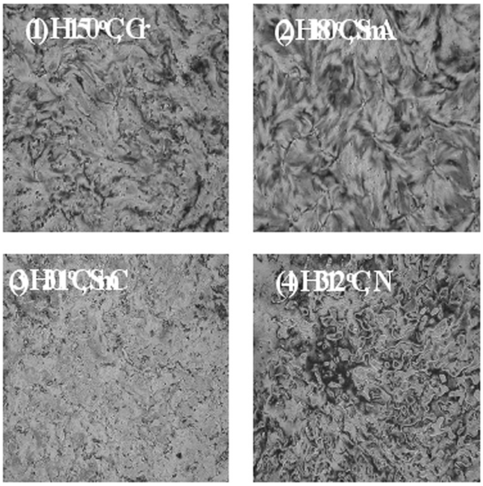

FIGURE 10 Optical textures of (1) Cr of 2ES$_{11}$ (150°C); (2) SmA of 2ES$_{11}$ (180°C); (3) SmC of 2ES$_{11}$ (301°C); (4) N of 2ES$_{11}$ (312°C).

C-nematic (SmC-N) transition of $2ES_8$ at 293°C at Figure 4(3) and nematic (N) phase of $2ES_8$ at 297°C is shown in Figure 4(4) respectively. The different mesomorphic phases of $2ES_{10}$ are shown in Figure 5(1)-crystalline phase at 153°C, 5(2)-smectic A phase at 183°C, 5(3)-smectic C phase at 300°C and 5(4)-nematic(N) phase at 316°C respectively. The different mesomorphic phases observed are consistent with the DSC analysis results.

4. CONCLUSIONS

We have synthesized a new series of ester and schiff base liquid crystalline compounds from diamine derivative containing 1,2-dioxyethylene as short spacer with aldehyde derivatives having terminal alkoxy chains ($n = 6$–12). The chemical structures of the final products were investigated by FT-IR and ^{1}H NMR spectroscopy. The mesomorphic properties and optical textures of the resultant dimers were characterized by DSC and POM. The existence of smectic C-nematic (SmC-N) transitions was confirmed by the observation of schlieren textures in optical microscopy when compound were heated from smectic phase. It was found that all the dimers show mesomorphic properties and the change in terminal alkoxy chain length has pronounced effect on the mesomorphic properties. The temperature range of smectic phase window widens and nematic phase window narrows sharply with increasing alkoxy chain length.

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