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Liquid Crystals

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A new series of liquid-crystalline bi-1,3,4-oxadiazole derivatives: synthesis and mesomorphic behaviour

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A new series of liquid-crystalline bi-1,3,4-oxadiazole derivatives: synthesis and mesomorphic behaviour

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Symmetrical bi-1,3,4-oxadiazole derivatives, namely 5,5'-bis(phenyl 4-alkoxybenzoate)-2,2'-bi-1,3,4-oxadiazole (BBOXD-*n*, *n*=6, 10, 14, 16), were synthesised. All BBOXD-*n* exhibited remarkably stable SmC phases by virtue of the high transition enthalpies of SmC–I. In addition, BBOXD-6 and BBOXD-10 showed an enantiotropic nematic phase with enthalpies of the N–I transition up to 5.16 kJ mol⁻¹. As confirmed by wide-angle X-ray diffraction analysis and MM2, molecules of BBOXD-*n* showed high-angle tilting (55–57°) within their smectic C phases.

Keywords: large-angle tilted SmC phase; bi-1,3,4-oxadiazole; remarkably stable nematic and smectic C phases

1. Introduction

In recent years the significant interest in mesomorphic heterocyclic compounds has dramatically increased due to their more diversified structural figures and distinct mesomorphic properties (1, 2). 1,3,4-oxadiazole derivatives, five-membered heterocyclic compounds constituting nitrogen and oxygen atoms, are one of the more widely investigated classes, both as additives and as mesogens (2). More recently, several discotic 1,3,4-oxadiazole mesomorphic compounds, showing columnar mesophases, in which the oxadiazole moiety was used as the discotic core have been reported (3–6). For example, a star-shaped discotic compound containing 1,3,5-triethynylbenzene core and oxadiazole-based rigid arms exhibited a discotic nematic phase (N_{Col}) (3). Lai et al. reported that 2,5-bis(3,4,5-trialkoxo phenyl)-1,3,4-oxadiazoles exhibited hexagonal columnar phases (Col_h), while their complexes with metals, Col_r and Col_h phases (5, 6). In our recent paper, we synthesised new types of polycatenar molecules by using bi-1,3,4-oxadiazole as the rigid core, namely 2,2'-bis(3,4,5-trialkoxo phenyl)-bi-1,3,4-oxadiazole. They have been confirmed to give rise to columnar mesophases (7).

The shape of a molecule has a critical effect on liquid crystallinity. Many new materials with non-linear boomerang-shaped architecture have been found to exhibit new mesophases referred to as banana phases (8). Specifically, the introduction of five-membered heterocycles within the central core of a calamitic molecule results in banana mesophases due to the dipolar moment and the bent angle (9–15).

As early as 1998 a boomerang-shaped molecule, namely 4,4'(1,3,4-oxadiazole-2,5-diyl) di-*p*-heptylbenzoate, attracted much attention because of the possibility of the occurrence of a biaxial nematic phase (9). Deuterium nuclear magnetic resonance (NMR) spectroscopy (10) and X-ray scattering (11) were employed to confirm this unique phase. As noted by Samulski, the oxadiazole heterocycle has a transverse dipole moment which led to a stabilisation of the biaxial nematic phase. Furthermore, Goodby et al. revealed enantioselective segregation in this achiral nematic liquid crystal (12). Its homologues reversing the sense of the ester moiety were also reported (13). Conoscopic experiments indicate the existence of a biaxial smectic phase. Analogously, Watanabe et al. reported an oxadiazole-based molecule which exhibits an interesting polymorphism in which the Bx phase is formed in the lower-temperature region rather than the well-known calamitic phases (14), i.e. nematic, SmA, and SmC phases. Such polymorphism, which includes banana and calamitic liquid crystals, is attributed to the moderate bend angle of the oxadiazole cores. In addition, enantioselective segregation in the Bx phase has been investigated (15).

In this work, as part of our continuing research in oxadiazole derivatives, we report the preparation and mesomorphic behaviour of a new series of compounds derived from a symmetric bi-1,3,4-oxadiazole core, e.g. 5,5'-bis(phenyl 4-alkoxybenzoate)-2,2'-bi-1,3,4-oxadiazole (BBOXD-*n*, *n*=6, 10, 14, 16). They are rod-like in shape although consist of 1,3,4-oxadiazole rings as a part of the rigid core. These

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discovery of these new materials are examples of the continuing effort to explore the limits of molecular core compatibility with liquid crystallinity. Remarkably stable nematic ($n=6, 10$) and smectic C ($n=6, 10, 14, 16$) phases were observed. Interestingly, molecules of BBOXD- n exhibited a large-angle ($\theta > 55^\circ$) tilting within their SmC phases.

2. Experimental

2.1. Characterisation

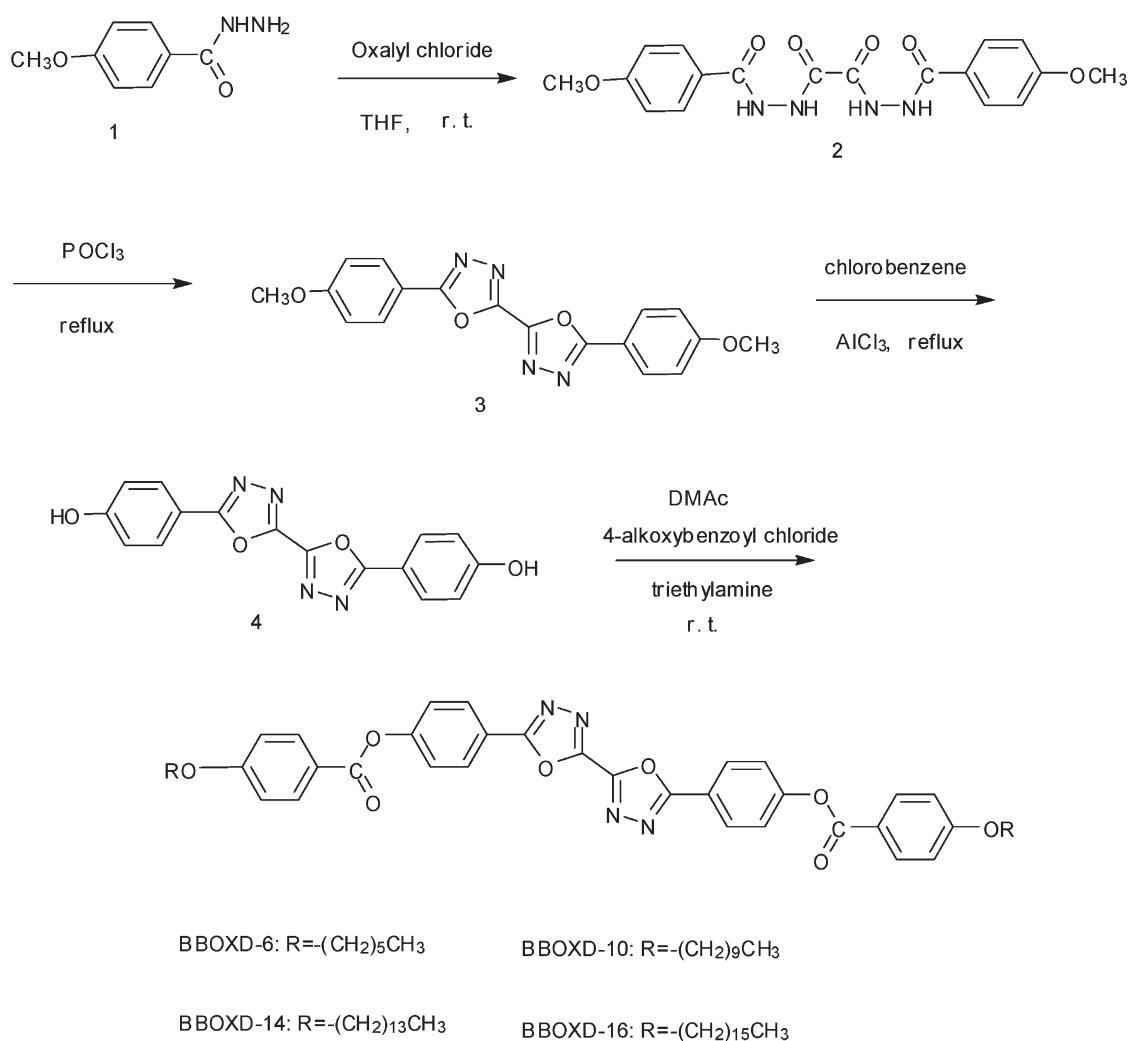
^1H NMR spectra were recorded with a Mercury-300BB 300 MHz spectrometer, using tetramethylsilane (TMS) as an internal standard. Phase transitional properties were investigated with a Netzsch DSC 204. The rate of heating and cooling was $10^\circ\text{C min}^{-1}$; the weight of the sample was about 3 mg, and indium and zinc were used for calibration. The peak maximum was taken as the

phase transition temperature. Texture observation was conducted on a Leica DMLP polarising optical microscope equipped with a Leitz 350 microscope heating stage. X-ray diffraction was carried out with a Bruker Avance D8 X-ray diffractometer. Fourier transform infrared (FT-IR) spectra were recorded with a Perkin-Elmer spectrometer (Spectrum One B). The sample was pressed into tablet with KBr.

2.2. Synthesis

4-methoxybenzoyl hydrazine and oxalyl chloride were used as received. Tetrahydrofuran (THF) was refluxed over sodium under argon and distilled before use.

Scheme 1 shows the synthetic route for BBOXD- n . Oxalyl chloride (11 mmol) was regularly injected into the THF solution of 4-methoxybenzoyl hydrazine (23 mmol) with vigorous stirring



Scheme 1. Synthetic route for BBOXD- n compounds.

at room temperature for 8 h, yielding the product of oxalyl acid *N', N'*-di(4-methyloxybenzoyl)-hydrazide **2**, which was purified by washing with alcohol. The purified **2** (10 mmol) was dissolved in phosphorous oxychloride (POCl₃) and refluxed for about 40 h. Excess POCl₃ was removed through distillation and the residue was slowly added to the ice-water. After the removal of solvent under reduced pressure, 2, 2-bis(4-methoxyphenyl)-bi-1, 3, 4-oxadiazole **3** was purified by recrystallisation with dimethyl sulfoxide (DMSO). (Because of the poor solubility, NMR spectra of Compounds **2** and **3** could not be obtained. However, X-ray single-crystal analysis of compound **3** will be published elsewhere.) 2, 2-Bis(4-hydroxyphenyl)-bi-1, 3, 4-oxadiazole **4** was prepared through the reaction of 2, 2-bis(4-methoxyphenyl)-bi-1, 3, 4-oxadiazole (9.4 mmol) in chlorobenzene (250 ml) at 110°C for about 1 h using aluminium trichloride (40 mmol) as catalyst. The crude product of **4** was precipitated out by pouring the reaction mixture into an excess of boiling water; it was further purified by recrystallisation with chloroform. The esterification between compound **4** and 4-alkoxybenzoyl chloride was performed in *N,N*-dimethylacetamide at room temperature for 8 h using triethylamine as catalyst. 4-alkoxybenzoyl chloride was prepared as described by Dave et al. (16). The purified **4** (8.3 mmol) was dissolved in *N,N*-dimethylacetamide (30 ml) by heating, and then slowly injected into the *N,N*-dimethylacetamide solution (150 ml) of excess 4-alkoxybenzoyl chloride with vigorous stirring at room temperature. A small amount of triethylamine (3–5 ml) as catalyst was added to the reaction mixture. The resulting precipitate was collected, dried under vacuum, and recrystallised from ethanol/chloroform to give pure BBOXD-*n* with a yield of more than 90% for further ¹H NMR, FTIR measurements and elemental analysis.

oxalyl acid *N', N'*-di(4-methyloxybenzoyl)-hydrazide

FT-IR (KBr, pellet, cm⁻¹): 3426, 3192, 2967, 2879, 1690, 1647, 1608, 1578, 1507, 1443, 1417, 1342, 1307, 1255, 1183, 1119, 1027, 907, 848, 826, 790, 690, 633, 617.

2, 2-Bis(4-methoxyphenyl)-bi-1, 3, 4-oxadiazole

FT-IR (KBr, pellet, cm⁻¹): 2974, 2877, 1609, 1549, 1491, 1465, 1450, 1423, 1309, 1295, 1254, 1178, 1149, 1120, 1081, 1016, 952, 841, 798, 740, 701, 636, 616.

2, 2-Bis(4-hydroxyphenyl)-bi-1, 3, 4-oxadiazole

¹H NMR (300 MHz, CDCl₃), (ppm, from TMS): 10.55 (s, 2H); 7.99 (d, 4H *J*=9.00 Hz); 7.02 (d, 4H, *J*=9.00 Hz).

FT-IR (KBr, pellet, cm⁻¹): 3331, 1611, 1597, 1564, 1494, 1468, 1332, 1281, 1229, 1170, 1114, 1079, 1013, 964, 950, 855, 825, 739, 702, 670, 625.

5,5'-bis(phenyl4-hexoxybenzoate)-2,2'-bi-1,3,4-oxadiazole.(BBOXD-6)

¹H NMR (300 MHz, CDCl₃), (ppm, from TMS): 8.32 (d, 4H, *J*=8.70 Hz); 8.14 (d, 4H, *J*=8.70 Hz); 7.47 (d, 4H, *J*=8.70 Hz); 7.00 (d, 4H, *J*=8.70 Hz); 4.06 (t, 4H, *J*=6.60 Hz); 1.81 (m, 4H); 1.37 (m, 12H); 0.93 (t, 6H, *J*=7.2 Hz).

FT-IR (KBr, pellet, cm⁻¹): 2955, 2925, 2869, 2858, 1731, 1606, 1580, 1552, 1513, 1491, 1471, 1421, 1399, 1315, 1304, 1261, 1204, 1166, 1019, 1010, 954, 844, 761.

Elemental analysis: calculated for C₄₂H₄₂N₄O₈ (%), C 69.03, H 5.79, N 7.67; found, C 69.13, H 5.92, N 7.79.

5,5'-bis(phenyl4-decoxybenzoate)-2,2'-bi-1,3,4-oxadiazole.(BBOXD-10)

¹H NMR (300 MHz, CDCl₃), (ppm, from TMS): 8.32 (d, 4H, *J*=8.70 Hz); 8.16 (d, 4H, *J*=8.70 Hz); 7.47 (d, 4H, *J*=8.70 Hz); 7.00 (d, 4H, *J*=8.70 Hz); 4.06 (t, 4H, *J*=6.60 Hz); 1.83 (m, 4H); 1.29 (m, 28H); 0.89 (t, 6H, *J*=6.9 Hz).

FT-IR (KBr, pellet, cm⁻¹): 2921, 2851, 1732, 1603, 1579, 1547, 1510, 1487, 1472, 1420, 1399, 1318, 1302, 1252, 1205, 1162, 1016, 952, 848, 762.

Elemental analysis: calculated for C₅₀H₅₈N₄O₈ (%), C, 71.24, H, 6.93, N, 6.65; found, C, 71.31, H, 6.93, N, 6.68.

5,5'-bis(phenyl4-tetradecoxybenzoate)-2,2'-bi-1,3,4-oxadiazole.(BBOXD-14)

¹H NMR (300 MHz, CDCl₃), (ppm, from TMS): 8.32 (d, 4H, *J*=8.40 Hz); 8.16 (d, 4H, *J*=8.70 Hz); 7.46 (d, 4H, *J*=9.00 Hz); 6.99 (d, 4H, *J*=8.70 Hz); 4.06 (t, 4H, *J*=6.60 Hz); 1.83 (m, 4H); 1.26 (m, 44H); 0.88 (t, 6H, *J*=6.5 Hz).

FT-IR (KBr, pellet, cm⁻¹): 2922, 2852, 1733, 1603, 1579, 1548, 1509, 1488, 1471, 1420, 1399, 1318, 1302, 1252, 1206, 1162, 1015, 1008, 952, 845, 762.

Elemental analysis: calculated for C₅₈H₇₄N₄O₈ (%), C, 72.93, H, 7.81, N, 5.87; found, C, 72.66, H, 7.95, N, 5.90.

5,5'-bis(phenyl4-hexadecoxybenzoate)-2,2'-bi-1,3,4-oxadiazole.(BBOXD-16)

¹H NMR (300 MHz, CDCl₃), (ppm, from TMS): 8.33 (d, 4H, *J*=8.40 Hz); 8.16 (d, 4H, *J*=9.30 Hz); 7.47 (d, 4H, *J*=9.00 Hz); 7.0 (d, 4H, *J*=8.70 Hz); 4.06 (t, 4H, *J*=6.60 Hz); 1.83 (m, 4H); 1.27 (m, 52H); 0.88 (t, 6H, *J*=6.5 Hz).

FT-IR (KBr, pellet, cm⁻¹): 2922, 2852, 1733, 1604, 1579, 1548, 1510, 1487, 1472, 1420, 1398, 1318, 1302, 1252, 1207, 1162, 1020, 1007, 952, 846, 762.

Elemental analysis: calculated for C₆₂H₈₂N₄O₈ (%), C, 73.63, H, 8.17, N, 5.54; found, C, 73.39, H, 8.36, N, 5.59.

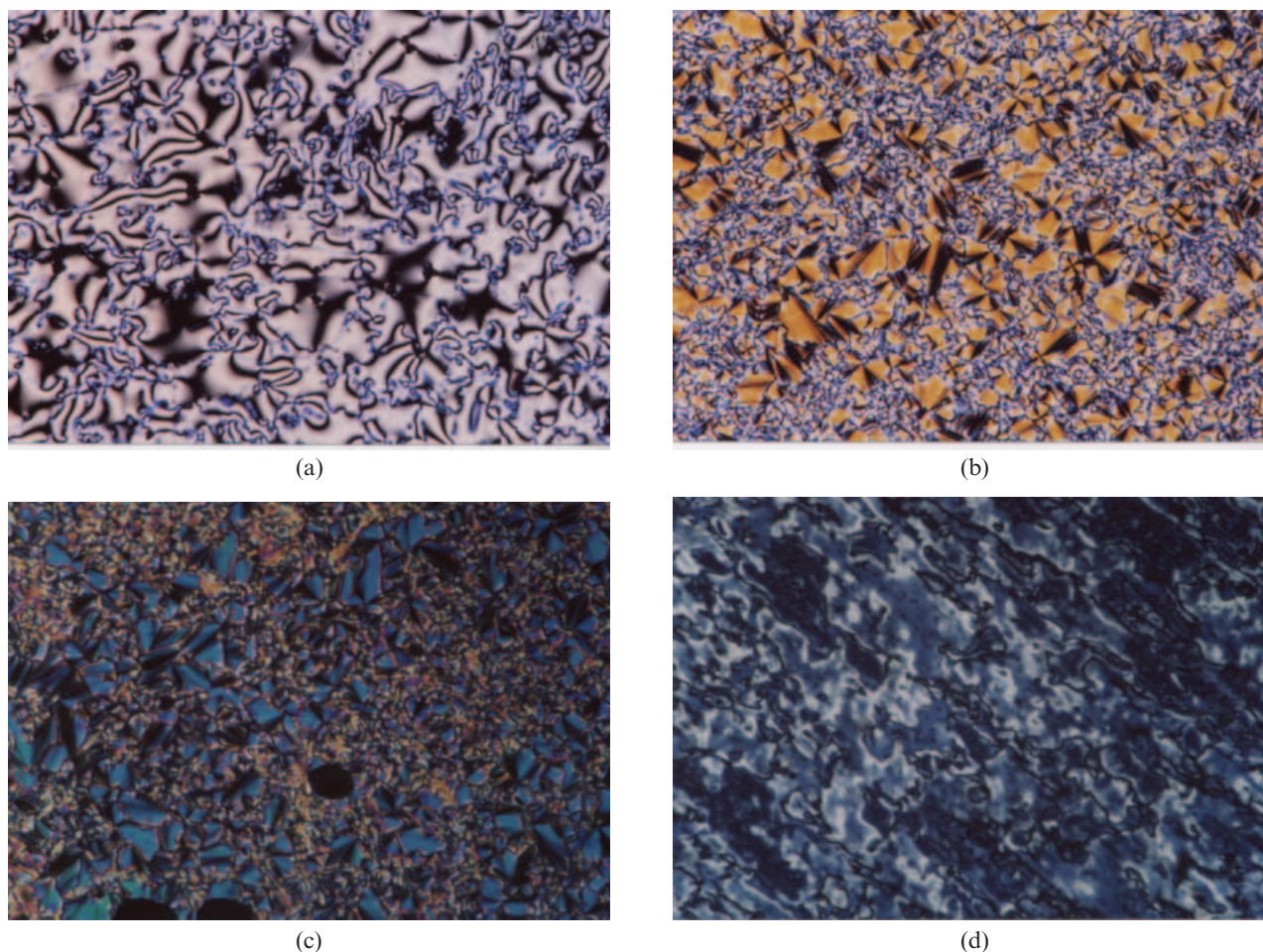


Figure 1. Polarising optical photomicrographs of BBOXD-6 and BBOXD-10. (a) Scheliren texture of BBOXD-10 at 288°C ($\times 400$); (b) the coexisting of Scheliren and fan-shaped texture of BBOXD-10 showing SmC phase at 220°C ($\times 400$); (c) fan-shaped texture of BBOXD-6 showing SmC phase at 180°C ($\times 400$); (d) Scheliren texture of BBOXD-6 after shearing at 180°C ($\times 400$).

3. Results and discussion

The mesomorphic behaviour of BBOXD- n was studied by polarised optical microscopy, differential scanning calorimetry and wide-angle X-ray diffraction. BBOXD-6 and BBOXD-10, showed schlieren textures ($S = \pm 1/2, \pm 1$) (17) (Figure 1 (a)), suggesting its nematic feature. Apart from the nematic phase, BBOXD-6 exhibited a broken fan-shaped texture (Figure 1 (c)) upon cooling from the nematic phase, which gave a lined schlieren texture (Figure 1 (d)), suggesting its smectic C feature. Schlieren coexistence fan-shaped textures were observed for the BBOXD- n ($n=10, 14, 16$) with long terminal alkyl chains, indicating the existence of SmC phase.

Figure 2 showed the DSC curves of BBOXD- n both in first cooling and the second heating cycles. The transitional temperatures and associated enthalpies of BBOXD- n are summarised in Table 1.

It can be seen that BBOXD-6 showed two transitions at 205°C and 344°C corresponding to Cr-N and N-I transitions during heating, and three exothermic peaks at 345°C, 208°C and 159°C corresponding to I-N, N-SmC and SmC-Cr transitions upon cooling from its isotropic phase. In contrast, BBOXD-10 showed enantiotropic nematic and smectic C phases in the ranges of 301–271°C and 271–171°C, respectively in the course of heating. Higher homologues of BBOXD- n ($n=14, 16$) exhibited an enantiotropic smectic C phase and their ranges of smectic C phase are 148–279°C and 151–284°C (second heating run), which are broader than that of BBOXD-6 and BBOXD-10. Obviously, BBOXD-6 with short terminal chains exhibited a remarkably stable nematic phase, which was characterised by a wide mesophase range (139°C), high clearing point (as high as 344°C) and a large isotropic transition enthalpy up to 5.16 kJ mol⁻¹, which is five

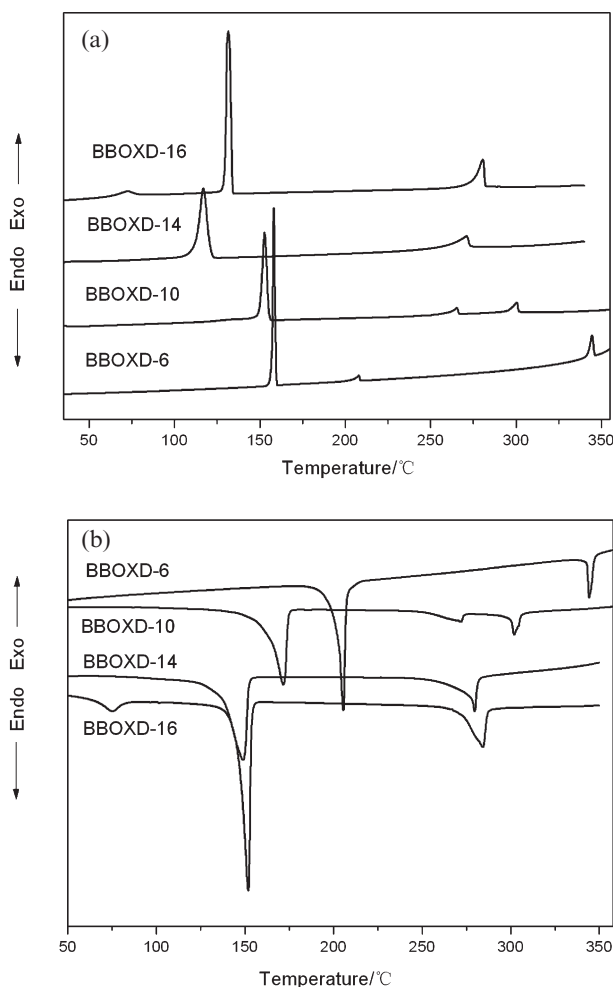


Figure 2. (a) DSC curves of BBOXD-*n* in the first cooling run. (b) DSC curves of BBOXD-*n* in the second heating run.

times larger than that of conventional N–I transitions. Elongation of the terminal chains of BBOXD-*n* led to the narrowing and finally the disappearance of nematic phase, and gave rise to the smectic C phase. Additionally, the transitional enthalpies of SmC–I increased and the SmC phase ranges of BBOXD-*n* broadened with increasing terminal chain length. This is due to an increasing microphase segregation effect arising from the enhanced incompatibility between the rigid aromatic rings and flexible alkoxy chains with increasing terminal chain length. Such a

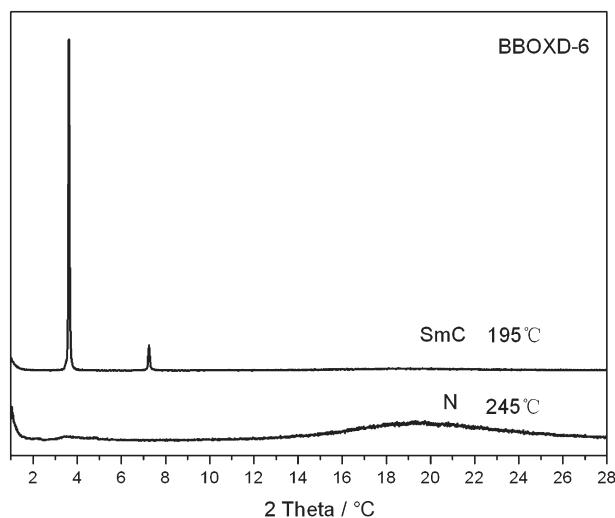


Figure 3. X-ray diffraction pattern of BBOXD-6 at different temperatures.

microsegregation effect was considered to be the driving force for the formation of the smectic phase (18, 19). The high stability of either the smectic C or nematic phase of BBOXD-*n*, indicated the presence of strong intermolecular interaction which might have stabilised the parallel alignment of molecules (20–24).

In order to evaluate the molecular arrangements in their mesophases, XRD was performed on BBOXD-*n* at different temperatures. Figure 3 shows the XRD patterns of BBOXD-6 in its nematic (at 245°C) and SmC phase (at 195°C). The characteristic patterns of smectic C phase consists of two sharp peaks in the low-angle region implying the formation of a layered structure, and a broad diffuse peak in the wide-angle region centred at *d*-spacing of 4.6 Å, indicating the liquid-like arrangement of the molecules within the layers. Considering its schlieren and broken fan-shaped texture, the molecules of BBOXD-6 are arranged in monolayers with the molecular long axis tilted at 55.6° with respect to the layer normal. Data for layer spacings (*d*), molecular lengths (*l*), and *d*/*l* ratios of BBOXD-*n* in their SmC phases are given in Table 2. The layer spacings (*d*) were found to be much smaller than the estimated all-trans molecular lengths of the most extended conformation, and the *d*/*l* ratios are in the

Table 1. Thermal transitional properties of BBOXD-*n*. Transitional temperatures (°C) and the enthalpies of transition (kJ mol⁻¹) in parentheses.

Compound	First cooling	Second heating
BBOXD-6	I 345 (4.89) N 208 (1.82) SmC 159 (25.76) Cr	Cr 205 (28.92) N 344 (5.16) I
BBOXD-10	I 300 (4.86) N 265 (3.86) SmC 153 (33.04) Cr	Cr 171 (33.78) SmC 271 (4.32) N 301 (5.12) I
BBOXD-14	I 271 (9.69) SmC 117 (33.59) Cr	Cr 148 (34.03) SmC 279 (11.92) I
BBOXD-16	I 280 (19.41) SmC 132 (53.60) Cr _b 72 (7.17) Cr _a	Cr _a 74 (7.14) Cr _b 151 (53.61) SmC 284 (19.79) I

Cr, SmC, N and I indicate crystalline state, smectic C phase nematic phase and isotropic liquid, respectively.

Table 2. WAXD results for BBOXD-*n* in their liquid-crystalline phases.

Compound	^a Molecular length(ℓ)/Å	$T/^\circ\text{C}$	Layer spacing(d)/Å	d/ℓ	$\theta/^\circ$
BBOXD-6	42.87	172	24.3	0.57	55.6
BBOXD-10	52.64	180	29.5	0.56	56.1
BBOXD-14	62.18	190	34.4	0.55	56.6
BBOXD-16	66.94	200	37.5	0.56	55.9

^aMolecular length was calculated by MM2.

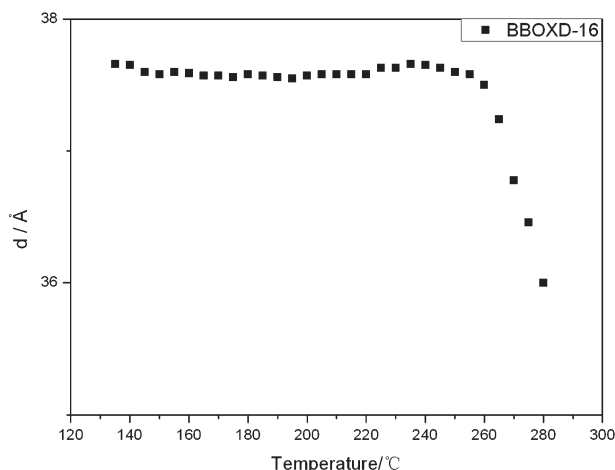


Figure 4. Plot of the layer spacing of BBOXD-16 versus temperature within its SmC phase.

range of 0.5~0.6. Thus molecules of BBOXD-*n* exhibited large tilting (c. 55.6–56.6°) from the layer normal within their smectic C phases.

Figure 4 shows the dependence of the layer spacing (d) of BBOXD-16 in the smectic C phase on temperature. Clearly, the layer spacing in its SmC phase remained almost unchanged (37.5 Å) within a wide range of temperature and decreased gradually at the onset of the isotropic phase. Considering the fact that molecules of BBOXD-*n* exhibited a high-angle tilting within the smectic C phase as well as the relatively large transitional enthalpies of SmC–I, it is reasonable to assume that there existed strong intermolecular interaction among BBOXD-*n* molecules.

In conclusion, a new series of calamitic 1,3,4-oxadiazole derivatives (BBOXD-*n*) was synthesised through rational incorporation of rigid bi-1,3,4-oxadiazole as part of the rigid core. They have been revealed to exhibit either a nematic or smectic C phase depending on the length of terminal alkyl chains. The appearance of high-angle tilting of the director within the smectic C phase as well as the highly stable nematic phase for the lower homogenous of BBOXD-*n* ($n=6, 10$) might indicate the presence of intermolecular attractive forces within the phases. The combination of lateral intermolecular interaction and the microphase

segregation effect may be the leading contributor to this stable smectic C phase.

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