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Dependence of phase transition of a bent-core liquid crystal on the heating rate

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Abstract. A bent-core liquid crystal compound N,N-bis (4-propoxybenzylidene)benzene-1,3-diamine was synthesized. Phase transition properties of the synthesized compound with the deferent heating rate were characterized with differential scanning calorimetry and polarized optical microscopy, respectively. Our results demonstrated that the bent-core compound exhibited the completely undivided multi-phases in heating-rate ranges from 1^{o}C/min to 6^{o}C/min while it showed a broad-peak crystal phase in higher heating-rate ranges of 7-10^{o}C/min for the first heating.

Introduction

The bent-core liquid crystal (LC) was first reported in 1929 by Vorlander [1, 2]. After a long time of intendance, bent-core LCs attracted intense attention when Niori et al reported ferroelectric properties in this kind of materials [3-5]. Several hundreds of bent-core compounds have been synthesized and their mesomorphic properties have been characterized with polarized optical microscopy (POM) and differential scanning calorimetry (DSC). Among those documented bent-core LCs, some of them consist of five benzene rings while the others have composed of four, six and seven benzene rings [6-13]. To study the influence of structure variations on the transition temperatures, bent-core compounds with different central cores, with different linking groups in the wings of the molecules, and with varying length of the terminal alkyl tails have been studied extensively. However, none of the documented bent-core liquid crystals contains three benzene-ring cores [5], and no one reported that three-ring bent-core liquid crystals can form mesophases at the deferent heating rates.

In this work, we synthesized a three-ring bent-core LC compound N,N-bis (4-propoxybenzylidene)benzene-1,3-diamine. Crystal-banana phase transition properties of the synthesized compound are investigated with DSC and POM, respectively. Our results indicate that the bent-core LC N,N-bis (4-propoxybenzylidene)benzene-1,3-diamine exhibits different phase-transition properties at the different heating rate.

Experiments

A bent-core LC compound N,N-bis (4-propoxybenzylidene)benzene-1,3-diamine were synthesized in our laboratory. Analytical reagents m-phenyldiamine, 4-propoxybenzaldehyde, 1,3-dicyclohexylcarbodiimide and dimethylaminopyridine were provided by Alfa Aesar (Tianjin, China) and Sinopharm Chemical Reagents Co (Shanghai, China), respectively. All these reagents were used as received. In a 250ml bottom flask, m-phenylenediamine (0.012 mol) and 4-propoxybenzaldehyde (0.02 mol) were dissolved into the solvent of ethyl alcohol absolute (50 ml). After having been stirred with a magnetic bar at room temperature for 2 hours, brown precipitates were resulted. The target compound N,N-bis (4-propoxybenzylidene)benzene -1,3-diamine was purified by successive filtration, washing, recrystallization and drying and its yield was about 31%.

NMR spectra were obtained with a Brucker DRX500 spectrometer, and tetramethylsilane (TMS) was employed as an internal marker. Element analysis was carried out using a Vario EL III (Germany). Differential scanning calorimetry (Nanjing Dazhan, China) was used to characterize the phase

transition of the synthesized bent-core compound. The heating rate of the compound was ranged from 1 ^{o}C/min to 10^{o}C/min. Using capillary method, the synthesized compound was filled into LC cell whose spacing was measured to be about four micrometers. The POM textures of the bent-core LC compound were recorded with a polarized optical microscope (Aipha-Tech Ltd, China). A hot stage was used to regulate the temperature of the bent-core LC compound.

The spectroscopic data were listed below for the three-benzene-ring bent-core compound N,N-bis (4-propoxybenzylidene)benzene-1,3-diamine: ^{1}HNMR (500 MHz, $CDCl_3$, δ/ppm): 8.50 (2H, s, 2CH=N), 7.00-7.65 (12H, 12Ar-H), 4.05 (4H, t, $2OCH_2$), 1.05 (6H, t, $2CH_3$), 1.13-1.94 (24H, $12CH_2$). Element analysis: calcalated for $C_{38}H_{48}N_2O_4$, C 76.48, H 8.11, N 4.69, O 10.72; found C 75.50, H 8.84, N 5.57, O 10.09.

Results and discussion

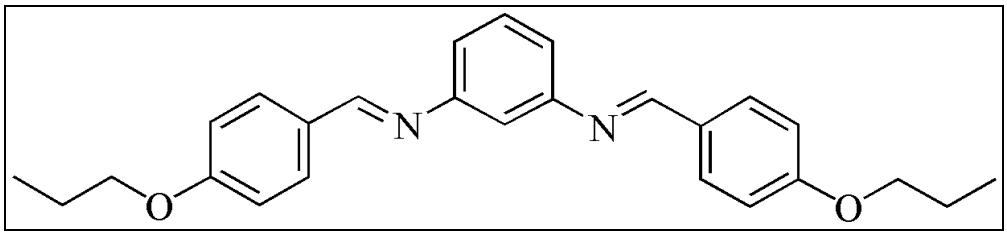

Figure 1. Molecular structure of the bent-core compounds N,N-bis (4-propoxybenzylidene)benzene-1,3-diamine.

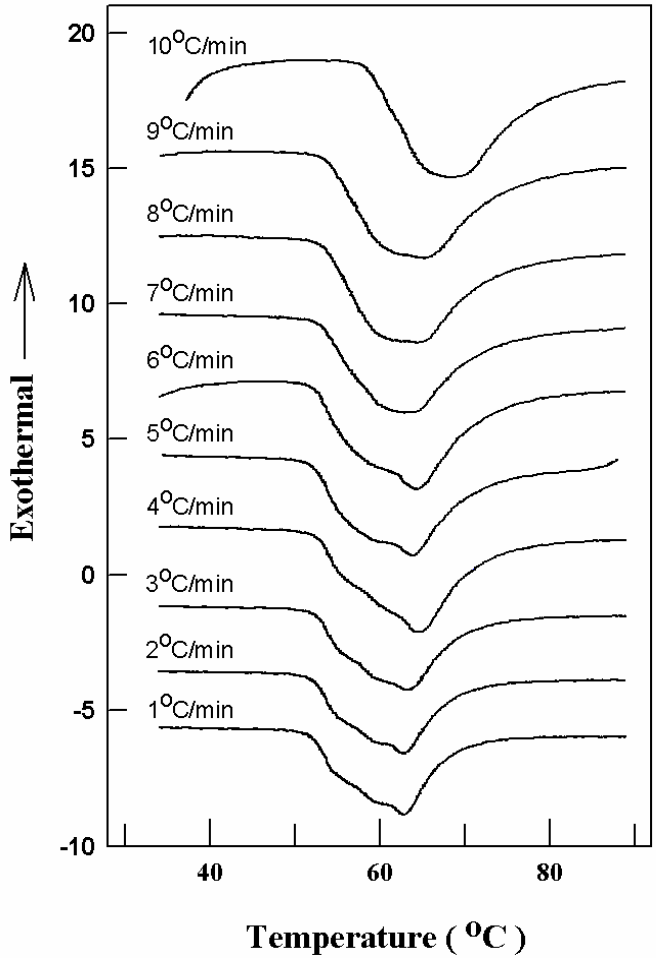

Figure 2. DSC graphs of the bent-core compound N,N-bis (4-propoxybenzylidene) benzene-1,3-diamine in the heating-rate ranges of 1-10^{o}C/min.

The molecular structure of the bent-core compound N,N-bis (4-propoxybenzylidene)benzene -1,3-diamine is shown in Fig. 1. This LC molecular structure contains phenylene groups as connecting units between the aromatic rings while their bent cores have a Schiff base where the alkyl tails are connected to the bent core through an ether linkage.

To check whether the three-ring bent-core compound exhibits mesophases, we characterized the bent-core compound with DSC. Figure 2 depicts the DSC curves of the bent-core compound for the first heating. As shown in Fig. 2, the synthesized bent-core compound containing 3 carbon atoms in terminal alkyloxy chain exhibited the completely undivided multi-phases in the heating-rate ranges of 1-6^{o}C/min for the first heating while the bent-core compound showed broad-peak crystal phase in the heating-rate ranges of 7-10^{o}C/min. Whether the three-ring bent-core compound exhibits mesophases depend mainly on the heating rates of the bent-core LC. The phase-transition properties in the deferent heating rates were also evidenced by our observations with polarized optical microscope.

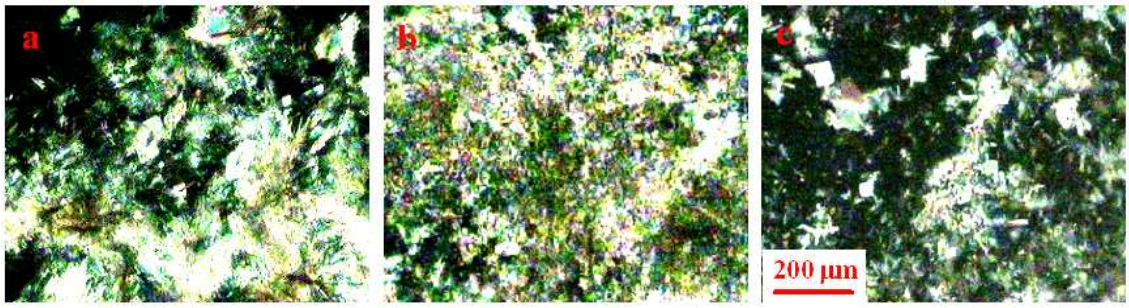

Figure 3. Polarized optical micrographs of the bent-core compound N,N-bis (4-propoxybenzylidene)benzene-1,3-diamine when it experienced the first heating in the heating-rate ranges of 1-6^{o}C/min.

Figure 3 shows photographs of the bent-core compound N,N-bis (4-propoxybenzylidene)benzene -1,3-diamine observed by polarized optical microscope when it experienced the first heating in the heating-rate ranges of 1-6^{o}C/min. As shown in Fig. 3, the synthesized bent-core compound N,N-bis (4-propoxybenzylidene)benzene-1,3-diamine exhibits the gathered particle-like textures in the heating-rate ranges of 1-6^{o}C/min for the first heating.

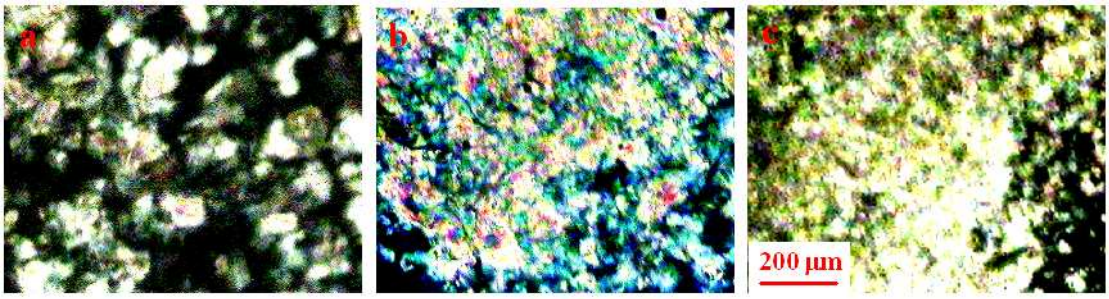

Figure 4. Polarized optical micrographs of the bent-core compound N,N-bis (4-propoxybenzylidene)benzene-1,3-diamine when it experienced the first heating in the heating-rate ranges of 7-10^{o}C/min.

Figure 4 shows photographs of the bent-core compound N,N-bis (4-propoxybenzylidene) benzene-1,3-diamine observed by polarized optical microscope when it experienced the first heating in the heating-rate ranges of 7-10^{o}C/min. From polarized optical micrographs shown in figure 4, the bent-core compound N,N-bis (4-propoxybenzylidene)benzene-1,3-diamine shows particle-like gathered crystal textures in the heating-rate ranges of 7-10^{o}C/min for the first heating. Obviously, our results are identical to the result characterized by differential scanning calorimetry in Fig. 2.

Conclusion

A three-ring bent-core compound N,N-bis (4-propoxybenzylidene)benzene-1,3-diamine was synthesized and characterized with differential scanning calorimetry and polarized optical microscopy. It exhibited the completely undivided multi-phases in the heating-rate range of 1-6^{o}C/min for the first heating while it showed crystal phase in the heating-rate range of 7-10^{o}C/min.

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