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Synthesis of a New Ferroelectric Liquid Crystalline Compound with Ester Linkage

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SYNTHESIS OF A NEW FERROELECTRIC LIQUID CRYSTALLINE COMPOUND WITH ESTER LINKAGE

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Abstract: A new ferroelectric liquid crystalline compound, 4-(2-butyloxycarbonyl)-Phenyl-4-(4- dodecyloxycarbonyl) benzoate (6), has been synthesized by esterification method. The DSC and optical microscopy studies reveals that the compound exhibit ferroelectric and smectic phases at lower and higher temperatures respectively. The NMR & IR spectral studies have also been carried out to verify the ester linkage.

INTRODUCTION

Chirality in liquid crystals have been shown increasing importance with respect to both their fundamental scientific significance and their applicability to electrooptic application[1,2]. In order to increase the range of the ferroelectric phase with high spontaneous polarization, we tried to synthesize various kinds of optically active compounds useful for chiral dopents[3]. In the present investigation we synthesized 4-(2-butyloxycarbonyl)-{phenyl}-4-(4-dodecyloxycarbonyl)benzoate(6), which is exhibiting ferroelectric phase in the reasonable range of temperature. The chiral centre used here is 2-butanol. We introduced three esterification linkages in

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the molecule. One of the main reason for the increase of spontaneous polarization[4] is to hinder the motion of the chiral centre. The material synthesized at present exhibit ferroelectric phase(Sc*) and highly ordered phase S_G*. DSC, polarizing microscopy, NMR and IR studies have been carried out to identify the different phases.

EXPERIMENTAL

DSC thermogram is taken using Perkin-Elmer DSC-2 instrument facility avilable at Raman Research Institute, Bangalore. The DSC trace as shown in fig (1) illustrate that, the specimen exhibit smectic G* phase, chiral smectic phase and smectic A phase (Cr 63°C - S_G* 92°C - Sc* 171°C - S_A - Iso) sequentially. Several experimental studies have been performed with various heating rates.

OPTICAL TEXTURE STUDIES

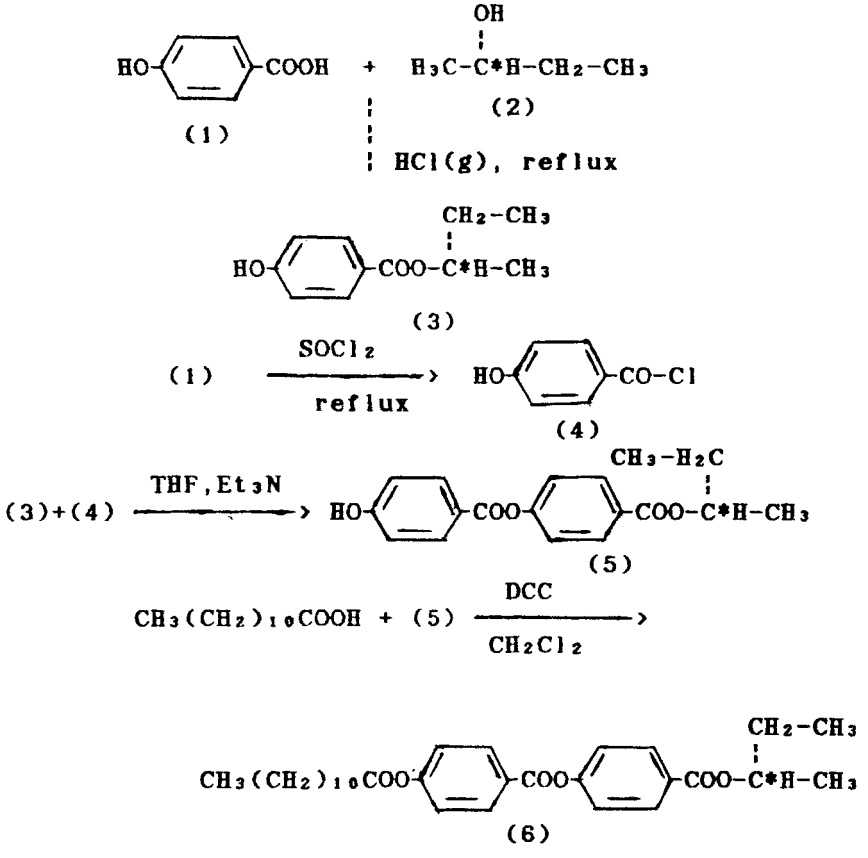
The specimen is taken in the form of thin film between the glass and coverslip and by using the Leitz polarizing microscope the transition temperature of the various phases were measured and it is found that the transition temperature measured by optical and DSC methods agrees very well. When the specimen is cooled from isotropic phase, focal conic textures are observed at 171°C which is the characteristics of S_A phase as shown in fig2(a). On further cooling the specimen stripes are developed on the fans of the focal conics. This texture corresponds to chiral smectic phase as shown in fig2(b). Finally this texture change over to S_G* phase at 92°C.

The measurements like spontaneous polarization, pitch and viscocity are under progress.

RESULT AND DISCUSSIONS

Synthesis

The compound described in this paper was prepared as shown in the following scheme:



a) 2-butyloxycarbonyl-4-hydroxy benzene

It was prepared according to the method given in[5].

b) Synthesis of 4'-(3-butyloxycarbonyl)-phenyl-4-hydroxy benzoate

Mixture of 4-hydroxy-(3-methyl propyl) benzoate and Et_3N in dry THF is slowly added to p-hydroxy benzoyl chloride while stirring at room temperature for 16 hours. The formed triethyl ammonium chloride was removed by filtration and the mixture was concentrated by distilling

under reducing pressure. It is further purified by column chromatography using chloroform as eluent to give (5).

c) Synthesis of 4-(2-butyloxycarbonyl)-phenyl-4-(4-dodecyloxycarbonyl)benzoate

4-(2-butyloxycarbonyl)-phenyl-4-(4-dodecyloxy carbonyl)benzoate was obtained by esterification of lauric acid and (5) in presence of DCC in anhydrous dichloromethane.

TECHNIQUES

The identification of products was carried out by the usual spectroscopic methods: IR spectrum as shown in fig(3) was recorded on Perkin-Elmer 399B spectrometer and ^1H NMR spectrum was obtained in CDCl3 on Hitachi Perkin-Elmer with TMS as an internal references and are expressed as δ values. The IR spectrum of the ester (6) showed absorptions at 1750 and 1700 cm^{-1} due to the aliphatic and aromatic ester linkage respectively. It also showed peak at 1640 cm^{-1} due to aromatic ring system and no absorption at 3300 to 3500 cm^{-1}. This indicates that the ester is free from -OH group. The structure is further confirmed by NMR studies. The observations of NMR peaks at δ 7.2 (d) and δ 7.8 (d) due to the para di substituted benzene derivative showed the linearity of the molecule. The other datas are included in the experimental section with detail. The purity of all products was checked by thin-layer chromatography.

^1H NMR($CDCl_3$): δ 0.9 (bt, 3H); 1.1-1.5 (bm, 18H); 1.6-2.0 (bm, 8H); 2.26-2.47 (t, 2H); 3.85-4.15 (m, 1H); 7.2 (d, 4H, ArH); 7.8(t, 4H, ArH).

IR(Nujol): 2960, 2840, 1750, 1700, 1640, 1420, 1350 cm^{-1}

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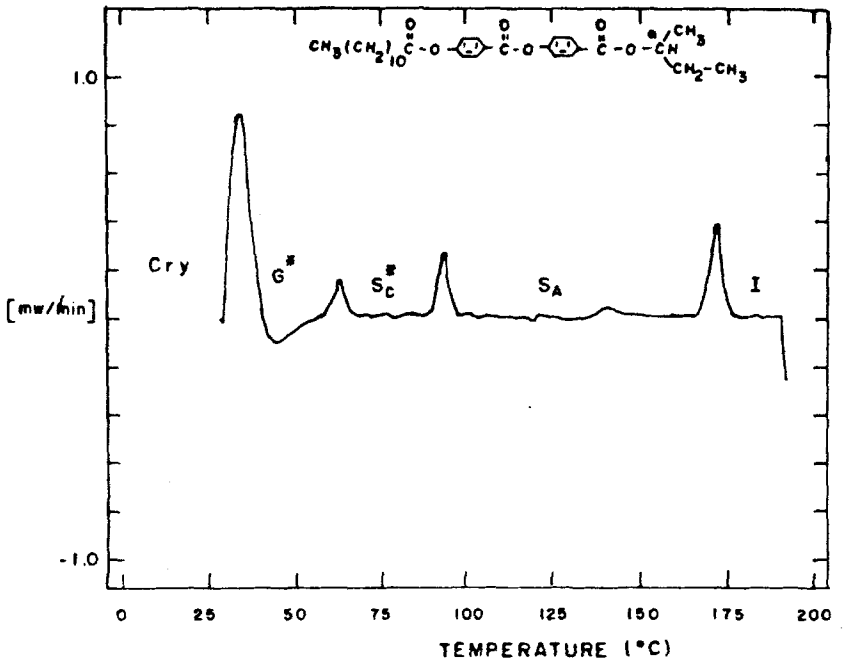

FIGURE 1 DSC Thermogram of the compound (6)

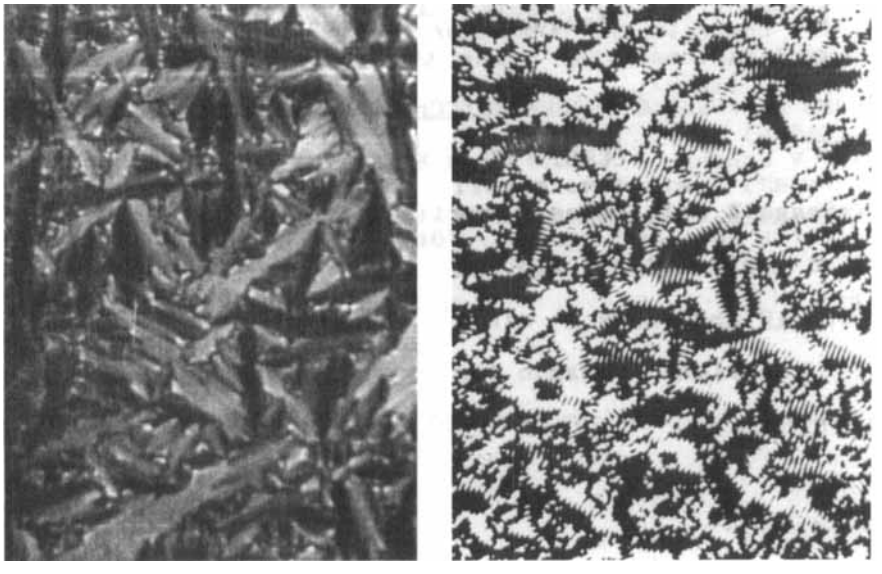

FIGURE 2 Microphotographs of a) S_A phase (150 X) b) Sc* phase (185 X)

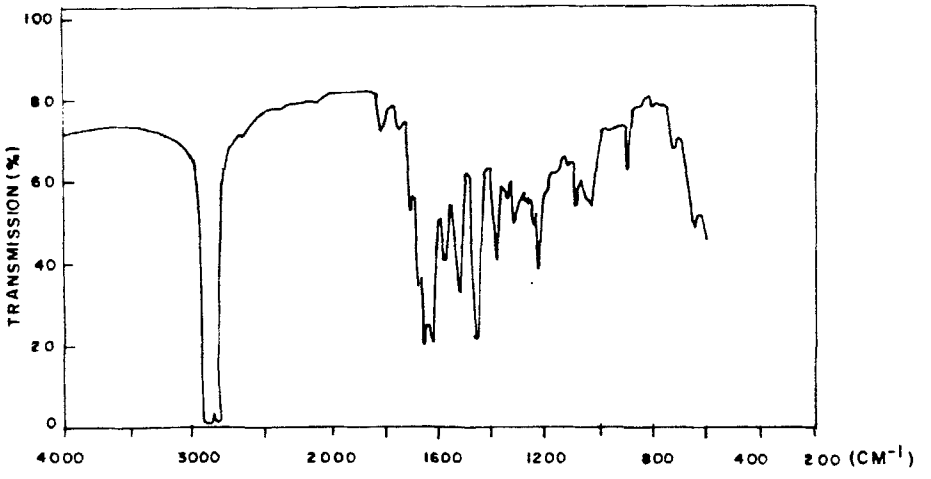

FIGURE 3 IR Spectrum of compound (6)

their valuable discussion. Prof Nagappa thanks DST, Government of India for financial support.

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