PREPARATION OF FLUORO-SUBSTITUTED LIQUID CRYSTALS CONTAINING AZOBENZENE MOIETIES

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DECLARATION

I hereby declare that this dissertation is based on my original work, except for quotations and summaries each of which have been fully acknowledged.

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ABSTRACT

The objective of this study was to synthesize liquid crystals compounds with fluoro-substituted and containing azobenzene moieties. The starting material, ethyl-4-aminobenzoate was undergone diazonium coupling reaction with 2-fluorophenol to give 4-(4-hydroxy-3-fluorophenylazo) ethyl benzoate. The second step of the reaction was Williamson ether synthesis on the diazonium salt to give 4-(4-hexyloxy-3-fluorophenylazo) ethyl benzoate. This compound was then hydrolyzed to yield 4-(4-hexyloxy-3-fluorophenylazo) benzoic acid. The final step was the esterification reaction of carboxylic acid with resorcinol, DCC and DMAP. The final compound formed was a bent-shape liquid crystal, 1,3-phenylene-bis-[4-(4-hexyloxy-3-fluorophenylazo) benzoate]. The structure and characteristic of the intermediates and final compound were confirmed by spectroscopic analysis with FT-IR, ^{1}H NMR and DSC. FT-IR spectrum for intermediate and final compound indicated the presence of functional groups included alkyl, aryl, ester, ether, carboxylic acid, amines etc. ^{1}H NMR spectrum showed the molecular structure of the final compound but with some impurities. Thermogram DSC showed the phase transition temperature of crystalline to mesophase (Cr-M) at 62.68°C and mesophase to isotropic (M-I) at 138.60°C in endothermic while in exothermic isotropic to measophase (I-M) and mesophase to crystal (M-Cr) phase appeared at 130.34°C and 59.90°C, respectively. These analyses had showed that the compound synthesized were liquid crystals but a the pure compound as projected.

PENYEDIAAN HABLUR CECAIR PENGGANTIAN FLUORO YANG MENGANDUNGI KUMPULAN BERFUNGSI AZOBENZENE

ABSTRAK

Objektif bagi penyelidikan ini adalah untuk mengsintesis suatu hablur cecair yang mengandungi florin dan kumpulan berfungsi azobenzena. Bahan permulaan iaitu etil-4-aminobenzoat menjalani tindak balas penduaan garam diazonium dengan 2-florofenol untuk menghasilkan 4-(4-hidrosi-3-florofenilazo) etil benzoate. Langkah kedua adalah penghasilan eter melalui tindak balas Williamson untuk menghasilkan 4-(4-heksilosi-3-florofenilazo) etil benzoate. Kemudian bahan ini menjalani hidrolisis untuk mendapatkan asid benzoik 4-(4-heksilosi-3-florofenilazo). Langkah terakhir dalam sintesis adalah tindak balas pengesteran asid karbosilik dengan kehadiran resorcinol, DCC dan DMAP. Kompaun akhir yang terhasil adalah 1,3-fenil-bis-[4-(4-heksilosi-3-florofenilazo) benzoat]. Struktur dan sifat kompaun pertengahan serta kompaun akhir dikenalpasti melalui analisis spektroskopi FT-IR ^1H NMR dan DSC. Spectrum FT-IR menunjukkan kehadiran kumpulan berfungsi (karbonil, ester, eter, alkyl, aril dll). Spektra ^1H NMR menunjukkan struktur molekul berdasarkan kedudukan proton tetapi tidak tulen kerana kehadiran bahan asing. Termogram DSC menunjukkan suhu perubahan fasa hablur dengan fasa mesogen (Cr-M) pada 62.68°C dan fasa mesogen dengan fasa isotropic pada 138.60°C dalam endotermik manakala untuk eksotermik fasa isotropic dengan fasa mesogen dan fasa hablur adalah 130.34°C dan 59.90°C masing-masing. Melalui analisis yang dijalankan, dapat dikenalpasti bahawa bahan yang disintesis merupakan hablur cecair tetapi tidak tulen seperti yang dijangkakan atas kehadiran bahan-bahan asing.

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Sm	Smectic phase
SmA	Smectic A phase
SmC	Smectic C phase
N	Nematic phase
N_D	Nematic discotic phase
$SmCP_A$	Lamellar antiferroelectric B_2 phase
SmC_SP_A	Racemic B_2 phase
SmC_AP_A	Chiral B_2 phase
ň	Director
Cr	Crystalline phase
M	Mesophase
I	Isotropic phase

CHAPTER 1

INTRODUCTION

1.1 Research Background

States of matter were classically thought in three phase where existed in solid, liquid, and gas. Actually, there are certain organic materials do nc have a single solid or liquid translation because they shows other intermediate states that are now known as liquid crystalline or mesomorphic phases.

The term liquid crystal designates a state of matt :r that is intermediate between the solid crystalline and the ordinary (isotropic) liquid phase. Liquid crystals are classically defined those orientationally ordered phases that occur between the breakdown of positional/transitional order on melting a solid, and the breakdown of orientational order on melting to a liquid (Demus *et al.*, 1998). Liq uid crystals flow like ordinary liquids where adopt the shape of the container besides exhibit anisotropic properties as solid crystals (Granbmaier, 1975).

Gases and liquids are isotropic because of their physical properties are the same in every direction within phase. Crystalline solids have a high degree order and crystal is anisotropic because it do depend on direction where cry tals are crack in one direction more easily (Silberberg, 2003). Thermotropic liquid crystal substance can pass from disordered liquid crystal through a series of distinct liquid crystal phases to an ordered crystal through a decrease in temperature while lyotropic substance can undergo similar changes through an increase in concentration (Silberberg, .003).

According to Demus *et al.* (1998), the development of liquid crystals may be divided into three phase where the first it was discovered by Friederich Reinitzer, a botanist who observed the colored phenomena occurring in melts of cholesteryl acetate and cholesteryl benzoate (Reinitzer, 1888). The second phase was the contribution of Vorlander and his groups in Halle to the growing number of compound to form liquid crystal phases by some are showing three different mesophases. Conventional thermotropic liquid crystals are formed by anisometric molecules (mesogen) that are either rod-shaped (calamitic) or disc-shaped (discotic) (Yelamaggad *et al.*, 2004). The third phase was development where field theory of nematic phase, focusing on London dispersion forces as the attractive interaction amongst molecules and upon the order parameter (Demus *et al.*, 1998).

Recently, Vill (1997) estimated that the known compounds of crystalline are more than 72,000. Liquid crystals are well established in basic research and development for application and commercial use because its represent a state intermediate between threedimensional solid and ordinary liquids (Demus *et al.* 1998). Many different tools and techniques are made used from these complexes physical properties of liquid crystals. According to Demus *et al.* (1998), liquid crystals play an important role in materials science for organic chemist in order to investigate the connection between physical properties and chemical structure.

Until now, liquid crystals used in Liquid Crystal Display (LCD), watches, CD-player and other electronic devices because it is thinner, lighter, and draw less power. It will only be said that the future prospects for liquid crystals look healthy, but they will only be maintained so if fundamental research by scientists of imagination is adequately funded to enable the exploration of new ideas, new aspects, and new possibilities, because history does demonstrate that many of the discoveries significant for applications and technology derive from sound basic science or a sound knowledge of established basic science (Demus *et al.*, 1998).

A survey of liquid crystals composed of bent-shaped molecules revealed that compounds with azo linkages have hardly been studied (Prasad, 2001). According to Wu *et al.* (2003), the azobenzene containing materials have potential technological applications as in high-density optical data storage, optical image processing, dynamic holography, optical computing, and pattern recognition. Azo compound are thermally very stable and are attractive from point of view of studying photoinduced effects (Prasad, 2001).

Recently, there are many studies about fluoro-substituted liquid crystals and azo linkages separately. Fluorine is the most electronegative element, the second smallest element next to hydrogen and the atom that can form a stronger bond with carbon than hydrogen (Kitazume & Takashi, 1998). Fluorine had been used as a lateral substituent on the central as well as outer phenyl rings for modifying mesomorphic properties (Reddy & Sadashiva, 2003). Fluorine can sometimes add an unexpected quality to organic molecules resulting from its special properties (Kitazume & Takashi, 1998).

This research concentrated on both azo linking group and fluorine lateral substitution. The synthesis product was fluoro-substituted liquid crystals containing azobenzene moieties.

1.2 Research Objectives

- To prepare fluoro-substituted liquid crystals containing azobenzene moieties.
- To determine functional groups of the compound using Fourier Transform Infrared (FT-IR) spectrometer.
- To determine the molecular structure of the compound using Nuclear Magnetic Resonance (NMR) spectrometer.
- To determine the phase transition of the compound by Differential Scanning Calorimetry (DSC).

1.3 Scope of the Study

This research focused on the organic synthesis of liquid crystals and the determination of liquid crystals properties. The procedures involved in the process were refluxes, hydrolysis, recrystallization and etherification using specific chemicals (ethyl-4-aminobenzoate, DCC, DMAP, resorcinol etc) and basic chemicals in lab (acetone, dichloromethane, methanol etc).

Instrument used are that available in industrial chemistry lab (DSC etc) and Institute of Borneo Tropical Research lab (FTIR and ^{1}H NMR) for molecular determination.

CHAPTER 2

LITERATURE REVIEW

2.1 Definition of Liquid Crystal

Liquid crystals are also called crystalline liquids, mesophases or mesomorphic phases and the compounds with mesomorphic properties may be called "mesogenic" (Stegemeyer & Guest, 1994). According to Grabmaier (1975), liquid Crystal can be define as "condensed fluid phases with spontaneous anisotropy" because it flow like isotropic liquid crystal phases and exhibit anisotropic properties as solid crystals (Figure 2.1)

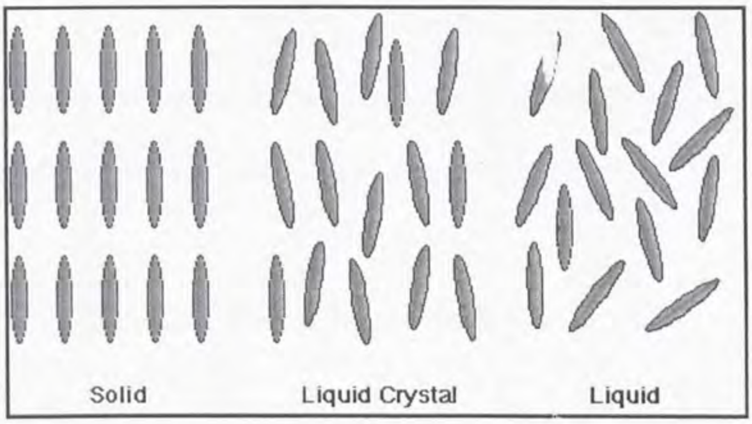

Figure 2.1 Arrangements of molecules in the solid, liquid crystal and liquid phase.

The position of liquid crystals is between the solid and the isotropic state. Molecular crystals possess a long-range order of the positions of the molecules and in addition, a long-range order of their orientations. With increasing temperature at the melting point, the crystal transforms to the isotropic liquid (Stegemeyer & Guest, 1994).

Crystalline solid ⟷ isotropic liquid

According to Demus *et al.* (1998), melting process can be characterized in three ways: first, an initial breakdown in order with the molecules oscillating or rotating rapidly about one or more axes; second, a collapse of the long-range positional ordering of the molecules to give a state where the molecules have short-range position order (1.5-70 nm) but yet they still have long-range orientational order; and third, a disruption in the short-range and long-range order to produce a completely disordered liquid.

2.2 Thermotropic Liquid Crystal

Thermotropic liquid crystals are of interest both from the standpoint of basic research and for applications in electro-optic displays, temperature and pressure sensors (Bahadur, 1990). At higher transition temperature, the turbidity suddenly vanishes giving way to the clear appearance of the ordinary liquid (Grabmaier, 1975).

Thermotropic mesophase formed by heating a solid or cooling an isotropic liquid, or by heating or cooling a thermodynamically stable mesophase (Demus *et al.*, 1998). Generally thermotropic liquid crystals formed by prolate (calamitic) molecules or oblate (discotic) molecules (http://cmt.dur.ac.uk). Thermotropic liquid crystalline phases may occur in either pure compounds or also mixtures (Stegeme,/er and Guest, 1994).

2.3 Molecular Structure of Liquid Crystal

Molecular structure of liquid crystal derived from the shape of the constituting molecules, which can generally categorize as calamitic (rod-like), discotic (disc-like), and sanidic (board-like).

2.3.1 Calamitic Liquid Crystal

Classical liquid crystals that occur in rod-like molecules and it is the greatest part of research. A typical calamitic mesogen consists of a rigid core unit (aromatic or alicyclic), ensuring the anisotropic character, flexible side chain which provide stabilizing effects within the liquid crystal phases (Whinnery *et al.*, 1977). The nature of the core and the side chains are widely influenced the physical properties of calamitic mesogen (Whinnery *et al.*, 1977). Figure 2.2 shows the classical structure of calamitic liquid crystals that consists of wings, rings and bridges.

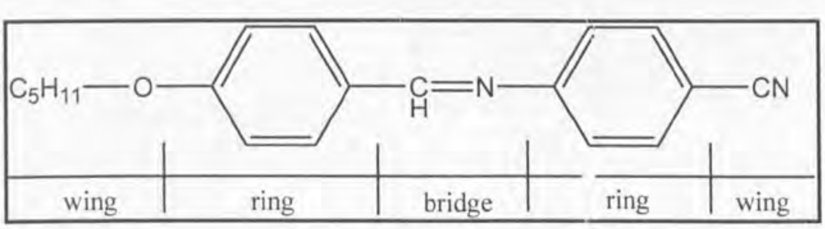

Figure 2.2 Classical structures of calamitic liquid crystals containing wings, rings and bridge.

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