

**PREPARATION OF LIQUID CRYSTALLINE
POLYMERS WITH ESTER TERMINAL**

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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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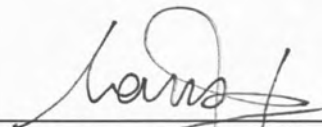
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ABSTRACT

The objective of this study was to prepare liquid crystalline polymers with ester terminal and determined the functional groups, chemical structure, phase transition and melting point of the product with Fourier Transform Infrared Spectroscopy (FT-IR); Nuclear Magnetic Resonance Spectroscopy (NMR); Differential Scanning Calorimetry (DSC) and capillarial melting point apparatus. The experiments involved three main steps and ethyl-4-aminobenzoate was used as a starting material. The initial step was diazonium salt coupling reaction with phenol at pH 10 to obtain 4-(4-Hydroxyphenylazo) ethyl benzoate, which have a melting point in the range 125 °C to 144 °C. The next reaction was Williamson ether reaction with bromopropene to obtain 4-(4-Alloxyloxy-phenylazo) ethyl benzoate with a melting point in the range 85 °C to 105 °C. Subsequently, this compound was undergone additional polymerization reaction where the compound is dissolved into dry Tetrahydroferam (THF) and degassed using Nitrogen with catalytic Azobisisobutyronitrile (AIBN) under 60°C to yield the desired liquid crystalline polymer with ester terminal. The final products have a melting point in the range 125 °C to 135 °C. All compounds synthesized in each steps shown C-H in alkyl, carbonyl group (C=O), C=C in aromatic, C-O. The chemical structure of the final products was confirmed by NMR. Besides that, two obvious peak of T_m (melting temperature) and T_c (crystallization temperature) was obtained through the DSC thermogram of final products.



**PENYEDIAAN POLIMER HABLUR CECAIR YANG MENPUNYAI KUMPULAN
BERFUNKSI ESTER PADA HUJUNG RANTAI POLIMER**

ABSTRAK

Objektif kajian ini ialah menghasilkan bahan hablur cecair yang mempunyai ciri-ciri mesogenik serta menunjukkan kumpulan ester pada hujung rantai polimer dan seterusnya menentukan kumpulan berfungsi, molecular structure, fasa perubahan dan takat lebur & takat beku produk akhir dengan Fourier Transform Infrared Spectroscopy (FT-IR); nuclear magnetic resonance spectroscopy (NMR); Differential Scanning Calorimetry (DSC) dan capillary melting point examiner. Experiment ini melibatkan tiga langkah utama dan penghasilan bahan ini dimulakan dengan etil-4-aminobenzoat. Langkah permulaan ialah tindak balas penduaan garam diazonium dengan fenol pada pH 10 untuk menghasilkan 4-(4-hidroksil-fenolazo) etil benzoat yang mempunyai 125 °C to 144 °C. Bahan yang diperolehkan seterusnya menjalankan tindak balas penhasilan eter Williamson dengan bromopropena untuk memperolehi 4-(4-alkoxy-fenolazo) etil benzoat yang mempunyai takat lebur 85 °C to 105 °C. Sebatian ini kemudiannya mengalami tindak balas pempolimeran, iaitu dilarutkan dengan Tetrahydroferum (THF) dan seterusnya digaskan dengan Nitrogen dengan kehadiran mangkin Azobisisobutyronitrile (AIBN) pada suhu 60°C. Hasil akhir ini mempunyai takat lebur 125 °C to 135 °C. Didapati bahawa sebatian yang dihasilkan pada setiap langkah mempunyai kumpulan berfungsi seperti C-H in alkyl, kumpulan karboksil group (C=O), C=C in aromatik, C-O. Molecular structure pada produk terakhir telah dikenalpasti oleh NMR. Daripada terogram DSC, terdapat dua puncak yang jelas telah didapati, salah satu puncak ialah T_m (suhu melebur) dan satu lagi ialah T_c (suhu membeku) bagi produk terakhir.



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LIST OF SYMBOLS

LC	Liquid Crystal
MC-LCPs	Main Chain Liquid Crystalline Polymers
SC- LCPs	Side Chain Liquid Crystalline Polymers
S_A	Smectic A phase
S_B	Smectic B phase
S_C	Smectic C phase
S^*_C	Achiral Smectic C phase
$^{\circ}\text{C}$	Degree Celsius
mg	milligram
mmol	millimol
ml	milliliter
%	percentage
H	Hydrogen atom
C	Carbon atom
N	Nitrogen atom
O	Oxygen atom
R	Aliphatic group
=	Double bond (pie bond)
-	Single bond (Sigma bond)
Δ	Molecular Polarizability
δ	Chemical shift
T_{N-1}	N-isotropic liquid transition temperature
T_g	Glass transition temperature
Ph	Phosphorus atom
CN	Cyano group
NO_2	Nitrogen dioxide group
Cl	Chloride atom
Br	Bromide atom



CH ₃	Methyl group
F	Fluorine atom
KBr	Potassium Bromide
THF	Tetrahydrofuran
AIBN	Azobisisobutyronitrile
FT-IR	Fourier Transform Infrared Spectroscopy
DSC	Differential Scanning Calorimetry
NMR	Nuclear Magnetic Resonance Spectroscopy
UK	United Kingdom
USA	United States of America



CHAPTER 1

INTRODUCTION

1.1 Preface

Liquid crystalline polymers (LCPs), as a new class of macromolecular materials that distinguish from the ordinary polymers, have great potential with the qualities of high strength, stiffness and chemical resistance, good dimensional stability and low linear thermal expansion coefficient (Chen *et al.*, 2005). It is very useful in developing high performance micro-optical, electrical, mechanical devices and nano components due to their anisotropy and rich mesophase features (He and Shen, 2006).

Liquid crystalline polymers combine the properties of polymers with those of liquid crystals. These "hybrids" show the same mesophases characteristic of ordinary liquid crystals, yet retain many of the useful and versatile properties of polymers. According to the chemical standpoint, there are two types of liquid crystalline polymers such as lyotropic liquid crystalline polymers, e.g. Kevlar based on poly (*p*-phenyleneterephthalamide) (Chen *et al.*, 2005) and thermotropic liquid crystalline polymers, e.g. Vectra based on wholly aromatic polyester (Chen *et al.*, 2005). The



thermotropic liquid crystalline polymer is most affected by changing temperature and can be made into three-dimensional structure by conventional processing, such as injection molding (Chen *et al.*, 2005). This makes thermotropic liquid crystalline polymers a unique material for wide range of applications.

Traditionally, thermotropic liquid crystalline polymers can be broadly divided into two classes of compounds; there are main-chain liquid crystalline polymers (longitudinal) and side-chain liquid crystalline polymers (comb) (Noel, 1992). More recently other variants have appeared; these are combined liquid crystalline polymers which are hybrid between main chain liquid crystalline polymers and side chain liquid crystalline polymers.

Main chain liquid crystalline polymers consist of repeating mesogenic monomer units that are incorporated into the main-chain of a polymer backbone. These stiff regions along the chain allow the polymer to orient in a manner similar to ordinary liquid crystals, and thus display liquid crystal characteristics. The incorporation of a rigid structure of mesogenic units to be the main-chain polymer gives rise to an increase in melting temperature, high modulus and high strength (Chen *et al.*, 2005).

Conversely, side chain liquid crystalline polymers are formed when the mesogens are connected as side chains to the polymer backbone by a flexible 'bridge' or 'spacer'. Side chain liquid crystalline polymers have three major structural components: the backbone, the spacer and the mesogen. These three major structural components are the



significant to realize the desired thermotropic behavior, process ability and end-use of liquid crystals polymers (Simmonds, 1992).

The liquid crystalline polymer in this present work has the characteristic of thermotropic, side chain liquid crystalline properties and have ester group as terminal. The significant of this liquid crystalline polymer is that it exhibits various liquid crystalline phases as a function of temperature; therefore it has great potential applications especially in the field of optics and optoelectronics (Khoo, 1995).

A leading motivation of side chain liquid crystalline polymers is the unique possibility of combining the functionality of conventional low molar mass liquid crystals with the properties of macromolecules. This is mainly possible due to the linking of the different mesogenic groups to the polymer main chain through flexible alkyl spacers of varying length (Hvilsted, *et al.*, 1995).

1.2 Research Objectives

The objectives of this research are:

- To prepare liquid crystalline polymers with ester group as the terminal.
- To determine the functional groups of the product using Fourier Transform Infrared Spectroscopy (FTIR) and identify chemical structure of product using Nuclear Magnetic Resonance Spectroscopy (NMR).



- To measure phase transition of product using Differential Scanning Calorimetry (DSC).
- To determine the melting point of each compounds synthesized by using capillarial melting point apparatus.

1.3 Scope of Research

This project is going to focus on the organic synthesis of liquid crystalline polymers with ester group as the terminal and determination of mesophase properties. The compounds synthesized will be characterized by Fourier Transform Infrared Spectroscopy (FT-IR) to determine the functional group of compounds. Besides that, the chemical structure of products will be determined by Nuclear Magnetic Resonance Spectroscopy (NMR). Differential Scanning Calorimeter (DSC) will be used to measure the phase transition for the product. The melting point of each compounds synthesized will be recorded by capillarial melting point apparatus.



CHAPTER 2

LITERATURE REVIEW

2.1 Discovery of Liquid Crystals.

According to Reinitzer in 1888, a botanist Friedrich, is the first person to describe the liquid crystals (LC) compounds from his study of substance cholesteryl benzoate. The next year, another German scientist, Otto Lehmann in 1889 coined the name 'liquid crystals'. Lehmann in 1889, confirmed the experimental results of Reinitzer, which was important, since some people did not believe Reinitzer. However, Reinitzer did not agree with Lehmann; he maintained that the 'liquid crystals' was wrong and constitutes a contradiction (Brostow, 1992).

After more than two decades of working on liquid crystal, there was another person named Vorlander in 1923, realized that liquid crystalline polymers must exist also. From his studies, he tried to determine what will be happens to molecules when one makes them longer and longer; and will the liquid crystalline state disappear when liquid crystal become longer and longer. According to the Brostow 's experience in 1992, there



was no limit to the state by chain elongation, unless the substances could not melt any more without decomposition and could not be seen under a microscope. Thus, some German researchers already had polymer liquid crystals in their hands in the nineteenth century (Brostow, 1992).

2.2 Liquid Crystals

Liquid crystals are known as mesomorphic state compounds that having long-range orientational order (e.g. crystalline solid) and exhibit either partial positional order or complete positional disorder (e.g. isotropic liquid) (Demus *et al.*, 1999). In short, liquid crystals is an intermediate structure which flows like a liquid but still possesses the anisotropic physical properties similar to crystalline solids (Khoo, 1995) as shows in Figure 2.1. Most liquid crystals are organic substances, which often called mesogenic or mesogen, and the various phases in which they could exist are termed mesophases (Khoo, 1995).

The most well known and widely studied mesophase are thermotropic. However, polymeric mesophases have getting more and more concerned recently. As a function of temperature, or depending on the constituents, concentration, substituents and others important criteria, these liquid crystals exist in many mesophases such as nematic, smectic, and cholesteric (Khoo, 1995). Liquid crystals flows like nearly incompressible viscous fluids but still remain several features, especially optical, characteristic of crystals.



According to Vigna (1994), mesogenic will give rise to mesophases under specific circumstances such as definite temperature, concentration and pressure.



Figure 2.1 The average alignment of molecules for each phase (Anon, 2006).

Base on the geometrical structure of the molecules, the liquid crystals can be divided into several types such as: rod like molecules, disc like molecules, and lath like species. The “classical” liquid crystals are derived from rod like molecules and according to a modern proposal may be called also “calamitic”. At present, there are about 20,000 rod like mesogenic compounds, 15,000 of which compiled in the book with the title as *Flussige Kristalle in Tabellen II* (Bahadur, 1990).

Liquid crystals derived from disc like (or discotic) molecules were not known until it was discovered by Chandrasekhar, Sadashiva, and Suresh in 1977 (Demus *et al.*, 1990). Discotic mesogen is relatively flat, disc like or sheet shape molecules (Collyer, 1992). Recently, many different discotic chemical species are available, which may exist in several different molecular arrangements. Besides that, lath like species is the

intermediate between the rod like and the disc like molecules, e.g.: fused twins or phasmidic compounds. Generally, the twins (eventually oligomers) are the link between the monomeric and the polymeric liquid crystals (Demus *et al.*, 1990).

Due to the chemical standpoint, liquid crystals can be divided into lyotropic liquid crystals and thermotropic liquid crystals. Lyotropic liquid crystalline is formed by the dissolution of amphiphilic molecules of a material in a suitable solvent. It depends on both temperature and concentration. In converse, thermotropic liquid crystalline is formed by heating a solid or cooling an isotropic liquid. One of the most distinguishing properties of thermotropic liquid crystalline polymers is the ease in producing a uniform molecular orientation under elongational flow (Orifici and Valles, 1996).

2.3 Thermotropic Liquid Crystals

Thermotropic liquid crystal is formed by heating or cooling a thermodynamically stable mesophase; or in others word it is formed by heating a solid or cooling an isotropic liquid (Demus *et al.*, 1999). It exhibits various liquid crystalline phases as a function of temperature (Khoo, 1995). Because of these unit criteria, thermotropic liquid crystals are widely used; and extensively studied for their linear as well as nonlinear optical properties (Singh, 2002).

Thermotropic liquid crystals exist in dependence of the temperature in certain temperature intervals. Thermotropic liquid crystals which are stable at temperatures



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