Synthesis and Characterization of Banana-shaped Liquid Crystals

Chua Mei Chee (20828)

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Synthesis and Characterization of Banana-shaped Liquid Crystals

CHUA MEI CHEE (20828)

This project report is submitted in partial fulfillment of the Bachelor’s Degree of Science Honours in Resource Chemistry

Supervisor: Assoc. Prof. Dr. Zainab Ngaini

Resource Chemistry Programme
Department of Chemistry
Faculty of Resource Science and Technology
UNIVERSITY MALAYSIA SARAWAK
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Declaration

I hereby declare that no portion of this dissertation has been submitted in support of any application for another degree of qualification of this or any other university or institution of higher learning.

(Chua Mei Chee)
Resource Chemistry Programme
Department of Chemistry
Faculty of Resource Science and Technology
University Malaysia Sarawak
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<td>DABCO</td>
<td>1,4-Diazabicyclo[2.2.2]octane</td>
</tr>
<tr>
<td>DCM</td>
<td>Dichloromethane</td>
</tr>
<tr>
<td>DSC</td>
<td>Different Scanning Calorimetry</td>
</tr>
<tr>
<td>FTIR</td>
<td>Fourier Transform Infrared</td>
</tr>
<tr>
<td>HCl</td>
<td>Hydrochloric acid</td>
</tr>
<tr>
<td>K$_2$CO$_3$</td>
<td>Potassium carbonate</td>
</tr>
<tr>
<td>KOH</td>
<td>Potassium hydroxide</td>
</tr>
<tr>
<td>MEK</td>
<td>Methyl Ethyl Ketone</td>
</tr>
<tr>
<td>MeOH</td>
<td>Methanol</td>
</tr>
<tr>
<td>NaH</td>
<td>Sodium hydride</td>
</tr>
<tr>
<td>NMR</td>
<td>Nuclear Magnetic Resonance</td>
</tr>
<tr>
<td>POM</td>
<td>Polarizing Optical Microscopy</td>
</tr>
<tr>
<td>SmCG</td>
<td>Triclinic symmetry</td>
</tr>
<tr>
<td>TBAB</td>
<td>Tetrabutylammonium bromide</td>
</tr>
<tr>
<td>TBAI</td>
<td>Tetrabutylammonium iodide</td>
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Abstract

Two series of compounds based on chalcone were synthesized. The initial step
involves the reaction of 4-alkoxyacetophenones 3a-e from 4-hydroxyacetophenones with alkyl
halides. The resulting ketones were condensed with 4-hydroxybenzaldehyde to form hydroxyl chalcone.
These compounds further reacted with naphthaloyl dichloride to form
5a-e. The synthesized compounds were characterized by 1H and
31P NMR, and Fourier Transform Infrared (FTIR). The
properties of these compounds were characterized using polarizing optical microscopy (POM) and
differential scanning calorimetry (DSC).

naphthaloyl dichloride, liquid crystal
Synthesis and Characterization of Banana-Shaped Liquid Crystal

Student’s name: Chua Mei Chee

Final report submitted as partial requirement of the Course Final Year Project 2 (STF3013)

Supervisor’s name: Associate Prof. Zainab Ngaini

Programme name: Resource Chemistry

Department name: Department of Chemistry

Universiti Malaysia Sarawak

Abstract

A series of banana-shaped compounds based on chalcone were synthesized. The initial step involved preparation of alkoxyacetophenone 3a-c from 4-hydroxyacetophenone with alkyl bromide using Williamson ether synthesis. 4-Alkoxyacetophenone 3a-c undergoes Claisen-Schmidt condensation with 4-hydroxybenzaldehyde to produce hydroxyl chalcone 4a-c. The hydroxyl chalcone further reacted with isophthaloyl dichloride to form banana-shaped compound 5a-c. The synthesized compound was characterized by $^1$H and $^{13}$C Nuclear Magnetic Resonance (NMR), and Fourier Transform Infrared (FTIR). The liquid crystal property was characterized using polarizing optical microscopy (POM) and different scanning calorimetry (DSC).

Key words: chalcone, isophthaloyl dichloride, liquid crystal

Abstrak

Satu siri sebatian berbentuk pisang berdasarkan kalkon telah disintesis. Langkah awal melibatkan sintesis 4-alkoksiasetofenon 3a-c daripada 4-hidroksiasetofenon dan alkil bromida dengan menggunakan sintesis eter Williamson. 4-Alkoksiasetofenon 3a-c mengalami kondensasi Claisen-Schmidt dengan 4-hidroksibenzaldehyde untuk menghasilkan kalkon hidroksil 4a-c. Hidroksi kalkon ditindakbalaskan dengan isofataloil klorida untuk menghasilkan sebatian berbentuk pisang 5a-c. Sebatian hasil sintesis dicirikan oleh $^1$H dan $^{13}$C Nuklear Resonansi Magnetik (NMR), dan Fourier Transform Infrared (FTIR). Sifat kristal cair dicirikan dengan menggunakan mikroskop polarisasi optik (POM) dan kalorimetri imbasan berbeza (DSC).

Kata kunci: kalkon, isofataloil klorida, kristal cair
1.0 Introduction

1.1 Chalcones

Chalcone 1 is also known as 1, 3-diphenyl-2-propene-1-one. The general structure of chalcone is shown in Figure 1. They consist of two aromatic rings which are linked by a three carbon α, β-unsaturated carbonyl system. Chalcone is abundant in edible plants and is considered as intermediate precursors in the biosynthesis of flavonoids and isoflavonoids. Chalcones have been reported to possess various biological activities such as antimicrobial, anti-inflammatory, antiplatelet, antimalarial, anticancer, antioxidant, antitubercular, antihyperglycemic, and inhibition of tyrosinase (Solankee et al., 2009).

Apart from biological activities, other studies were also reported on compounds containing chalcone moiety having photochemical properties. For instance, Choi et al. (2001) who reported on chalcone derivatives having photodimerization and photopolymerization behavior with photoinitiator to exhibit good liquid crystal alignment upon near UV exposure (Makita et al., 1998).

![Figure 1: Molecular structure of chalcone](image_url)
1.2 Liquid crystal

Liquid crystals are intermediate phase which exhibits features from both solid and fluid state. The intermolecular force of liquid crystals in solid phase is not equal in all direction. This is because molecular motion is increased and overcomes weaker forces as the material is heated (Shakhashiri, 2007). However, its molecules remain hold by the stronger forces. This produces regular arrangement of molecules in some direction and random arrangement of molecules in other direction. The layers can also slide over one another (Shakhashiri, 2007). This molecular mobility form the fluidity characteristic of a liquid (Shakhashiri, 2007). Thus, they possess some typical properties of liquid and solid.

Liquid crystals are classified into thermotropic and lyotropic (Wang, 2007). The lyotropic liquid crystals consist of amphiphilic molecules, that is polar and non-polar parts form liquid crystal phases (Elliott & Brian, 2006). Its transition depends on the concentration of solvent (Wang, 2007). On the other hand, the molecules in thermotropic liquid crystals consist of a rigid core and a flexible tail (as illustrated in Figure 2) (Wang et al., 1993). The transitions involving thermotropic liquid crystals are effected by changes in temperature (Wang, 2007). Thermotropic liquid crystals can be subclassified into nematic, cholesteric, and smetic (Andrienko, 2006).

![Figure 2: General structure of thermotropic liquid crystal](image-url)
Liquid crystal has only been used for electronically driven displays in 25 years ago. The high switching speed combined with a large change in the effective birefringence and constant optical axis of the liquid crystal materials provide useful features in various applications, such as liquid crystal television, beam steering, spatial light modulators, and a variety of other applications (Jakli & Antal, 2008).
1.2.1 Banana-shaped or Bent-core Liquid Crystals

Banana-shaped liquid crystals often contain 1, 3-disubstituted phenyl 2 (as shown in Figure 3). Banana-shaped liquid crystal had been synthesized in the early 20th century. Before banana-shaped had been synthesized, the ferroelectricity only can appear in fluid phase of liquid crystalline which are formed by a tilted arrangement of enantiomerically enriched chiral molecules in layers (Meyer et al., 1975). In 1983, Prostand Baros predicted that achiral molecules contain switching behavior. This property fabricating the molecules could order in polar layers. Niori et al. (1996) were first reported that smectic phase of banana-shaped liquid crystal with achirality could have ferroelectric properties. Jaklic et al. (2001) experimentally proved that ferroelectric properties can exist in smectic phases with triclinic symmetry (SmCG) of banana-shaped liquid crystal without chiral molecules. In addition, Shimbo et al. (2006) found that banana-shaped liquid crystal has advantages such as in plane switching, continuous gray level, and high contrast ratio (3000:1) when applied to liquid crystal display mode.

Figure 3: Molecular structure of banana-shaped liquid crystal (Takezoe&Takanishi, 2006)
1.3 Objectives

Based on the literature precedent, the objectives of this study are

1. to synthesize 4-alkoxyacetophenone via etherification process (Scheme 1).

\[
\text{H}_3\text{C} - \text{C} = \text{O} + \text{R-Br} \xrightarrow{\text{K}_2\text{CO}_3, \text{TBAI}} \text{H}_3\text{C} - \text{C} = \text{O} - \text{OR} \quad \text{OR} = \text{CH}_3\text{OH, C}_7\text{H}_7\text{OH, C}_13\text{H}_{29}\text{OH}
\]

\text{Scheme 1: Proposed reaction of etherification}

2. to synthesize hydroxylated chalcone via Claisen-Schmidt condensation process (Scheme 2).

\[
\text{H}_3\text{C} - \text{C} = \text{O} - \text{OR} + \text{H} - \text{C} - \text{CH} - \text{OH} \xrightarrow{\text{MeOH, Reflux}} \text{H}_3\text{C} - \text{C} = \text{O} - \text{OH} \quad \text{OR} = \text{CH}_3\text{OH, C}_7\text{H}_7\text{OH, C}_13\text{H}_{29}\text{OH}
\]

\text{Scheme 2: Proposed reaction structure for synthesizing hydroxyl chalcone via Claisen-Schmidt condensation process}
3. to synthesize banana-shaped liquid crystal by esterification between chalcone derivatives and benzene-1, 3-dicarbonyl chloride (as shown in Scheme 3).

\[
\begin{align*}
\text{O} & \quad \text{C} & \quad \text{O} \\
\text{OH} & \quad \text{Cl} & \quad \text{NaOH} & \quad \text{THF} & \quad \text{5a: R=C,H}_2 & \quad \text{5b: R=C,H}_2 & \quad \text{5c: R=C,H}_2 \\
& & & & \\
\end{align*}
\]

Scheme 3: Proposed reaction structure for synthesizing banana-shaped liquid crystal through esterification process

4. to characterize the synthesized compounds using \(^1\)H & \(^{13}\)C NMR and FTIR.

5. to test for liquid crystal properties by POM and DSC which are available in USM.
2.0 Literature Review

2.1 Synthesis of Chalcones

Chalcones can be synthesized via Claisen–Schmidt condensation in the presence of base or acid as catalyst. Prasad et al. (2007) have synthesized series of chalcones 8 via Claisen-Schmidt condensation of appropriate acetophenones 6 with appropriate aromatic aldehydes 7 in the presence of KOH as base. The reaction is shown in Scheme 4. The obtained yield was 82%. There are other bases such as NaOH, or Ba(OH)_2 used to perform aldol reaction. The base catalyzed reaction has some disadvantages such as reaction reciprocity and long reaction time (Al-Issa & Andis, 2005).

\[
\text{KOH} \quad \text{Room Temp., 24h}
\]

R_1=OH, R_2=R_4=R_5=H, R_3=R_6=Cl

Scheme 4: Synthesis of chalcone by Claisen–Schmidt condensation

In 2003, Eddarir et al. utilized Suzuki coupling reaction in the synthesis of chalcone 13. The reaction takes place either between phenylboronic acids 9 and cinnamoyl chlorides 10 or between benzoyl chlorides 11 and phenylvinylboronic acids 12 in the presence of palladium catalyst (as shown in Scheme 5). This method gives better yield with less unwanted side product.
Chalcone can also be synthesized in the absence of solvent. This was reported by Al-Issaand Andis (2005) on the synthesis of chalcone 16 from acetophenone derivative 14 and aromatic aldehyde derivative 15 (as shown in Scheme 6). This reaction was carried out under microwave irradiation without solvent in the presence of catalytic amount of Lewis acid. They utilized microwave energy to activate organic reaction. This had been proven by the reaction time. The time brought down from hours to seconds and the yield was improved.

Besides that, the use of catalyst in aldol condensations under solvent-free conditions could also give good yield for chalcones and accelerate reaction rate. In 2007, Kumar and Akanksha reported that zirconium chloride is a safe, economical, and highly efficient catalyst for the aldol condensation of aldehyde derivative 17 and ketone derivative 18.
under solvent free conditions (as shown in Scheme 7). The yields that obtained for substitute chalcones 19 were ranging from 70-90 % with the use of 20 mol % of zirconium chloride as catalyst. Another research reported that iodide was also an efficient catalyst (Wang & Zeng, 2009). They utilized 10 mol % of iodide as catalyst to produce a satisfactory yield ranging from 83 % to 95 %. Regarding on the research, iodide could be considered as a more efficient catalyst than zirconium chloride. Moreover, synthesis of chalcone using either iodine or zirconium chloride as catalyst has advantages. Both of these catalysts are environmental friendly and have shorter reaction time.

\[
\begin{align*}
\text{Scheme 7: Synthesis of chalcone in the presence of ZrCl}_4 \text{catalyst} \\
&\text{a: ZrCl}_4 (20 \text{ mol\%}) \\
&\text{b: neat/dry DCM at room temperature} \\
&R_1, R_2, R_3, R_4, R_5: \text{substituents}
\end{align*}
\]
3.2 Williamson ether synthesis

Williamson reaction is the most common and convenient method in the preparation of ether from an organohalide and an alcohol in the presence of a base. This reaction was developed by Alexander Williamson in 1850. Different types of solvent, catalyst and base are utilized in Williamson method could affect the percentages of yield.

In 2006, Holden prepared Nerolin 22 through Williamson method. The reaction carried out between β-naphthol 20 with methyl iodide 21 in the presence of polar solvent, methanol (as shown in Scheme 8). In this reaction, KOH was used as a base to afford nerolin in 77%.

Other studies on Williamson reaction was also reported by Kadkin et al. (2007) in the preparation of phenolic ether 22. 4-Dodecyloxyacetophenone 25 was prepared from 4-hydroxyacetophenone 23 with dodecylbromide 24 in the presence of dry potassium carbonate (K₂CO₃) under refluxing polar aprotic solvent, acetone (as shown in Scheme 9). The yield obtained was 95% which was much higher compared to yield of nerolin 22.

In 2008, Pandey et al. reported on the use of TBAB as an efficient catalyst for etherification reaction. The reaction was carried out between polyfunctional aromatic compounds 26 with different alkyl halides in the presence of K₂CO₃ (as shown in Scheme...
The R can be served as aromatic substituent or aliphatic substituent. This preparation is more economical and gives higher yield up to 90%.

Scheme 10: Etherification of phenol in polyfunctional aromatic compounds

Etherification could also be performed under solvent-free condition. For instance, Cao et al. (2000) who synthesized ether from phenols with dimethyl sulfate or alkyl halides in the presence of solid-liquid phase transfer catalyst PEG400 under solvent-free system. This preparation is environmental friendly, and gives excellent yields (above 86%) and purity for aromatic ethers.
2.3 Esterification

Traditional method in the preparation of esters is from the reaction of carboxylic acid with alcohol by the removal of water (Claff, 1998). This reaction normally cannot be applied to the synthesis of esters from phenols due to the acidity of phenol (Nezhad et al., 2007). The leaving group property of acyl halide is reactive towards nucleophilic substitution reaction when reacts with a phenol. This method is more expensive but readily carried out on a small scale (McMurry, 2000). Recently, Baghernejad (2010) reported on the used of 1,4-Diazabicyclo[2.2.2]octane (DABCO) as a solid catalyst in the preparation of aliphatic and aromatic ester 27 from reaction of acid chloride derivative 28 with alcohol derivative 29 (as shown in Scheme 11). DABCO is a highly reactive, easy handle and non-toxic catalyst. The reaction had been conducted in excellent yield and short reaction time.

\[
\begin{align*}
\text{R}_1\text{C}^\text{Cl} & \quad + \quad \text{R}_2\text{OH} \quad \xrightarrow{\text{DABCO}} \quad \text{R}_1\text{C}^\text{O}\text{R}_2 \\
28 & \quad 29 & \quad 30
\end{align*}
\]

**Scheme 11: Esterification in the presence of DABCO**

In order to produce two ester linkages of compound, diacid chloride is commonly use to react with hydroxyl compound. Cho et al. (2003) has synthesized mesogenic compunds with two esters unit, 33. Ester linkages were formed by the reaction from isophthaloyl dichloride 31 with 1-(4-hydroxyphenyl)-4-octylbutadiyne 32 under stirring tetrahydofuran (THF) at room temperature (as illustrated in Scheme 12). In this reaction, NaH was used as base.
Scheme 12: Synthesis of mesogenic compounds with two esters unit

Besides, Novotna et al. (2006) also carried out esterification of isophthaloyl dichloride to synthesis of banana-shaped molecules 36 which possess ferroelectric-like behavior. This banana-shaped molecule 36 was prepared by esterification of mesogenic phenols 35 with isophthaloyl dichloride 34 under refluxing dichloromethane (DCM) (as illustrated in Scheme 13). Besides, pyridine was used as base. This reaction produced banana-shaped molecules 36 and compound 37. In this reaction, compound 37 was produced in majority. However, a banana-shaped molecule 36 was produced in lower yield.

Scheme 13: Esterification between diacid chloride and mesogenic phenol, M