

PREPARATION AND CHARACTERIZATION COMPOSITE
OF HYBRID ORGANIC-INORGANIC MATERIAL OF
BITHOPHENONE AND TETRAETHOXYSILANE

ZURAIDAH BINTI HASSIM

FACULTY OF SCIENCE AND TECHNOLOGY
KOLEJ UNIVERSITI SAINS DAN TEKNOLOGI MALAYSIA
2005

1100038668

Perpustakaan
Kolej Universiti Sains Dan Teknologi Malaysia (KUSTEM)

LP 22 FST 6 2005



1100038668

Preparation and characterization composite of hybris organic-inorganic material of bithiophene and tetraethoxysilane



PERPUSTAKAAN

KOLEJ UNIVERSITI SAINS & TEKNOLOGI MALAYSIA
21030 KUALA TERENGGANU

1100038668

Lihat sebelah



PREPARATION AND CHARACTERIZATION COMPOSITE OF HYBRID ORGANIC-INORGANIC MATERIAL OF BITHIOPHENE AND TETRAETHOXYSILANE

By

Zuraidah binti Hassim

**Research Report submitted in partial fulfillment of
the requirements for the degree of
Bachelor of Science (Chemical Sciences)**

**Department of Chemical
Faculty of Science and Technology
KOLEJ UNIVERSITI SAINS DAN TEKNOLOGI MALAYSIA
2005**

1100038668

M. A. S. H. Q.

**JABATAN KIMIA
FAKULTI SAINS DAN TEKNOLOGI
KOLEJ UNIVERSITI SAINS DAN TEKNOLOGI MALAYSIA**

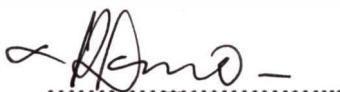
**PENGAKUAN DAN PENGESAHAN LAPORAN
PROJEK PENYELIDIKAN I DAN II**

Adalah ini diakui dan disahkan bahawa laporan penyelidikan bertajuk:

**PREPARATION AND CHARACTERIZATION COMPOSITE OF HYBRID
ORGANIC-INORGANIC MATERIAL OF BITHIOPHENE AND
TETRAETHOXYSILANE**

Oleh Zuraidah binti Hassim, No. Matrik UK 5095 telah diperiksa dan semua pembetulan yang disarankan telah dilakukan. Laporan ini dikemukakan kepada Jabatan Kimia sebagai memenuhi sebahagian daripada keperluan memperolehi Ijazah Sarjana Muda Sains (Sains Kimia), Fakulti Sains dan Teknologi, Kolej Universiti Sains dan Teknologi Malaysia.

Disahkan oleh:



Penyelia Utama:

Nama: Dr. Nanok Kancono

Cop Rasmi:

Dr. Nanok Kancono

Pensyarah

Jabatan Sains Kimia

Fakulti Sains dan Teknologi

Kolej Universiti Sains dan Teknologi Malaysia

21030 Kuala Terengganu.

Tarikh: **26/04/2005**

Penyelia Kedua

Nama: En. Mohamad Hussin bin Hj. Zain

Cop Rasmi: **MOHAMAD HUSSIN HAJI ZAIN**

Pensyarah

Jabatan Sains Kimia

Fakulti Sains dan Teknologi

Kolej Universiti Sains dan Teknologi Malaysia

21030 Mengabang

Kuala Terengganu.

Tarikh: **26/04/2005**

Ketua Jabatan Kimia

Nama: Prof. Madya Dr. Ku Halim bin Ku Bulat

Cop Rasmi: **PROF. MADYA DR. KU HALIM KU BULAT**

Ketua

Jabatan Sains Kimia

Fakulti Sains dan Teknologi

Kolej Universiti Sains dan Teknologi Malaysia

21030 Kuala Terengganu.

Tel: 09-6683257

Tarikh: **26/04/2005**

ACKNOWLEDGEMENTS

First of all, thanks to God for giving me the strength, confidence and patience in preparing this research report. I would like to express my deepest and warmest sense of thanks and appreciation to my honourable project supervisor, Dr Nanok Kancono for his valuable assistance, constructive, criticisms and inspiring guidance. I am also grateful to my co-supervisor , Mr. Mohamad Hussin bin Hj Zain for his moral support, patience and guidance to me.

Sincere thanks to all lecturers in Department of Chemistry and those who had contributed to the success of this project in one way or another. Thanks are also extended to all Laboratory Assistants in Chemistry Department, especially to Mr. Jamaluddin, Mrs. Ashbah, Mr. Tarmizi, Mr. Muzaffiq, Ms. Noriayati and Mr. Maswadi for their favorable help.

Finally, I would like to express my deepest gratitude to my parent, Mr. Hassim bin Awing and Mrs. Zamun binti Mat Zin, my sibling (Mr. Zumizam, Ms. Zuzilawati and Ms Zuraini Asilah) and my beloved friends especially my dear Mr. Shahril bin Hussain for their endless encouragement, patience and sacrifices, which had help me in understanding and completing this research project.

TABLE OF CONTENTS

APPROVAL FORM	ii
ACKNOWLEDGEMENTS	iii
TABLE OF CONTENTS	iv
LIST OF TABLES	vii
LIST OF FIGURES	viii
LIST OF SCHEMES	x
LIST OF ABBREVIATIONS	xi
LIST OF SYMBOLS	xii
LIST OF APPENDICES	xiii
ABSTRACT	xiv
ABSTRAK	xv
CHAPTER	
1 INTRODUCTION	
1.1 Composite	1
1.2 Hybrid Organic-Inorganic	3
1.2.1 Class 1	4
1.2.2 Class 2	6
1.3 History of Liquid Crystals	7
1.4 Definition of Liquid Crystal	9
1.5 Classification of Liquid Crystals	10
1.5.1 Thermotropic Liquid Crystals	11
1.5.2 Lyotropic Liquid Crystals	14
1.6 Polymer Liquid Crystal	14
1.6.1 Main Chain Liquid Crystal Polymer	15
1.6.2 Side Chain Liquid Crystal Polymer	17
1.7 Structural Features of Liquid Crystal Polymer	19
1.7.1 Polymer Backbone	19
1.7.2 Spacer Linkage	20
1.7.3 Mesogenic Group	21
1.8 Properties of Liquid Crystals Polymer	22
1.8.1 Application of Liquid Crystals	22

1.8.2	Liquid Crystal Thermometer	22
1.8.3	Mood Ring	23
1.8.4	Liquid Crystal Display (LCD)	23
1.9	Method used to identify LC phase	23
1.10	Objectives of the Study	25

CHAPTER

2 LITERATURE REVIEW

2.1	History of Thiophene	26
2.2	Synthesis of Sol-gel Bis-(trimethoxysilyl)terthiophene and Phenomenon Study of their Birefringence	29
2.3	Bithiophene as Starting Monomers for Polythiophene Synthesis	31
2.4	Trialkoxysilyl Mono-, Bi-, and Terthiophenes as Molecular Precursors of Hybrid Organic-Inorganic Materials	32
2.5	Structural and Electrical Characterization of Processable Bis-Silylated Thiophene Oligomers	33

CHAPTER

3 METHODOLOGY

3.1	Materials	35
3.2	Characterization	
3.2.1	Nuclear Magnetic Resonance (NMR)	36
3.2.2	Ultraviolet (UV) Spectrometer	37
3.2.3	Infrared (IR) Spectroscopy	38
3.3	Preparation	
3.3.1	Preparation of bithiophene	39
3.3.2	Preparation of 5,5'-dibromobithiophene	40
3.3.3	Preparation composite of hybrid organic-inorganic material from bithiophene and tetraethoxysilane	41

CHAPTER

4	RESULT AND DISCUSSION	
4.1	Preparation	43
4.2	Result and Discussion	44
4.2.1	bithiophene	44
4.2.1a	Ultraviolet Spectrum (UV)	44
4.2.1b	Infrared Spectrum (IR)	46
4.2.1c	NMR Spectrum of bithiophene	49
4.2.2	Composite of hybrid organic-inorganic material from bithiophene and tetraethoxysilane	53
4.2.2a	Ultraviolet Spectrum (UV)	53
4.2.2b	Infrared Spectrum (IR)	56
4.2.2c	NMR Spectrum of Composite of Hybrid Organic-Inorganic Material from Bithiophene and tetraethoxysilane	59

CHAPTER

5	CONCLUSION AND RECOMMENDATION	
5.1	Conclusion	64
5.2	Recommendation for future work	65

REFERENCES	66
APPENDICES	69
CURRICULUM VITAE	71

LIST OF TABLES

Table	Page
1.1 Typical Backbone of Side Chain Liquid Crystal Polymer	20
1.2 Examples of Linkage Components	21
2.1 Physical and physicochemical properties of thiophene	28
3.1 List of chemicals	35
4.1 UV Data for bithiophene	45
4.2 Infrared Characteristic Bands of bithiophene	47
4.3 NMR Data Analysis of bithiophene	50
4.4 UV Data for Composite of Hybrid Organic-Inorganic Material from bithiophene and tetraethoxysilane	55
4.5 Infrared Characteristic Bands of Composite of Hybrid Organic-Inorganic Material from bithiophene and tetraethoxysilane	57
4.6 $^1\text{H-NMR}$ Data Analysis of Composite of Hybrid Organic-Inorganic Material from bithiophene and tetraethoxysilane	61

LIST OF FIGURES

Figure	Page
1.1 Illustrates the Development of Liquid Crystal	11
1.2 Nematic Phase	12
1.3 Smectic phase	13
1.4 Cholesteric phase	14
1.5 The structure of the MCPLCs	15
1.6 Example of the first type of MCPLCs	16
1.7 The example of the second type of MCPLCs	16
1.8 The mesogenic unit which incorporated for polymer as side chain	17
1.9 The structure of the SCPLCs	17
1.10 Typical repeating unit in a SCPLCs	18
1.11 Schematic Representation of a Model Liquid Crystalline Polymer	19
1.12 Typical components in a mesogenic group	22
2.1 The Structure of thiophene	29
4.1 UV Spectrum for bithiophene	45
4.2 Infrared Spectrum Characteristic Bands of bithiophene	48
4.3 $^1\text{H-NMR}$ Spectrum of bithiophene	51
4.4 $^{13}\text{C-NMR}$ Spectrum of bithiophene	52
4.5 UV Spectrum for composite of hybrid organic-inorganic material from bithiophene and tetraethoxysilane	55
4.6 Infrared Spectrum Characteristic Bands of composite of hybrid organic-inorganic material from bithiophene and tetraethoxysilane	58

4.7	¹ H-NMR Spectrum of Composite of Hybrid Organic-Inorganic Material from bithiophene and tetraethoxysilane	62
4.8	¹³ C-NMR Spectrum of Composite of Hybrid Organic-Inorganic Material from bithiophene and tetraethoxysilane	63

LIST OF SCHEMES

Scheme	Page
3.1 Synthesis Scheme of bithiophene	39
3.2 Synthesis Scheme of 5,5'-dibromobithiophene	40
3.3 Preparation of composite of hybrid organic-inorganic material from bithiophene and tetraethoxysilane	41
3.4 The formation of composite of hybrid organic-inorganic material from bithiophene and tetraethoxysilane	41

LIST OF ABBREVIATIONS

LCs	Liquid Crystals
LLCs	Lyotropic Liquid Crystals
TLCs	Thermotropic Liquid Crystals
LCPs	Liquid Crystal Polymers
SCLCPs	Side Chain Liquid Crystal Polymers
MCLCPs	Main ChainLiquid Crystal Polymers
TCNQ	Tetracyanoquinodimethane
FET	Field Effect Transistor
LCD	Liquid Crystal Display
UV	Ultraviolet
IR	Infrared
FTIR	Fourier Transform Infrared
NMR	Nuclear Magnetic Resonance
MP	Polarisation Microscope
MHz	Megahertz
XRD	X-Ray Diffraction
SEM	Scanning Electron Microscope
TEOS	Tetraethoxysilane
CDCl ₃	Deuterated chloroform
THF	Tetrahydrofuran
NBS	N-bromosuccinimide
MgSO ₄	Magnesium sulphate
HOAC	Acetic acid

LIST OF SYMBOLS

ϵ	molar absorption
A	total absorption
c	concentration in molar
L	cell length in cm
λ	wavelength
ppm	part per million

LIST OF APPENDICES

Appendix A	^1H -NMR for 2,5-bis(trimethoxysilyl)terthiophene	70
Appendix B	^{13}C -NMR for 2,5-bis(trimethoxysilyl)terthiophene	71

ABSTRACT

Preparation and characterization of hybrid organic-inorganic composite material of bithiophene and tetraethoxysilane were prepared from the reaction of 5,5'-dibromobithiophene with tetraethoxysilane in tetrahydrofuran. The precursor of the reaction, bithiophene was prepared from the decoupling of bromothiophene in tetrahydrofuran with NiCl_2 as catalyst. While 5,5'-dibromobithiophene compound was prepared from the reaction of bithiophene with N-bromosuccinimide (NBS) in the mixture of chloroform and acetic acid. The yield of the compound was tested by using the Infrared (IR) Spectrometry, Ultraviolet (UV) Spectrometer, ^1H and ^{13}C of Nuclear Magnetic Resonance (NMR).

ABSTRAK

Penyediaan komposit hibrid bahan organik-tak organik bithiophene dan tetraethoxysilane telah dilakukan dengan tindakbalas 5,5'-dibromobithiophene bersama tetraethoxysilane di dalam tetrahydrofuran. Bahan awal tindakbalas iaitu bithiophene telah disediakan dengan tindakbalas berganda bromothiophene di dalam tetrahydrofuran dan telah dimangkinkan oleh NiCl_2 . Sementara itu, 5,5'-dibromobithiophene telah disediakan melalui tindakbalas bithiophene dengan N-bromosuccinimide (NBS) dalam campuran kloroform. Hasil daripada sebatian yang telah diperolehi telah diuji dengan Spektrometer Inframerah (IR), Spektrometer Ultraviolet (UV), ^1H dan ^{13}C Nuklear Magnetik Resonans (NMR).