



شبكة المعلومات الجامعية  
التوثيق الإلكتروني والميكرو فيلم

# بسم الله الرحمن الرحيم



**HANAA ALY**



شبكة المعلومات الجامعية  
التوثيق الإلكتروني والميكروفيلم



# شبكة المعلومات الجامعية التوثيق الإلكتروني والميكروفيلم



**HANAA ALY**



شبكة المعلومات الجامعية  
التوثيق الإلكتروني والميكروفيلم

# جامعة عين شمس التوثيق الإلكتروني والميكروفيلم

## قسم

نقسم بالله العظيم أن المادة التي تم توثيقها وتسجيلها  
علي هذه الأقراص المدمجة قد أعدت دون أية تغييرات



## يجب أن

تحفظ هذه الأقراص المدمجة بعيدا عن الغبار



**HANAA ALY**

**Ain Shams University**  
**Faculty of Science**  
**Chemistry Department**



**“Preparation of polyurethane foam using bio-based polyhydroxyl oils and investigation of its effect on mortar pastes”**

Thesis submitted  
For the Degree of Doctor of Philosophy of Science  
(Chemistry)

by

**Ahmed Abdel Hamid Ma'moun Salman**

M.Sc. in Chemistry, Faculty of Science  
Ain Shams University, 2016

Under Supervision of

**Prof. Dr. El-Sayed Ahmed Soliman Abdel Aziz**

Prof. of Organic Chemistry,  
Chemistry Department, Faculty of Science, Ain Shams University

**Prof. Dr. El-Sayed Abdel Raouf Abdel Kader Nasr**

Prof. of Properties and Testing of Materials,  
Structural Engineering Department, Faculty of Engineering, Ain Shams University

**Associate Prof. Ahmed Adel Mahmoud Mahmoud**

Associate Prof. of Applied Chemistry,  
Physics and Mathematics Department, Faculty of Engineering, Ain Shams University

Cairo 2019



Approval Sheet

**“Preparation of polyurethane foam using bio-based polyhydroxyl  
oils and investigation of its effect on mortar pastes”**

Submitted by

**Ahmed Abdel Hamid Ma'moun Salman**

M.Sc. in Chemistry, Faculty of Science  
Ain Shams University, 2016

For the Degree of Doctor of Philosophy of Science  
(Chemistry)

**This thesis has been approved by :**

**Prof. Dr. El-Sayed Ahmed Soliman Abdel Aziz** .....

Prof. of Organic Chemistry,  
Chemistry Department, Faculty of Science, Ain Shams University

**Prof. Dr. El-Sayed Abdel Raouf Abdel Kader Nasr** .....

Prof. of Properties and Testing of Materials,  
Structural Engineering Department, Faculty of Engineering,  
Ain Shams University

**Associate Prof. Ahmed Adel Mahmoud Mahmoud** .....

Associate Prof. of Applied Chemistry, Physics and Mathematics  
Department, Faculty of Engineering, Ain Shams University

**Head of Chemistry Department**  
**Prof. Dr. Ayman Ayoub Abdel-Shafi**

**Ain Shams University**  
**Faculty of Science**  
**Chemistry Department**



### **Information about the researcher**

**Student name:** Ahmed Abdel Hamid Ma'moun Salman

**Scientific Degree:** B.Sc., 2010

M.Sc., 2016

**Faculty name:** Faculty of Science – Ain Shams University

**Graduation year:** 2010

**Granting year:**

**Head of Chemistry Department**  
**Prof. Dr. Ayman Ayoub Abdel-Shafi**

بِسْمِ اللَّهِ الرَّحْمَنِ الرَّحِيمِ

وَمَلِكًا لِّكَرِيمًا

وَكَأَن فُضِّلَ اللَّهُ عَلَيْكَ عَظِيمًا

# ACKNOWLEDGMENT

---

**First of all, I would like to thank god for every gift bestowed on me...**

I would like to extend sincere thanks to my advisor, **Prof. Dr. El-Sayed Ahmed Soliman** for providing the guidance necessary to complete this research and also for his constant encouragement, support and friendship which was the motivating force that kept work on my thesis in force until completion.

I would like to extend my sincerest appreciation to my advisor, **Prof. Dr. El-Sayed Nasr** for his guidance, continuous, valuable guidance and the investment, giving me the opportunity to be involved in such interesting research.

I would like to express my deepest thanks and appreciation to **Associate Prof. Ahmed Adel Mahmoud**, for his continuous advice, keen interest, valuable supervision and for his reviewing of the manuscript.

Also I would like to thank all members of the Chemistry Department, Faculty of Engineering, Ain Shams University for their great efforts.

Also I would like to thank my friend **Dr. Alaa Mohsen** who stood beside me during conducting this work.

This research was supported by **Royal Foam Co.** through the supply of chemicals and industrial experience required to fulfill the experimental program for this work.

Chemistry Lab. at the Faculty of Engineering Ain Shams University (ASU), Analysis help by chemistry Lab. at American University in Cairo

(AUC), National Institute for Standards (NIS) and Housing & Building National Research Center (HBRC) are highly appreciated.

Finally, I would like to deeply thank to my family, and appreciate the sincere help, support, and encouragement, especially in the difficult times, of my father, my mother to whom I dedicate the thesis.



# **Preparation of polyurethane foam using bio-based polyhydroxyl oils and investigation of its effect on mortar pastes**

**Ahmed Abdel Hamid Ma'moun Salman**  
**Master of Science, 2016**

## **ABSTRACT**

---

Polyurethane foams are versatile polymeric materials which find broad applications such as: thermal insulation, sound insulation, coating, adhesive, sealant and construction building industry.

Researchers are making great efforts to develop and improve their mechanical and thermal properties. The use of bio renewable resources for polyurethane manufacturing and polymer industry as a whole has become highly desirable for both economic and environmental reasons.

Synthesis of the polyurethane foam was involved two main reactions which are gelling and blowing reaction. The gelling reaction is related to generating of the urethane linkages which were created from isocyanate group and hydroxyl group. The blowing reaction is the reaction that creating of air bubbles entrapped inside the reactive mixture.

In this work vegetable oils polyhydroxyl compound were fully and partially used (25%, 50%, 75% and 100%) as renewable resource for the preparation of semi rigid polyurethane foam. The structure of prepared polyurethane foam was characterized by FTIR spectra. Also, the thermal stability of polyurethane samples was estimated using TGA apparatus. The morphological structure of polyurethane foam was investigated using scanning electron microscope. As well, properties like compression strength, tensile strength as well as density were determined.

Finally, the effects of the prepared polyurethane polymer on the properties of hardened cement mortar was investigated by measuring thermal resistance and sound absorption coefficient then compare the results with mixes containing 100% petroleum polyhydroxyl compounds.

## TABLE OF CONTENTS

<b>ACKNOWLEDGMENT</b>	
<b>ABSTRACT</b>	
<b>TABLE OF CONTENTS</b>	I
<b>LIST OF TABLES</b>	IV
<b>LIST OF FIGURES</b>	VI
<b>CHAPTER (1)</b> <b>INTRODUCTION</b>	<b>1</b>
1.1 Polyurethane foam	1
1.2 Study objectives	2
1.3 Planned Methodology	2
1.4 Thesis Organization	3
<b>CHAPTER (2)</b> <b>LITERATURE REVIEW</b>	<b>5</b>
2. A. Petroleum Based Polyurethane Foam	5
2. A.1 Polyol	5
2. A.1.1 Polyether Polyol	5
2. A.1.2 Polyester polyol	12
2. A.1.3 Other polyols	16
2. A.1.3.1 Polycarbonate polyol	16
2. A.1.3.2 Polyacrylate Polyols	18
2. A.1.4 Filled Polyols	20
2. A.1.4.1 Copolymer polyol	20
2. A.1.4.2 Polyurethane dispersion (PHD) polyols	23
2. A.1.4.3 Polyisocyanate polyaddition (PIPA) Polyols	24
2. A.1.5 Prepolymers	26
2. A.2 Isocyanates	27
2. A.2.1 Toluene Diisocyanate (TDI)	27
2. A.2.2 Diphenyl Methane Diisocyanate (MDI)	30
2. A.3 Chain Extenders	32
2. A.4 Catalyst	33
2. A.5 Water blowing agent	36
2. A.6 Silicon Surfactant	36
2. B Chemistry of Polyurethane Foam	38
2. B.1 Urea formation	39
2. B.2 Urethane formation	39

2. B.3 Allophanate Formation	40
2. B.4 Formation of Biurets	40
2. B.5 Formation of Uretidione and Isocyanurate	41
2. B.6 Formation of Amides	42
2. C Bio-based Polyurethane foam	43
2. C.1 Bio-based content replacement	43
2. C.1.1 Bio-based polyols	43
2. C.1.1.1 Castor oil	49
2. C.1.1.2 Soybean oil	52
2. C.1.1.3 Other bio based polyols	54
2.D. Chemistry of Cement	57
2.E. Energy saving materials	62
<b>CHAPTER (3)</b>	66
<b>MATERIALS AND EXPERIMENTAL PROGRAM</b>	
3.1 Materials	66
3.1.1 Characteristics Of Mortar Materials	66
3.1.1.1 Cement	66
3.1.1.2 Sand	67
3.1.1.3 Water	68
3.1.2 Characteristics of polyurethane foam materials	69
3.1.2.1 Polyol	69
A. Petroleum polyol	69
B. Bio- based polyol	69
3.1.2.2 4,4 Methylene diphenyl diisocyanate (MDI)	70
3.1.2.3 Catalysis	70
3.2 Experimental Program	73
3.2.1 Program of Research Work	73
3.2.2 Polyurethane foam preparation	74
3.2.3 Bio based Polyurethane foam preparations	75
3.2.4 Polyurethane Foam Characterizations	76
3.2.4.1 Fourier Transformed Infrared Spectroscopy (FTIR)	76
3.2.4.2 X-Ray Diffraction (XRD)	77
3.2.4.3 Thermogravimetric Analysis (TGA/DTG)	78
3.2.4.4 Scanning Electron Microscope (SEM)	78
3.2.5 Mechanical Properties of Polyurethane Foam	79
3.2.5.1 Compressive Strength	79

3.2.5.2 Tensile Strength	80
3.2.5.3 Density	80
3.2.6 Polyurethane Mortar Preparation	80
3.2.7 Polyurethane Mortar Tests	81
3.2.7.1 Thermal Insulation	81
3.2.7.2 Sound absorption coefficient	81
<b>CHAPTER (4)</b> <b>RESULTS AND DISCUSSION</b>	<b>85</b>
4.1 Petroleum based polyurethane foam results	89
4.1.1 Characterization of PU foam by FTIR	89
4.1.2 Scanning Electron Microscope	90
4.1.3 Mechanical tests results for PU foam	91
A) Compressive strength results	91
B) Tensile Strength Results	92
4.1.4 Thermal Insulation and Sound Absorption	93
4.2 Bio based Polyurethane Foam Results	95
4.2.1 Characterization	95
A) Fourier Transform Infrared Spectroscopy	95
B) X – Ray Diffraction (XRD)	97
C) Scanning Electron Microscope (SEM)	98
D) Thermogravimetric analysis (TGA/DTG)	99
4.2.2 Physical - Mechanical Properties of Bio-Based Polyurethane Foam	100
A) Density	100
B) Compression Strength	101
C) Tensile Strength	102
4.2.3 Sound Absorption and Thermal Insulation	103
A) Sound absorption	103
B) Thermal insulation	106
<b>CHAPTER (5)</b> <b>SUMMARY AND CONCLUSIONS</b>	<b>150</b>
5.1 Summary	150
5.2 Conclusions	151
References	154

## **LIST OF TABLES**

<b>Table</b>	<b>Title of Tables</b>	<b>Page</b>
<b>CHAPTER (2)</b>		
2.1	Common polymerization initiators for polyether polymerization	10
2.2	The most important diols and triols used for polyester polyol synthesis	15
2.3	Aliphatic dicarboxylic acids used for polyester polyol synthesis	15
2.4	Aromatic dicarboxylic acids and their derivatives used for polyester polyol synthesis	15
2.5	Fatty acid composition of common seed oils used for polyester polyol synthesis	43
2.6	General features of the main types of Portland cement	59
2.7	Typical range in mineral proportions in modern Portland cements	61
2.8	Comparison between thermal insulation materials	64
<b>CHAPTER (3)</b>		
3.1	Physical properties of cement.	67
3.2	Chemical analysis of cement	67
3.3	Physical and chemical properties of the used sand.	68
3.4	Chemical analysis of the used water	68
3.5	Physical properties of polyether polyol	69
3.6	Physical properties of castor oil	70
3.7	Physical properties of triethylenediamine	71
3.8	Physical properties of tin octoate.	72
3.9	Physical properties of polydimethyl siloxane	73
3.10	Formulations of rigid polyurethane foam	75
3.11	Typical Formulations of bio based rigid polyurethane foam.	76
<b>CHAPTER (4)</b>		
4.1	Effect of adding tertiary amine catalyst on compressive strength	91
4.2	Effect of adding water on the Compressive strength	91
4.3	Effect of adding tertiary amine catalyst on tensile strength	92