



Ain Shams University



Faculty of Science

Synthesis and Evaluation of Some Nonionic Surfactant Derived From Heterocyclic Compound as Corrosion Inhibitors in Petroleum Equipment

A Thesis submitted for
the Award of the Ph.D. Degree of Science in Chemistry

By

Ahmed Abdel Rahman Ibrahim Ghareeb

M.Sc. in Organic Chemistry - Helwan University (2014)

Thesis Supervisors

Prof. Dr. Maher Abd El Aziz El Hashash

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

Prof. Dr. Nabel Abdel Moneem Negm

*Prof. of Petrochemicals, Petrochemicals Department
Egyptian Petroleum Research Institute*

Dr. Salah Mahmoud Tawfik

*Researcher of Petrochemicals, Petrochemicals Department
Egyptian Petroleum Research Institute*

2018



Ain Shams University



Faculty of Science

Approval Sheet

Title: - Synthesis and Evaluation of Some Nonionic Surfactant Derived From Heterocyclic Compound as Corrosion Inhibitors in Petroleum Equipment

Name of Candidate: Ahmed Abdel Rahman Ibrahim Ghareeb

This Thesis has been approved for submission by supervisor:-

Thesis Supervisors

Thesis approved

Prof. Dr. Maher Abd El Aziz El Hashash

Prof. (DSc) Organic Chemistry

Faculty of Science Ain Shams University

M. A. El Hashash

Prof. Dr. Nabel Abdel Moneem Negm

Prof. of Petrochemicals

Petrochemicals Department

Egyptian Petroleum Research Institute

Nabel Negm

Dr. Salah Mahmoud Tawfik

Researcher of Petrochemicals

Petrochemicals Department

Egyptian Petroleum Research Institute

Salah Tawfik

Head of Chemistry Unit

Faculty of Science Ain Shams University

Prof . Dr. Ibrahim H. A. Badr



Ain Shams University



Faculty of Science

Approval Sheet

Title:- "Synthesis and Evaluation of Some Nonionic Surfactant Derived From Heterocyclic Compound as Corrosion Inhibitors in Petroleum Equipment"

Name of Candidate: Ahmed Abdel Rahman Ibrahim Ghareeb

Examiners Committee

Approval

Prof. Dr. Maher Abd El Aziz El Hashash

*Prof. (DSc) Organic Chemistry
Faculty of Science Ain Shams University*

M. A. El Hashash

Prof. Dr. Nabel Abdel Moneem Negm

*Prof. of Petrochemicals
Petrochemicals Department
Egyptian Petroleum Research Institute*

Nabel Negm

Prof. Dr. Nadia Gharib Kandile

*Prof. of Organic Chemistry
Faculty Of women Ain Shams University*

Nadia Kandile

Prof. Dr. Hasan Ahmed Shehata

*Prof. of Physical Chemistry
Faculty of Science -Al-Azhar university*

Dr. Hassan Shehata

*Head of Chemistry Unit
Faculty of Science Ain Shams University*
Prof. Dr. Ibrahim H. A. Badr



Ain Shams University



Faculty of Science

Researcher Data

Name: - Ahmed Abdel Rahman Ibrahim Ghareeb

Date of Birth: - 26/6/1977

Academic Degree: - Ph.D. Degree of Science

Field Of specification: - Chemistry

University issued the Degree:- Ain shams University -
Faculty of science- Chemistry Department

Graduation year: - 1999

Date of issued Degree:- 2018

Current Job: - Chemical lab Assistant General Manager
Khalda Petroleum Company

ACKNOWLEDGEMENT

Before all and above, I thank and pray to *ALLAH* for unlimited help and uncounted reasons.

I am greatly indebted to **Prof. Dr. Maher Abd El Aziz El Hashash**, Professor of Organic Chemistry, Faculty of Science, Ain Shams University, for his fruitful discussion, effective participation and deep concern of this work.

I am immensely grateful and highly indebted to my revered guide **Prof. Dr. Nabel Abdel Moneem Negm**, Professor of Petrochemical, Petrochemicals department, Egyptian Petroleum Research Institute, for suggesting the topic of this Thesis, planning of experimental work, continuous following and enormous time spent in careful revision.

I am thankful to Dr. **Salah Mahmoud Tawfik**, Researcher of Petrochemicals, Petrochemicals department, Egyptian, Petroleum Research Institute

Finally, I cannot forget complete assistance and encouragement of my Family, and I would like to dedicate this work to the soul of my dear late father.

Contents

List of figures.....	III
List of tables.....	VI
List of Schemes	VII
ABSTRACT.....	VIII

Chapter one

1	Introduction	1
1.1	Definition of corrosion	1
1.2	Forms of corrosion	2
1.2.1	Uniform corrosion	2
1.2.2	Localized corrosion	2
1.2.3	Intergranular corrosion	3
1.2.4	Selective corrosion	3
1.2.5	Erosion corrosion.....	4
1.2.6	Stress corrosion cracking.....	4
1.3	Serious effect of corrosion	5
1.4	Mitigation of corrosion	6
1.4.1	Material selection	6
1.4.2	Coatings	7
1.4.3	Inhibitors.....	7
1.4.4	Cathodic protection	8
1.4.5	Design.....	8
1.5	Surfactants	9
1.5.1	Classification of surfactants	9
1.5.2	Surfactant as corrosion inhibitors.....	20
1.5.3	Properties monitoring of Corrosion Inhibitors	103

Chapter Two

2	Experimental.....	114
2.1	Chemical used	114

2.2	Instrumentation	115
2.3	Synthesis	116
2.3.1	Preparation of Sebacic acid-antipyrine amide	116
2.3.2	Preparation of nonionic Surfactant	116
2.4	Measurements	117
2.4.1	Surface Tension Measurements (γ)	117
2.4.2	Interfacial Tension Measurements	117
2.4.3	Emulsion Stability	118
2.4.4	Weight Loss Measurements	118
2.4.5	Polarization Measurements	119
2.4.6	Electrochemical impedance spectroscopy (EIS)	122

Chapter three

3	Results and Discussion	124
3.1	Chemical Structure of the Synthesized Compounds.....	124
3.1.1	Compound I	126
3.1.2	Compound (II)	130
3.1.3	Compound (III).....	134
3.1.4	Compound (IV)	138
3.2	Surface activity of the prepared nonionic surfactants	142
3.2.1	Surface tension	142
3.3	Evaluation of the Nonionic Surfactants as Corrosion Inhibitors for Carbon Steel in 1.0 M HCl	149
3.3.1	Weight loss point of view	149
3.3.2	Gravimetric measurements	149
3.3.3	Electrochemical polarization studies	174
3.3.4	Electrochemical impedance spectroscopy	182
3.3.5	Surface activity-corrosion inhibition efficiency relationship	191
References		192
Summary and conclusion.....		224

List of Figures

Figure 1: Eight types of corrosion damage.	4
Figure 2 Simplified surfactant structure.....	9
Figure 3: The schematic representative of the electrical cell used in the study.....	121
Figure 4: FTIR spectrum for Compound I	128
Figure 5: ¹ HNMR Spectrum for Compound (I)	129
Figure 6: FTIR Spectrum for Compound (II).....	132
Figure 7: ¹ H-NMR Spectrum for Compound (II).....	133
Figure 8: FTIR Spectrum for Compound (III).	136
Figure 9: ¹ H-NMR Spectrum for Compound (III).	137
Figure 10: FTIR Spectrum for Compound (IV).	140
Figure 11: ¹ H-NMR Spectrum for Compound(IV).	141
Figure 12: Surface tension vs. log concentration of the prepared nonionic surfactants at 25 °C.....	147
Figure 13: Effect of immersion time on corrosion rate of mild steel inhibited with 25 ppm dose from synthetized compounds (II, III, IV).....	152
Figure 14: Effect of immersion time on corrosion rate of mild steel inhibited with 50 ppm dose from synthetized compounds (II, III, IV).....	153
Figure 15: Effect of immersion time on corrosion rate of mild steel inhibited with 100 ppm dose from synthetized compounds (II, III, IV).....	154
Figure 16: Effect of immersion time on corrosion rate of mild steel inhibited with 200 ppm dose from synthetized compounds (II, III, IV).....	155

Figure 17: Effect of immersion time on corrosion rate of mild steel inhibited with 400 ppm dose from synthesized compounds (II, III, IV).....	156
Figure 18: Effect of immersion time on corrosion rate of mild steel inhibited with 800 ppm dose from synthesized compounds (II, III, IV).....	157
Figure 19: Effect of inhibitor concentration (Compounds II,III & IV) on corrosion rate of mild steel after immersion time 24 Hr.....	162
Figure 20: Effect of inhibitor concentration (Compounds II,III & IV) on corrosion rate of mild steel after immersion time 48 Hr.....	163
Figure 21: Effect of inhibitor concentration (Compounds II,III & IV) on corrosion rate of mild steel after immersion time 72 Hr.....	164
Figure 22: Effect of inhibitor concentration (Compounds II,III & IV) on corrosion rate of mild steel after immersion time 96 Hr.....	165
Figure 23: Effect of inhibitor concentration (Compounds II,III & IV) on corrosion rate of mild steel after immersion time 120 Hr.....	166
Figure 24: Effect of ethylene oxide units on the corrosion rate of.....	169
Figure 25: Effect of ethylene oxide units on the corrosion rate of.....	170
Figure 26: Effect of ethylene oxide units on the corrosion rate of.....	171
Figure 27: Effect of ethylene oxide units on the corrosion rate of.....	172
Figure 28: Effect of ethylene oxide units on the corrosion rate of.....	173
Figure 29: Current–potential relationships (cathodic and anodic) for	178
Figure 30: Current–potential relationships (cathodic and anodic) for	179
Figure 31: Current–potential relationships (cathodic and anodic) for	180
Figure 32: Nyquist plots for carbon steel in 1 M HCl in the presence of different concentrations of II inhibitor (ppm by weight) at 25 °C ..	186

Figure 33: Nyquist plots for carbon steel in 1 M HCl in the presence of different concentrations of III inhibitor (ppm by weight) at 25 °C.	187
Figure 34: Nyquist plots for carbon steel in 1 M HCl in the presence of different concentrations of IV inhibitor (ppm by weight) at 25 °C.	188
Figure 35: Equivalent circuit for the corrosion behavior of studied systems.....	189