

UNIVERSITI TEKNOLOGI MARA

**SYNTHESIS, CHARACTERIZATION
AND CORROSION INHIBITION
STUDIES OF BENZOYL THIOUREA
DERIVATIVES**

NOOR KHADIJAH BINTI MUSTAFA KAMAL

Thesis submitted in fulfillment
of the requirements for the degree of
Master of Science (Applied Chemistry)

Faculty of Applied Sciences

November 2017

ABSTRACT

A series of benzoylthiourea compounds, namely *N*-phenyl-*N'*-benzoylthiourea (B1) and its methoxy (B2), nitro (B3) and chloro (B4) derivatives were successfully synthesized from the reaction of benzoyl chloride with amine derivatives in acetone reflux. The effectiveness of these compounds as corrosion inhibitor on mild steel in 1.0 M H₂SO₄ solution was measured using weight loss method, linear polarization resistance (LPR) technique and electrochemical impedance spectroscopy (EIS). The inhibition efficiency (IE%) was affected by the presence of substituent such as methoxy, nitro and chloro. Compound B2 that contain methoxy substituent has the highest IE% which is 90.59%. The order of the effectiveness of the compounds as corrosion inhibitors is B2>B1>B4>B3. All four compounds are mixed-type inhibitors and the inhibition of these compounds obeyed the Langmuir adsorption isotherm.

ACKNOWLEDGEMENT

Firstly, I wish to thank God for giving me the opportunity to embark on my Master and for completing this long and challenging journey successfully. My gratitude and thanks go to my supervisor Dr. Karimah Kassim, and co-supervisor, Puan Adibatul Husna Fadzil. Thank you for the support, patience and ideas in assisting me with this project. I also would like to express my gratitude to the UiTM staff Puan Noor Haida, for providing the facilities, knowledge and assistance.

Special thanks to my colleagues and friends for helping me with this project. Finally, this thesis is dedicated to my parents for the vision and determination to educate me. This piece of victory is dedicated to both of you. Alhamdulillah.

TABLE OF CONTENTS

	Page
CONFIRMATION BY PANEL OF EXAMINERS	ii
AUTHOR'S DECLARATION	iii
ABSTRACT	iv
ACKNOWLEDGEMENT	v
TABLE OF CONTENTS	vi
LIST OF TABLES	ix
LIST OF FIGURES	xi
LIST OF SCHEMES	xiii
LIST OF ABBREVIATIONS	xiv
CHAPTER ONE: INTRODUCTION	1
1.1 Corrosion	1
1.2 Thiourea and Its Derivatives	4
1.3 Problem Statement	5
1.4 Significance of Study	6
1.5 Objectives of the Study	7
1.6 Scope and Limitation of the Study	8
CHAPTER TWO: LITERATURE REVIEW	9
2.1 Corrosion Inhibitor	9
2.1.1 Organic Compound as Corrosion Inhibitor	10
2.1.2 Thiourea and Its Derivatives as Corrosion Inhibitor	11
2.1.3 Corrosion Inhibition in Acidic Media	14
2.1.3.1 Interaction of The Inhibitor With a Metal Surface	14
2.1.3.2 Interaction between Adsorbed Inhibitors	16
2.1.3.3 Interaction of the Inhibitor with Water Molecules	16
2.1.3.4 Relationship between Inhibitor Reactivity and Efficiency	17
2.2 Synthesis of Thiourea Derivatives	17

2.3 Characterization of Thiourea Derivatives	17
2.3 Corrosion Inhibition Studies	26
2.3.1 Weight Loss Method	26
2.3.2 Linear Polarization Technique	27
2.3.3 Electrochemical Impedance Spectroscopy	28
2.3.4 Mechanism of Inhibition of Thiourea Derivatives and Effects of Substituents on Corrosion Inhibition Efficiency	29
2.4 Adsorption isotherm	32
2.4.1 Langmuir Adsorption Isotherm	33
2.4.2 Temkin Adsorption Isotherm	34
2.4.3 Frumkin Adsorption Isotherm	34
2.4.4 Florry-Huggins Adsorption Isotherm	35
2.5 Thermodynamic Principles of Corrosion	35
CHAPTER THREE: METHODOLOGY	37
3.1 Introduction	37
3.2 Chemicals	38
3.3 Sample	38
3.4 Synthesis of Benzoylthiourea Derivatives	38
3.4.1 <i>N</i> -phenyl- <i>N'</i> -benzoylthiourea, B1	38
3.4.2 <i>N</i> -(2-methoxyphenyl)- <i>N'</i> -benzoylthiourea, B2	39
3.4.3 <i>N</i> -(2-nitrophenyl)- <i>N'</i> -benzoylthiourea, B3	39
3.4.4 <i>N</i> -(2-chlorophenyl)- <i>N'</i> -benzoylthiourea, B4	39
3.5 Characterization of Benzoylthiourea Derivatives	41
3.5.1 CHNS Elemental Analyzer	41
3.5.2 Fourier-Transform Infrared (FTIR) Spectroscopy	41
3.5.3 Nuclear Magnetic Resonance (NMR) Spectroscopy	41
3.5.4 Ultraviolet-Visible (UV-Vis) Spectroscopy	42
3.6 Corrosion Inhibition Studies	42
3.6.1 Weight Loss Method	42
3.6.2 Linear Polarization Technique	42
3.6.3 Electrochemical Impedance Spectroscopy (EIS)	43
3.7 Adsorption Isotherm	44