### SYNTHESIS, CHARACTERISATION AND ANTI-CORROSION SCREENING OF Ni(II) N-METHYLCYCLOHEXYLDITHIOCARBAMATE AND N-ETHYLCYCLOHEXYLDITHIOCARBAMATE COMPLEXES

# SITI RAUBBAH BINTI MOHD ZAMRI

Final Year Project Report Submitted In Partial Fulfillment of The Requirement for The Degree of Bachelor of Science(Hons.) of Chemistry in the Faculty of Applied Sciences Universiti Teknologi MARA

JULY 2017

The Final Year Project Report entitled "Synthesis, Characterisation And Anti-Corrosion Screening of Ni(II) *N*-methylcyclohexyldithiocarbamate And Ni(II) *N*-ethylcyclohexyldithiocarbamate Complexes" was submitted by Siti Raubbah Binti Mohd Zamri, in partial fulfilment of the requirements for the Degree of Bachelor of Sciences (Hons.) Chemistry, in the Faculty of Applied Sciences, and was approved by

> Nur Nadia Dzulkifli Supervisor B. Sc. (Hons.) Chemistry Faculty of Applied Sciences Universiti Teknologi MARA Kuala Pilah Campus 72000 Kuala Pilah Negeri Sembilan

Nurul Huda Abdul Halim Project Coordinator B. Sc. (Hons.) Chemistry Faculty of Applied Sciences Universiti Teknologi MARA Kuala Pilah Campus 72000 Kuala Pilah Negeri Sembilan Mazni Musa Head of Programme B. Sc. (Hons.) Chemistry Faculty of Applied Sciences Universiti Teknologi MARA Kuala Pilah Campus 72000 Kuala Pilah Negeri Sembilan

Date : \_\_\_\_\_

# **TABLE OF CONTENTS**

		Pages		
ACKNOWLEDGEMENTS				
TABLE OF CONTENTS				
LIST OF TABLES				
LIST OF FIGURES				
LIST OF ABB	REVIATIONS	Х		
ABSTRACT		xi		
ABSTRAK		xii		
CHAPTER 1	INTRODUCTION	1		
1.1	Background of study	1		
1.2	Problem statement	2		
1.3	Significant of Study	3		
1.4	Objective of Study	3		
CHAPTER 2	LITERATURE REVIEW	5		
2.1	Synthesis of Ni(II) <i>N</i> -ethylo and <i>N</i> -	cyclohexyl-		
	methylcyclohexyldithiocart	pamates		
	Complexes	5		
2.2	Characterisation of Ni(II) N	-ethylcyclohexyl-		
	and <i>N</i> -	amatas		
	Complexes	6		
	2.2.1 Elemental analyser	(carbon hydrogen 6		
	nitrogen and sulfur)	(carbon, nyarogen, 0		
	2.2.2 FTIR-ATR spectros	scopy 7		
	2.2.3 Ultraviolet Visible	UV-Vis 8		
	2.2.4 Nuclear Magnetic R	Resonance (NMR) 8		
2.3	Mode of coordination	9		
2.4	Application			
	2.4.1 Anti-corrosion	10		

2.4.2	Biological activity	11
2.4.3	Dye removal	12

<b>CHAPTER 3</b>	METHODOLOGY		13
3.1	Mater	Materials	
	3.1.1	Chemical	13
	3.1.2	Apparatus	13
	3.1.3	Instrument	13
3.2	Methods		14
	3.2.1	Synthesis of Ni(II) N-ethylcyclohexyl-	14
		and N-methylcyclohexyldithiocarbamates	complexes
3.3	Chara	cterisation	15
	3.3.1	Elemental analyser (carbon, hydrogen,	15
		nitrogen and sulfur)	
	3.3.2	FTIR-ATR spectroscopy	15
	3.3.3	Ultraviolet Visible, UV-Vis	15
	3.3.4	Nuclear Magnetic Resonance (NMR)	16
	3.3.5	Molar conductivity	16
	3.3.6	Gravimetric analysis	16
3.4	Corrosion Inhibitor Study		17
	3.4.1	Preparation of solution	17
	3.4.2	Weight Loss method	17
снартер 4	DECI	II T AND DISCUSSION	19
CHAFTER 4	Synthe	Synthesis of Ni(II) N-	
4.1	methy	lcyclohexyldithiocarabamate	18
	and N-ethylcyclohexyldithiocarbamate		
	Comp	lexes	
4.2	Characterisation		21
	4.2.1	Elemental analyser	21
	4.2.2	FTIR-ATR spectroscopy	23
	4.2.3	Ultraviolet-visible(UV-Vis)	28
	4.2.4	Nuclear Magnetic Resonance (NMR)	30
	4.2.5	Molar conductivity	33
	4.2.6	Gravimetric analysis	33
4.3	Corrosion Inhibitor Study		34
	4.3.1	Weight Loss method	35

# ABSTRACT

#### SYNTHESIS,

### CHARACTERISATION AND ANTI-CORROSION SCREENING OF NI(II) N-METHYLCYCLOHEXYLDITHIOCARBAMATE AND Ni(II) N-ETHYLCYCLOHEXYLDITHIOCARBAMATES COMPLEXES

Dithiocarbamate is a ligand that can act as a corrosion inhibitors due to the sulphur and nitrogen atom. Ni(II) Npresence of methylcyclohexyldithiocarbamate, Ni[MeCycHexdtc]<sub>2</sub> and Ni(II) Nethylcyclohexyldithiocarbamates, Ni[EtCycHexdtc]<sub>2</sub> complexes were synthesised through one-pot synthetic method at room temperature and have been characterised by using elemental analyser (CHNS), fourier transform infraredattenuated total reflectance (FTIR-ATR) spectroscopy, Ultraviolet-Visible (UV-Vis) spectroscopy, nuclear magnetic resonance (NMR) and molar conductivity. The stretching frequency of thioureide band, v(C - N) appeared at 1497 cm<sup>-1</sup> for Ni[MeCycHexdtc]<sub>2</sub> and 1484 cm<sup>-1</sup> for Ni[EtCycHexdtc]<sub>2</sub> while the stretching frequency of v(C=S) has shifted to lower wavenumber as it changes from double bonding to partial bonding, v(C - S) at  $(1000 \pm 70 \text{ cm}^{-1})$  when compared with the raw material. The structures of both complexes were confirmed as the calculated of percentage for carbon, hydrogen, nitrogen and sulphur were similarly as the results by the elemental analyser. Meanwhile, the percentage of the metal of the complexes carried by gravimetric analysis which 12.8% are is (Ni[MeCycHexdtc]<sub>2</sub>) and 12.25% (Ni[EtCycHexdtc]<sub>2</sub>). This was supported by the result from <sup>1</sup>H NMR and <sup>13</sup>C NMR with the amount of the hydrogen and carbon in the symmetry of complexes. The Ni[MeCycHexdtc]<sub>2</sub> complex indicated the square planar complex but not for Ni[EtCycHexdtc]<sub>2</sub> complex where it shows the tetrahedral complex. There also a new absorption peak appeared at range 400-600 nm for UV-Vis spectra which show the presence of *d*-*d* transition and proved that the complexes were successfully formed. The corrosion inhibition was determined by weight loss method which also give the percentage of inhibition efficiency. Based on the experiment, the mass loss will decrease when the concentration of the complexes were increased. Thus, percentage of efficiency will also increase.