# SYNTHESIS AND CHARACTERIZATION OF MgO NANOPOWDERS BY SOL-GEL METHOD INCORPORATED REFLUX APPROACH

NORAZIAHWATI IBRAHIM

BACHELOR OF SCIENCE (Hons.) CHEMISTRY FACULTY OF APPLIED SCIENCES UNIVERSITI TEKNOLOGI MARA

OCTOBER 2010

#### ACKNOWLEDGEMENTS

Firstly, I would like to express my thankful to the Almighty for blessing me with the force and capability to complete this final year project on the time required. In addition, I would like to express my deep appreciation especially to my supervisor, Mr. Mohd Sufri Mastuli for his guidance, advices and helps me a lot in order to make me understand very well about this project. I am sincerely also record my appreciation to him for his useful comments on several chapters. Similarly my heartfelt thanks for all of lecturers, lab assistants that give me wonderful cooperation and helps. Last but not least, enormous gratitude to my parents Ibrahim Ahmad and and the rest of my family members for their support, encouragement and contribution. Special thanks also to my friends for their support, help, advice and sharing their idea with me in completing my project. Thanks a lot and may Allah bless all of you.

Noraziahwati Ibrahim

# TABLE OF CONTENTS

# Page

ACKNOWLEDGEMENTS TABLE OF CONTENTS LIST OF TABLES LIST OF FIGURES LIST OF ABBREVIATIONS ABSTRACT ABSTRAK		iii iv v vi vii viii ix
СПА	στερ 1 Ιντροριζτιον	
СПА 11	Background and problem statement	1
1.1	Significance of study	5
1.3	Objectives of study	5
CHA	PTER 2 LITERATURE REVIEW	
2.1	Overview of MgO	6
2.2	Sol-gel method for synthesis MgO powders	8
2.3	Various sizes and morphologies of MgO powders	9
2.4	Characterization of MgO powders	
	2.4.1 Thermogravimetric Analyze (TGA)	13
	2.4.2 X-Ray Diffractometer (XRD)	14
	2.4.3 Scanning Electron Microscope (SEM)	17
CHA	PTER 3 METHODOLOGY	
3.1	Reagents and chemicals	19
3.2	Synthesis of MgO powders	19
3.3	Characterization of MgO powders	20
CHA	PTER 4 RESULTS AND DISCUSSION	
4.1	Thermal analysis of sol-gel products	21
4.2	Phase evolution of MgO nanopowders	23
4.3	Morphological studies of MgO nanopowders	28
CHAPTER 5 CONCLUSION AND RECOMMENDATIONS		30
CITE	D REFERENCES	31
APPE	ENDICES	33
CURI	RICULUM VITAE	36

#### ABSTRACT

# SYNTHESIS AND CHARACTHERIZATION OF MgO NANOPOWDERS BY SOL-GEL METHOD INCORPORATED REFLUX APPROACH

Magnesium oxide powders have been prepared by sol-gel method in reflux condition. The powders are synthesized using magnesium acetate tetrahydrate and oxalic acid as precursors with ethanol as a solvent. The concentration of the oxalic acid have been varied which are 0.5 M and 1.0 M. All the sol-gel products were annealed at 600 °C, 700 °C and 800 °C for 6 hours. The annealed products were systematically investigated by Thermogravimetric Analyzer (TGA), X-Ray Diffraction (XRD) and Scanning Electron Microscope (SEM). The effects of annealing temperature and the concentration of oxalic acid onto phases and morphologies of MgO were investigated throughout this study. Thermogravimetric analysis provided insight into the decomposition process of the magnesium acetate tetrahydrate. The XRD analysis indicates that all the products were of cubic lattice with increasing degree of crystallinity when increased the annealing temperatures. Besides that, the crystallite sizes of MgO were increased due to increasing of annealing temperatures and the concentration of oxalic acid. In this study, the crystallite size of the MgO products was in a range from 13.5 nm to 22.7 nm. The morphology of MgO was nanospheres.

#### **CHAPTER 1**

### **INTRODUCTION**

# **1.1 Background and problem statement**

Magnesium oxide (MgO) is a versatile oxide material due to its unique physical and chemical properties. For example, this material is very stable thermally. In addition, MgO has high melting point (2850 °C) and high boiling point (3600 °C) (Klabunde, 2001). Thus, these factors cause MgO compounds become extremely important for use in industries and very useful for various applications. For instances, they are used in catalysis, toxic waste remediation, antibacterial materials, refractory material industries paints and superconductor products (Wang *et al.*, 2006).

Since the discovery of carbon nanotubes, compounds with such interesting morphologies and specific structures, are getting much attention by researchers due to their special properties. It is well known that the shape and size of the nanostructures have much on their properties (Yang *et al.*, 2005). Therefore, more researches have been focused on the synthesis and characterization of magnesium oxides with various sizes and morphologies. There are many novel nanostructures of MgO have been synthesized, including nanoparticles (Elaheh *et al.*, 2008), nanoplates (Meshkani *et al.*,