STABILITY OF CYPERMETHRIN AGAINST A PREPARED HIGH SURFACE AREA MAGNESIUM OXIDE

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A thesis submitted in fulfilment of the
requirements for the award of the degree of
Master of Science (Chemistry)

Faculty of Science
Universiti Teknologi Malaysia

JUNE 2006

Dedicated to my beloved father Hj Mohd Yusoff bin Hussin,
Mother Hjh Wan Bidah binti Wan Sulaiman,
family and friends.

ACKNOWLEDGEMENT

First of all, in a humble way I wish to give all the Praise to Allah, the Almighty God for giving me the strength and the time to complete this work.

I would like to take this opportunity to express my sincere thanks to my supervisor Assoc. Prof. Dr Abdul Rahim bin Yacob for believing in me. I am also grateful with his patience, endless efforts in guiding and imparting his vast knowledge to me. Writing this thesis is a remarkable accomplishment, thanks to the ever constant advice from my supervisor.

At the same time, I would also like to thank all lab assistants from the Department of Chemistry for their help and advice with the handling of the equipment in the laboratories. To all my colleagues, thank you for your help and support. I am also grateful to the Department of Chemistry, Faculty of Science Universiti Teknologi Malaysia for all the facilities and financial support.

Lastly, I would like to acknowledge my family, whose patience love enabled me to complete this research.

ABSTRACT

High surface area magnesium oxide was prepared by various methods of preparation using modified dehydration technique. Commercial magnesium oxide was first transformed into its hydroxide. The magnesium hydroxide was heated under high vacuum at different temperatures of 200, 400, 600 and 800 degree Celsius and high surface area magnesium oxide was obtained. The activated magnesium oxide sample was labeled as dehydrated magnesium oxide. The same magnesium hydroxide was also calcined at various temperatures and was labeled as dehydrated and calcined magnesium oxide. Finally, the third magnesium oxide sample was labeled as magnesium oxide sol gel, produced by synthesis of Mg turnings to form sol gel solution and was dried using autoclave which will convert to its magnesium hydroxide sol gel. Again, activation using high vacuum will convert magnesium hydroxide sol gel into its high surface area magnesium oxide. To characterize the resulting magnesium oxide samples, several spectroscopic techniques were used which includes Fourier Transform Infra-Red, X-Ray Diffraction, Nuclear Magnetic Resonance and Electron Spin Resonance. For better understanding of the magnesium oxide surface area and morphology, Nitrogen Gas Adsorption and Scanning Electron Microscope, was used. Pyrethrin is the most effective insecticide. However it is not harmful to mammals or humans. Synthetic pyrethroid that is cypermethrin later was developed. Since cypermethrin was sprayed towards furnitures, floors, walls at home and at the agriculture field, thus their stability towards these matrices is important. In this study, the stability of cypermethrin under variously prepared high surface area magnesium oxide effect was studied using Fourier Transform Infra-Red spectroscopy and Gas Chromatography with Electron Capture Detector. For comparison, inert matrix of activated carbon was used.

ABSTRAK

Magnesium oksida berluas permukaan tinggi telah disediakan dengan menggunakan pelbagai kaedah. Langkah pertama adalah dengan menukarkan magnesium oksida komersil kepada magnesium hidroksida dan telah melalui penggunaan vakum berkuasa tinggi dengan pemanasan pada pelbagai suhu 200, 400, 600 dan 800 darjah celsius, telah menghasilkan magnesium oksida berluas permukaan tinggi. Sampel ini dilabel sebagai magnesium oksida terhidrat. Magnesium hidroksida yang sama, turut dipanaskan secara pengkalsinan pada pelbagai suhu dan dilabel sebagai magnesium hidroksida terhidrat dan kalsin. Akhir sekali, sampel magnesium oksida yang ketiga dikenali sebagai sol gel magnesium oksida kerana teknik penyediakan adalah sedikit berbeza iaitu melibatkan sintesis yang menukarkan pita magnesium menjadi cecair sol gel dan dikeringkan dengan menggunakan 'autoclave' menukarkan ia kepada serbuk sol gel magnesium hidroksida. Pemanasan menggunakan vakum pada pelbagai suhu telah menukarkannya kepada magnesium oksida berluas permukaan tinggi. Bagi pencirian ke atas sampel magnesium oksida yang telah disediakan, beberapa kaedah spektroskopi telah digunakan iaitu Spektoskopi Inframerah, Pembelauan Sinar-X, Resonans Magnet Nukleus dan Resonans Spin Elektron. Bagi mengetahui lebih lanjut tentang permukaan sampel magnesium oksida yang diperolehi, penjerapan Gas Nitrogen dan Mikroskop Imbasan Elektron telah dijalankan. Pyrethrin merupakan racun serangga yang sangat berkesan. Walau bagaimanapun ia tidak merbahaya kepada mamalia mahupun manusia. Kemudian, pyrethroid sintetik iaitu cypermethrin telah dihasilkan. Memandangkan penggunaan cypermethrin adalah meliputi penyemburan di sofa, lantai, dinding rumah dan juga dalam bidang pertanian, maka kestabilan terhadap pelbagai matrik adalah amat penting. Dalam kajian ini, kestabilan cypermethrin di dalam pelbagai jenis magnesium oksida berluas permukaan tinggi dikaji dengan menggunakan Spektrokopi Inframerah and juga Kromatografi Gas dengan Pengesan Penangkap Elektron. Sebagai perbandingan, arang teraktif juga telah turut digunakan.

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LIST OF SYMBOLS/ABBREVIATIONS

λ	-	Wavelength
2θ	-	Bragg Angle
FTIR	-	Fourier-Transformed Infrared
KBr	-	Potassium Bromide
XRD	-	X-ray Diffraction
Cu K_α	-	X-ray diffraction from Copper K energy levels
PDF	-	Powder Diffraction File
FWHM	-	Full Width Half Maximum
SEM	-	Scanning Electron Microscopy
EDX	-	Energy Dispersive X-Ray Analysis
BET	-	Brunnauer, Emmett and Teller
IUPAC	-	International Union of Pure and Applied Chemistry
NA	-	Nitrogen Adsorption
ESR	-	Electron Spin Resonance
NMR	-	Nuclear Magnetic Resonance
GC-ECD/FID	-	Gas Chromatography with Electron Capture Detector / Flame Ionization Detector
MgO	-	Magnesium Oxide
D-MgO	-	Dehydrated MgO
DC-MgO	-	Calcine MgO
SG-MgO	-	Sol Gel MgO
H_2	-	Hydrogen
OH	-	Hydroxyl
P/P_o	-	Relative pressure; obtained by forming the ratio of the equilibrium pressure and vapour pressure P_o of the adsorbate at the temperature where the isotherm is measured

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CHAPTER 1

INTRODUCTION

1.1 Background of Research

1.1.1 Metal Oxide

Magnesium is one of the elements in Group 2, also known as alkaline earth metal. The main minerals in which magnesium is found are carnellite, magnesite and dolomite. Magnesium is the eight most abundance elements in the earth's crust. According to Knozinger *et al.* (1993) and Webb *et al.* (2003), magnesium oxide, MgO exhibits a simple rock salt structure with minimal surface defect and may easily be produced as high surface area material and also an excellent model compound for the study of absorption phenomena.

1.1.2 Insecticides

One of the oldest insecticides that have been discovered is pyrethrum (Garfield. E, 1990). Pyrethrins insecticides are derived from pyrethrum flowers, such as chrysanthemums. The aromatic flower heads are powdered or extracted with solvents. The resultant substance or product is contact poisons and nerve toxins for

insects (Hitmi *et al.* 1998 and Garfield, 1990). Pyrethrins boost several distinctions of advantages such as-low mammalian toxicity, high toxicity to a broad spectrum of insects at very low dosages and minor environmental impact (Chen and Wang, 1996, Pedigo, 1989). Commercial uses ranges from household to industrial insect control (Elliott, 1977).

Pyrethrins are however expensive and not stable especially when exposed to sunlight, thus; synthetic pyrethrin also known as synthetic pyrethroid have been introduced such as allethrin, cypermethrin, fenvalerate and permethrin (Elliott, 1977 and Pedigo, 1989).

1.1.3 Problem Statement

Cypermethrin is a type of insecticide which originated from a flower, since the insecticide was sprayed towards tree trunks (garden) or on the floors and walls (house), thus; their stability studies, with respect to times towards these matrices inert and ionic, can open up a new dimensions in synthetic pyrethrins study of nontoxic insecticides and pesticides. If this stability is understood, the effectiveness of these new improved insecticides, which are not harmful to human are a major breakthrough in agriculture sector and human consumption. This application was to discover the ability of prepared high surface area MgO

1.2 Objectives of the Research

The objectives of this research are to investigate the potential of MgO as a reaction medium as well as matrix in pesticide. This could be further divided into three:

1) To synthesize dehydrated MgO, sol gel MgO and to prepare calcined MgO.
2) To characterize all three types of MgO
3) To apply synthesized and prepared MgO in the stability study of cypermethrin.

1.3 Scope of the Research

The scope of the research is to prepare and characterize three types of high surface area MgO that has been identified as dehydrated method MgO (D-MgO), open calcinations MgO (DC-MgO) and sol gel method MgO (SG-MgO). All these three types of MgO were applied as a matrix in the stability study of cypermethrin.

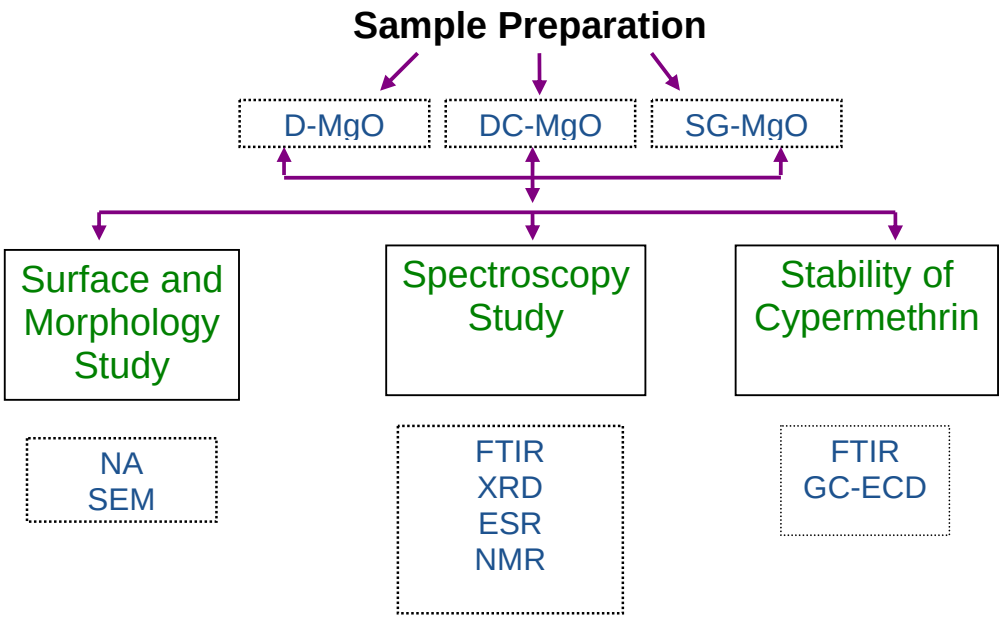

Figure 1.1 Schematic layout of research scope

Resulting MgO samples were characterized using spectroscopy instruments which includes Fourier Transform Infra-Red (FTIR), X-Ray Diffraction (XRD), Electron Spin Resonance (ESR) and Nuclear Magnetic Resonance. To have better understanding on the surface and morphological properties, Nitrogen Gas Adsorption (NA) and Scanning Electron Microscopes (SEM) were used. In stability of cypermethrin, samples were analyzed using Fourier ransform Infra-Red (FTIR) and Gas Chromatography with Electron Capture Detector (GC-ECD).

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