



UNIVERSITI PUTRA MALAYSIA

***SYNTHESIS, CHARACTERIZATION, AND APPLICATION OF
CALCIUM METHOXIDE AS HETEROGENEOUS CATALYST FOR
TRIMETHYLOLPROPANE ESTER CONVERSION REACTION***

HASSAN MASOOD

FK 2011 144

**SYNTHESIS, CHARACTERIZATION, AND
APPLICATION OF CALCIUM METHOXIDE AS
HETEROGENEOUS CATALYST FOR
TRIMETHYLOLPROPANE ESTER
CONVERSION REACTION**

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HASSAN MASOOD

**MASTER OF SCIENCE
UNIVERSITI PUTRA MALAYSIA**

2011

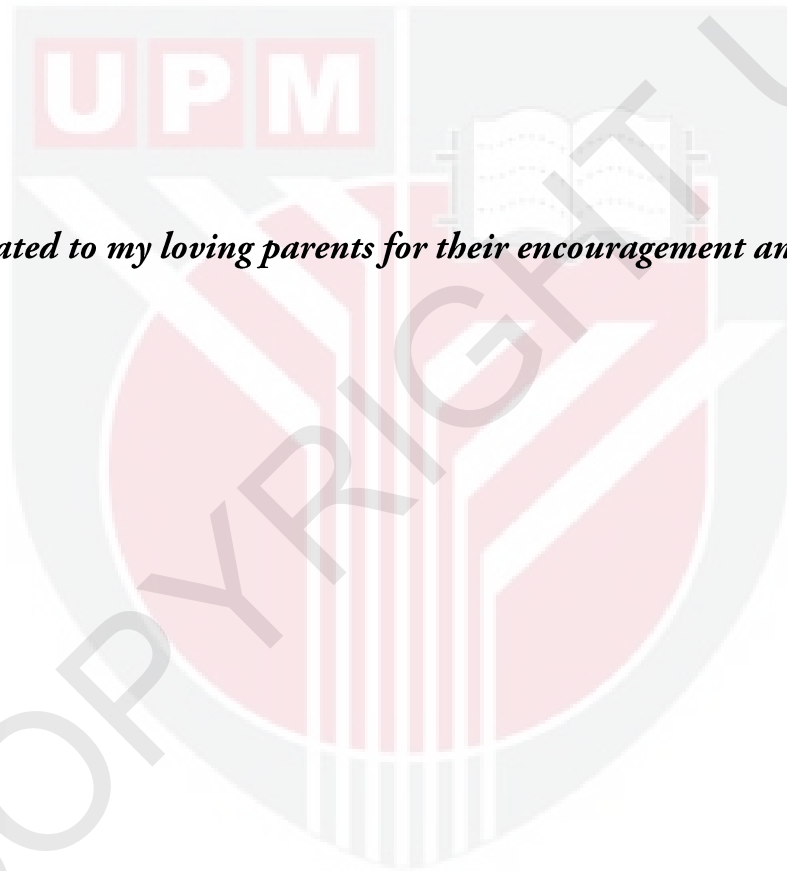
**SYNTHESIS, CHARACTERIZATION, AND APPLICATION OF CALCIUM
METHOXIDE AS HETEROGENEOUS CATALYST FOR
TRIMETHYLOLPROPANE ESTER CONVERSION REACTION**

By

HASSAN MASOOD

**Thesis Submitted to School of Graduate Studies, Universiti Putra Malaysia, in
Fulfillment of the Requirement for the Degree of Master of Science**

December 2011



“Dedicated to my loving parents for their encouragement and concern”

Abstract of thesis presented to the Senate of Universiti Putra Malaysia in fulfillment of the requirement for the degree of Master of Science

SYNTHESIS, CHARACTERIZATION, AND APPLICATION OF CALCIUM METHOXIDE AS HETEROGENEOUS CATALYST FOR TRIMETHYLOLPROPANE ESTER CONVERSION REACTION

By

HASSAN MASOOD

December 2011

Chairman: Professor Robiah Yunus, PhD

Faculty: Faculty of Engineering

Trimethylolpropane esters (TMPE) hold the potential as biolubricant basestock. This research was conducted with an aim to study the synthesis, characterization and application of calcium methoxide as heterogeneous catalyst for production of TMPE. Calcium methoxide was synthesized utilizing a two stage process through calcination and reflux. The synthesis process was optimized by studying the effect of various factors, and the optimum conditions were found to be: calcination time 1.5 hr, amount of methanol 100 mL, and reaction time 2 hr. Synthesized and commercial calcium methoxide were characterized by X-ray diffraction (XRD), fourier transform infra-red spectroscopy (FTIR), Brunauer-Emmett-Teller (BET) surface area measurement, particle size distribution, scanning electron microscopy (SEM), energy-dispersive X-ray spectroscopy (EDX) and thermogravimetric/simultaneous differential thermal analysis (TGA/SDTA). The XRD results revealed calcium

methoxide was successfully synthesized with good purity. The comparison of XRD diffractogram of synthesized and commercial calcium methoxide showed acceptable results. FTIR and EDX results further confirmed the similarities between synthesized and commercial calcium methoxide in terms of having same functional groups and nearly same quantitative composition of calcium, oxygen and carbon. SEM results displayed thermally resistant surface structure with good porosity; BET showed high surface area; particle size analysis evidenced reasonable particle size; and TGA/SDTA revealed good thermal stability of synthesized calcium methoxide. Moreover, it was found to possess mesoporous surface by Barrett–Joyner–Halenda (BJH) method. The results of transesterification reaction proved good catalytic activity of both synthesized and commercial calcium methoxide, and more than 90% yield of trimethylolpropane triesters was obtained after 8 hr reaction time. Overall, this research successfully reported the synthesis of calcium methoxide and its effectiveness to act as a heterogeneous catalyst for conversion of trimethylolpropane (TMP) and palm oil methyl esters (POME) to TMPE.

Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia
sebagai memenuhi keperluan untuk ijazah Master Sains

**SINTESIS, PENCIRIAN, DAN PEMAKAIAN KALSIUM METOKSIDA
SEBAGAI PEMANGKIN HETEROGEN UNTUK ESTER
TRIMETILOLPROPANA TINDAK BALAS PENUKARAN**

Oleh

HASSAN MASOOD

Disember 2011

Pengerusi: Profesor Robiah Yunus, PhD

Fakulti: Kejuruteraan

Trimetilolpropana ester (TMPE) berpotensi sebagai stok asas minyak pelincir bio. Penyelidikan ini telah dijalankan dengan bertujuan untuk mengkaji sintesis, pencirian dan aplikasi kalsium metoksida sebagai pemangkin heterogen bagi penghasilan TMPE. Kalsium metoksida telah disintesis secara dua peringkat iaitu melalui pengkalsinan dan refluks. Proses sintesis tersebut telah dioptimumkan dengan mengkaji kesan daripada pelbagai faktor, dan keadaan optimum telah didapati: masa pengkalsinan 1.5 jam, jumlah metanol 100 mL, dan tempoh tindak balas 2 jam. Pencirian kalsium metoksida komersial dan yang telah disintesis telah dilakukan menggunakan: pembelauan X-ray (XRD), spektroskopi inframerah tranformasian Fourier (FTIR), sukatan luas permukaan Brunauer-Emmett-Teller (BET), taburan saiz zarah, mikroskopi elektron pengimbasan (SEM), spektroskopi X-ray sebaran tenaga (EDX) dan analisis terma untuk pembezaan termogravimetri/serentak (TGA/SDTA). Keputusan analisis XRD telah

menunjukkan bahawa kalsium metoksida berketulenan tinggi telah berjaya disintesis. Perbandingan difraktogram XRD di antara kalsium metoksida yang telah disintesis dan juga yang komersial menunjukkan keputusan-keputusan yang dapat diterima. Keputusan analisis FTIR dan EDX membuktikan terdapat persamaan di antara kalsium metoksida yang komersial dan yang telah disintesis, dari segi kumpulan berfungsi dan komposisi kuantitatif kalsium, oksigen dan karbon. Analisis SEM telah menunjukkan struktur permukaan yang berkeliatan baik dan tahan haba; BET menunjukkan saiz permukaan yang besar; manakala analisis saiz partikel membuktikan saiz liang yang baik; dan analisis TGA/SDTA membuktikan kalsium metoksida yang disintesis mempunyai kestabilan haba yang baik. Tambahan pula, analisis daripada kaedah Barrett-Joyner-Halenda (BJH) menunjukkan kalsium metoksida mempunyai permukaan berliang-meso. Keputusan proses transesterifikasi pula telah membuktikan bahawa pemangkin ini mempunyai aktiviti mangkin yang tinggi sebagai pemangkin heterogen berasaskan pepejal dan hampir 90% TMP triester telah berjaya dihasilkan dalam masa 8 jam. Secara keseluruhan, penyelidikan ini telah dilaporkan penghasilan kalsium metoksida dan keberkesannya untuk bertindak sebagai pemangkin heterogen untuk penukaran TMP dan POME kepada TMPE dengan jayanya.

ACKNOWLEDGEMENTS

First and foremost, I am grateful to Allah who always stands by me and enables me to overcome the difficulties in critical situations. I would like to express my deepest gratitude and appreciation to my supervisor, Dr. Robiah Yunus, for her invaluable supervision, assistance, technical guidance, constructive advices and encouragement throughout the duration of my research.

I would like to extend my thanks and heartiest gratitude to my project leader and supervisory committee member, Dr. Thomas Choong, whose sincerity, dedication and encouraging attitude towards this project has motivated me to strive harder for success. I am highly obliged to Dr. Taufiq Yap, my supervisory committee member, for his support during my research.

I am especially grateful to my friends and colleagues Saiful, Hamidah and Ummi for their kind assistance. My thanks to all the staff members whom I came across at Department of Chemical and Environmental Engineering, Department of Chemistry and Institute of Advanced Technology for their cooperation. Not but the least, I am grateful to Government of Malaysia for providing me financial sponsorship as Graduate Research Assistant (GRA) under the Science Fund with grant no.: 5450511.

APPROVAL

I certify that a Thesis Examination Committee has met on 29th December 2011 to conduct the final examination of Hassan Masood on his thesis entitled “Synthesis, characterization, and application of calcium methoxide as heterogeneous catalyst for trimethylolpropane ester conversion reaction” in accordance with University Colleges Act 1971 and the Constitution of Universiti Putra Malaysia [P.U.(A) 106] 15 March 1998. The Committee recommends that the student be awarded the degree of Master of Science. Members of the Examination Committee were as follows:

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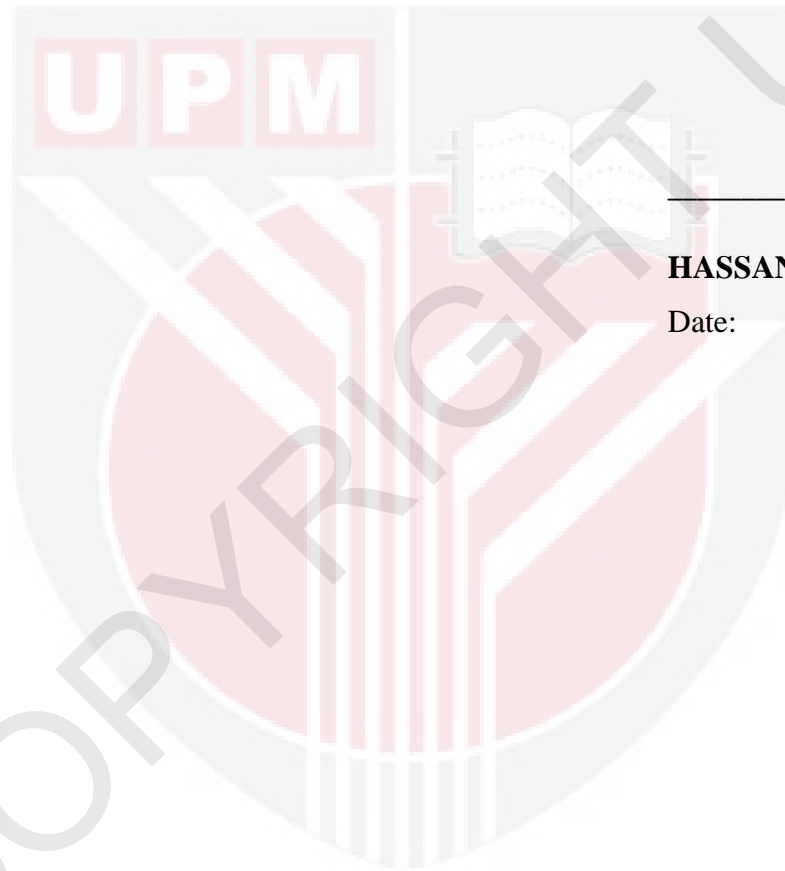
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DECLARATION

I declare that the thesis is on my original work except for quotations and citations which have been duly acknowledged. I also declare that it has not been previously or concurrently submitted for any other degree at UPM or other institutions.



HASSAN MASOOD

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