EXPERIMENTAL AND THEORETICAL STUDIES ON THE PRODUCTION OF BIO-LUBRICANTS BASED ON PALM OLEIN AND JATROPHA OIL

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DEDICATION

This thesis is dedicated to Papa, Mama, Adeq, Yan, Lin, Girl, Chek, and Ayahchik who offered me unconditional love and support throughout the course of this thesis.

I also dedicate this thesis to Ayah Ku who has been my friend, guide and philosopher.

Finally, to Schuque who has always stood by me, helped me and believed that I could do it.

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Faculty : Science and Technology

The aim of this research project was to produce bio-based lubricant from triacylglycerol of palm olein and jatropha oil. The process involves three steps of reactions; epoxidation, oxirane ring opening, and followed by alkoxylation. For the epoxidation, the base oil was reacted with peracetic acid generated for safety reasons *in situ* by reacting acetic acid with hydrogen peroxide in the presence of sulfuric acid as a catalyst. Before alkoxylation, the epoxide needs to undergo ring opening process in water medium using perchloric acid as the catalyst. The hydroxylated intermediate was then reacted with acetic anhydride using homogeneous catalyst. Three different catalysts, pyridine, 4-dimethylaminopyridine, and 3-methylpyridine, were utilized to test their performances in this esterification process. The end-product of bio-based lubricant, the intermediates and the base oil were characterized using spectroscopic techniques, Infrared and Nuclear Magnetic Resonance, and physicochemical analyses such as viscosity, pour point, flash point, acid value, iodine value and thermal stability. Results showed that all the three catalysts successfully produced the alkoxylated lubricants for both palm olein and jatropha where pyridine was found

to be the best catalyst. Due to a high viscosity of the products, especially for jatropha based, these lubricants are suitable to be utilized for high operating temperature lubrication. Results of *ab initio* calculation using Gaussian 03 at the theoretical level of RHF/6-311G (d,p) were used in the very best way to ease the interpretation of the IR and NMR spectra of the end-products, intermediates and the base oils. In some cases, results of Gaussian were combined with Monte-Carlo procedure, Mulliken Population Analysis, and Natural Bond Orbital Theory to explain the irregularities of experimental results such as in justifying the different molar ratio of hydrogen peroxide consumed by palm olein and jatropha during epoxidation, and also in describing the fluctuation of viscosities due to functional group changes.

Abstrak tesis yang dikemukakan kepada Senat Universiti Malaysia Terengganu sebagai memenuhi keperluan untuk Ijazah Sarjana Sains

KAJIAN SECARA EXPERIMEN DAN TEORI TERHADAP PENGHASILAN MINYAK PELINCIR BIO BERASASKAN MINYAK SAWIT OLIN DAN MINYAK JATROPHA

SITI BALQIS CHE OTHMAN

Oktober 2010

Pengerusi : Prof Madya Dr. Ku Halim Ku Bulat

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Tujuan penyelidikan adalah untuk menghasilkan minyak pelincir berasaskan minyak tumbuhan dari triacylgliserida minyak sawit olin dan minyak jatropha. Proses melibatkan tiga langkah utama iaitu pengepoksidaan, pembukaan gelang oksirana dan diikuti proses pengalkoksilan. Bagi proses pengalkoksilan, minyak asas ditindakbalaskan dengan asid perasetik yang dihasilkan secara *in situ* atas untuk sebab-sebab keselamatan secara menindakbalaskan asid asetik dengan hydrogen peroksida dengan kehadiran asid sulfurik sebagai mangkin. Sebelum melakukan proses pengalkoksilan, gelang oksirana perlu dibuka untuk menghasilkan kumpulan hidroksil menggunakan asid perklorik sebagai mangkin di dalam medium air. Bahan perantara minyak terhidroksil ini kemudiannya ditindakbalaskan dengan asetik anhidrida menggunakan mangkin homogen. Tiga jenis mangkin iaitu piridina, 4-dimetilaminopiridina, dan 3-metilpiridina telah diuji prestasinya dalam memangkinkan proses pengalkoksilan ini. Hasil akhir minyak pelincir, bahan perantara dan minyak asas dicirikan menggunakan kaedah spektrokopi infra merah

dan resonans magnet nuklues dan juga melalui kaedah fizikokimia seperti kelikatan, takat tuang, takat kilat, nilai asid, nilai iodin dan kestabilan terma menggunakan kaedah analisis termogravimetri. Keputusan eksperimen menunjukkan bahawa ketiga-tiga mangkin telah berjaya menghasilkan minyak pelincir teralkoksilan bagi kedua-dua minyak sayuran, sawit olin dan minyak jatropha, dimana piridina merupakan mangkin terbaik. Disebabkan minyak pelincir yang terhasil ini mempunyai nilai kelikatan yang tinggi, terutamanya minyak jatropha, minyak pelincir teralkoksilan ini sesuai digunakan untuk jenis pelinciran bersuhu tinggi. Hasil pengiraan ab initio kuantum mekanik menggunakan Gaussian 03 pada aras teori RHF/6-311G(d,p) telah berjaya digunakan untuk membantu memudahkan penginterpretasian spektra infra merah dan resonans magnet nukleus untuk hasil akhir minyak pelincir, bahan perantara dan juga minyak asas. Dalam beberapa kes tertentu, hasil dari pengiraan Gaussian digabungkan dengan kaedah Monte-Carlo, Analisis Populasi Mulliken dan Analisis Teori Orbital Ikatan untuk menerangkan ketidakseragaman dalam hasil eksperimen seperti dalam menjelaskan kenapa terdapat perbezaan nisbah molar hidrogen peroksida yang diperlukan oleh minyak sawit olin dan jatropha semasa pengepoksidaan, dan juga untuk menerangkan turun naik nilai kelikatan apabila berlaku perubahan kumpulan berfungsi rantai minyak triacylgliserida.