UNIVERSITI PUTRA MALAYSIA

SYNTHESIS OF JATROPHA BIOLUBRICANT USING SODIUM METHOXIDE AS CATALYST

MOHAMAD FAIZ MUKHTAR BIN GUNAM RESUL

SYNTHESIS OF JATROPHA BIOLUBRICANT USING SODIUM METHOXIDE AS CATALYST

by

MOHAMAD FAIZ MUKHTAR BIN GUNAM RESUL

Thesis submitted to the School of Graduate studies, Universiti Putra Malaysia, In Fulfillment of the Requirements for the Degree of Master of Science

August 2012

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

SYNTHESIS OF JATROPHA BIOLUBRICANT USING SODIUM METHOXIDE AS CATALYST

by

MOHAMAD FAIZ MUKHTAR BIN GUNAM RESUL

August 2012

Chairman : Tinia Idaty Mohd. Ghazi, PhD

Faculty : Engineering

Initially, jatropha methyl ester (JME) was synthesized from extracted jatropha crude oil (JCO) and methanol via transesterification, using sodium hydroxide (NaOH) as catalyst. JME produced, was later reacted with a type of polyol, trimethylolpropane (TMP) to produce jatropha biolubricant, a jatropha based triester, via transesterification using sodium methoxide ($NaOCH_3$) as catalyst. The produced jatropha biolubricant was analyzed by using gas chromatography (GC), differential scanning calorimetry (DSC), pour point test, wear test, viscosity test and biodegradability.

The optimum condition to synthesize jatropha biolubricant were as follows; reaction temperature of 150 °C, reactant molar ratio of 3.5:1 and catalyst loading of 0.8% (wt/wt). The kinetic of reaction was studied by varying the operating temperature from 120°C to 200°C, indicating a second order reaction with overall reaction

constant found at 3.175 x 10^{-1} (% wt/wt.min.°C)$^{-1}$. The thermal-oxidative stability was observed at T_{on}, 325°C, with the improvement of 56% from the JCO's thermal-oxidative stability which is at 205°C. Physical test on the viscosity index (VI) of the jatropha biolubricant, which was calculated at 183, revealed that the viscosity of the oil does not significantly change upon the variation of temperature. The improvement of pour point from 8°C of JCO to -6°C of jatropha biolubricant justified the chemical modification applied in this research. Furthermore, wear test shows a slightly better improvement of jatropha biolubricant over JCO whereby from the four-ball test, the average scar diameter for jatropha biolubricant was 0.33mm compared to 0.36mm for JCO. In terms of environmental friendliness, the biodegradability test shows that jatropha biolubricant was able to degrade more than 60% as required to be labeled as biodegradable material. Overall, the chemical modification was able to improve the utilization of jatropha based lubricant and resulting in improvements to the chemical and physical properties studied.

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

SINTESIS PELINCIR BERASASKAN JATROPHA MENGGUNAKAN NATRIUM METOKSIDA SEBAGAI PEMANGKIN

Oleh

MOHAMAD FAIZ MUKHTAR BIN GUNAM RESUL

Ogos 2012

Pengerusi: Tinia Idaty Mohd. Ghazi, PhD

Fakulti: Kejuruteraan

Pada peringkat permulaan, ester metil jatropha (JME) disintesis dari tindak balas transesterifikasi antara minyak mentah jatropha dan metanol dengan menggunakan natrium hidroksida (NaOH) sebagai pemangkin. JME yang terhasil kemudiannya di tindakbalaskan dengan sejenis poliol iaitu trimetilolpropana (TMP) bagi menghasilkan pelincir berasaskan jatropha iaitu sejenis triester, melalui kaedah transesterifikasi dengan menggunakan sodium metoksida ($NaOCH_3$) sebagai pemangkin. Pelincir asas jatropha yang terhasil kemudian dianalisa menggunakan kromatografi gas (GC), kalorimetri pengimbas pembezaan DSC, ujian takat tuang, ujian kehausan, ujian kelikatan dan biodegradasi.

Didapati, keadaan optimum bagi penghasilan pelincir asas jatropha adalah seperti berikut; suhu 150°C, nisbah molar bahan tindakbalas 3.5:1 dan amaun pemangkin sebanyak 0.8% (wt/wt). Kinetik tindak balas dikaji pada suhu operasi di antara 120°C hingga 200°C, yang menunjukkan tindak balas tertib kedua dengan kadar malar secara keseluruhan ialah 3.175 x 10^{-1} (% wt/wt.min.°C)$^{-1}$. Kadar kestabilan terma-oksidatif yang optimum didapati pada T_{on}, 325°C, dengan jumlah kenaikan sebanyak 56% berbanding kadar kestabilan terma-oksidatif JCO pada 205°C. Ujian fizikal indeks kelikatan (VI) pelincir asas jatropha yang didapati berjumlah 183, menunjukkan bahawa kelikatan minyak tersebut tidak berubah dengan ketara bagi setiap perubahan dalam suhu. Perubahan takat tuang dari 8°C oleh JCO kepada 6°C oleh pelincir asas jatropha telah memberi justifikasi kepada pengubahsuaian kimia yang diaplikasikan dalam penyelidikan ini. Tambahan lagi, ujian kehausan menunjukkan sedikit kenaikan prestasi pelincir asas jatropha berbanding JCO dimana melalui ujian empat bola, diameter calar purata untuk pelincir asas jatropha ialah 0.33mm berbanding 0.36mm oleh JCO. Dari segi kemesraan alam, ujian biodegradasi menunjukkan bahawa pelincir asas jatropha berupaya terdegradasi lebih dari 60% bagi memenuhi keperluan untuk dilabel sebagai bahan biodegradasi. Secara keseluruhan, modifikasi kimia yang dilakukan telah berjaya mengubah penggunaan pelincir berasaskan jatropha, dimana penambahan prestasi dicapai dalam setiap ciri-ciri kimia dan fizikal yang dikaji.

ACKNOWLEDGEMENT

Foremost, I would like to express my sincere gratitude to my supervisor Dr. Tinia Idaty Mohd. Ghazi for the continuous support of my M.Sc study and research, for her patience, motivation, enthusiasm, and knowledge. Her guidance has helped me through all the time of the study and writing of this thesis. I could not have imagined having a better supervisor and mentor for my study. Besides my supervisor, I would like to thank the rest of my supervisory committee, Prof. Azni Idris and Prof. Robiah Yunus, for their encouragement, insightful comments, and consistent help.

My sincere thanks also goes to my fellow labmates, Nabilah binti Mohd Sofian, Taha Kadir, Elaine Koh May Ying, Ismail Nasir, Al-Rumaisa' and Amir for the stimulating discussions, for the tireless lab work we were working together, and for all the fun we have had in the last four years.

Last but not the least, I would like to thank my family: my parents Gunam Resul and Shamshad Begum, for the guidance, encouragement and supporting me spiritually throughout my life.

I Certify that a Thesis Examination Committee has met on (insert the date of viva voce) to conduct the final examination of **Mohamad Faiz Mukhtar** on his thesis entitled **Synthesis of Jatropha Biolubricant using Sodium Methoxide as Catalyst** in accordance with the Universities and University Colleges Act 1971 and the Constitution of the Universiti Putra Malaysia [P.U.(A) 106] 15 March 1998. The committee recommends that the student be awarded the Master of Science.

Members of the Thesis Examination Committee were as follows:

Name of Chairperson, PhD
Title (eg:Prof)
Name of Faculty
Universiti Putra Malaysia
(Chairman)

Name of Examiner 1, PhD
Title (eg:Prof)
Name of Faculty
Universiti Putra Malaysia
(Internal Examiner)

Name of External Examiner, PhD
Title (eg:Prof)
Name of Department
Name of Organization
Country
(External Examiner)

SEOW HENG FONG, PhD
Professor and Deputy Dean
School of Graduate Studies
Universiti Putra Malaysia

Date:

This thesis was submitted to the Senate of Universiti Putra Malaysia and has been accepted as fulfillment of the requirement for the degree of Master of Science. The members of the Supervisory Committee were as follows:

Tinia Idaty Mohd. Ghazi, PhD
Lecturer
Faculty of Engineering
Universiti Putra Malaysia
(Chairman)

Azni Bin Hj Idris, PhD
Professor
Faculty of Engineering
Universiti Putra Malaysia
(Member)

Robiah Binti Yunus, PhD
Professor
Faculty of Engineering
Universiti Putra Malaysia
(Member)

_____

BUJANG BIN KIM HUAT, PhD
Professor and Dean
School of Graduate Studies
Universiti Putra Malaysia

Date:

DECLARATION

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

MOHAMAD FAIZ MUKHTAR

Date: 9 August 2012

TABLE OF CONTENTS