



UNIVERSITI PUTRA MALAYSIA

**PRODUCTION OF SYNTHETIC BASED DRILLING
FLUID FROM PALM OIL**

NOR SAIFUL HAFIZ BIN ABDUL HABIB

FK 2013 46



**PRODUCTION OF SYNTHETIC BASED DRILLING
FLUID FROM PALM OIL**

NOR SAIFUL HAFIZ BIN ABDUL HABIB

**MASTER OF SCIENCE
UNIVERSITI PUTRA MALAYSIA**

2013



PRODUCTION OF SYNTHETIC BASED DRILLING FLUID FROM PALM OIL

By

NOR SAIFUL HAFIZ BIN ABDUL HABIB

Thesis Submitted to the School of Graduate Studies, Universiti Putra Malaysia,
in Fulfilment of the Requirements for the Degree of Master of Science

April, 2013

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

**PRODUCTION OF SYNTHETIC BASED DRILLING FLUID FROM
PALM OIL**

By

NOR SAIFUL HAFIZ ABDUL HABIB

April 2013

Chairman : Prof RobiahYunus, PhD

Faculty : Engineering

The use of ester as a base fluid in synthetic base fluids (SBF) has become a trend in drilling operations due to many advantages compared to the conventional drilling fluids. The production of ester for SBF from vegetable oils is found to be the most successful invention in terms of environmentally friendliness and drilling costs since the vegetable oil is biodegradable and shows competitive performance as drilling fluids. The synthesis of ester as biodegradable base oil was conducted via transesterification reaction of palm oil methyl ester (POME) with 2-ethylhexanol (2EH). High oleic POME was used in the reaction. The selection of 2-ethylhexanol as the raw material is to produce the low viscosity but branched ester. The catalyst used in the reaction was sodium methoxide. Sodium methoxide which is a base catalyst proved to have better performance in terms of productivity and reactivity compared to many acid catalysts. The reaction was carried out at different temperatures (70°C to 140°C) and the vacuum pressure was fixed at 1.5 mbar.

The reactant molar ratio of POME to 2EH was 1:1.5, 1:2 and 1:2.2. Since the reaction with 2-ethylhexanol only involves a single reaction, this transesterification reaction is considered as a single step reversible reaction; hence, the reaction was completed in a very short time. 98% palm based EHE were successfully synthesized in less than 30 minutes at 1.5 mbar pressure, 70°C, and 1:2 molar ratio of POME to 2EH. The analysis of the product (EHE) was performed using gas chromatography which was operated at an initial temperature of 80°C, held for 3 minutes then increased at 6°C/min to 340°C and held for another 8 minutes. The injector and detector temperature were set at 320°C and 340°C, respectively. This procedure provided a complete separation of the reaction products, palm based methyl ester and monoesters.

The reversible rate constant of the reaction is negligible, thus the equilibrium constant of the reaction is the rate constant. The rate constant of the reaction (k) obtained from the kinetics study was in the range of 0.44 s^{-1} to 0.66 s^{-1} . The activation energy of the reaction was 15.6 kJ.mol^{-1} . The preliminary investigations on the lubrication properties of drilling mud formulated with palm based EHE indicated that the base oil has a great potential to substitute the synthetic ester base oil for drilling fluid. Its high kinematic viscosity provides better lubrication to the drilling fluid compared to other ester-based oils. The pour point and the flash point are superior for the drilling fluid formulation. The plastic viscosity, High Pressure High Temperature filtrate loss and emulsion stability of the drilling fluid had given acceptable values, while gel strength and yield point could be improved by blending it with proper additives.

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

**PENGHASILAN LUMPUR PENGERUDIAN SINTETIK DARIPADA
MINYAK KELAPA SAWIT**

Oleh

NOR SAIFUL HAFIZ ABDUL HABIB

April 2013

Pengerusi : Prof Robiah Yunus, PhD

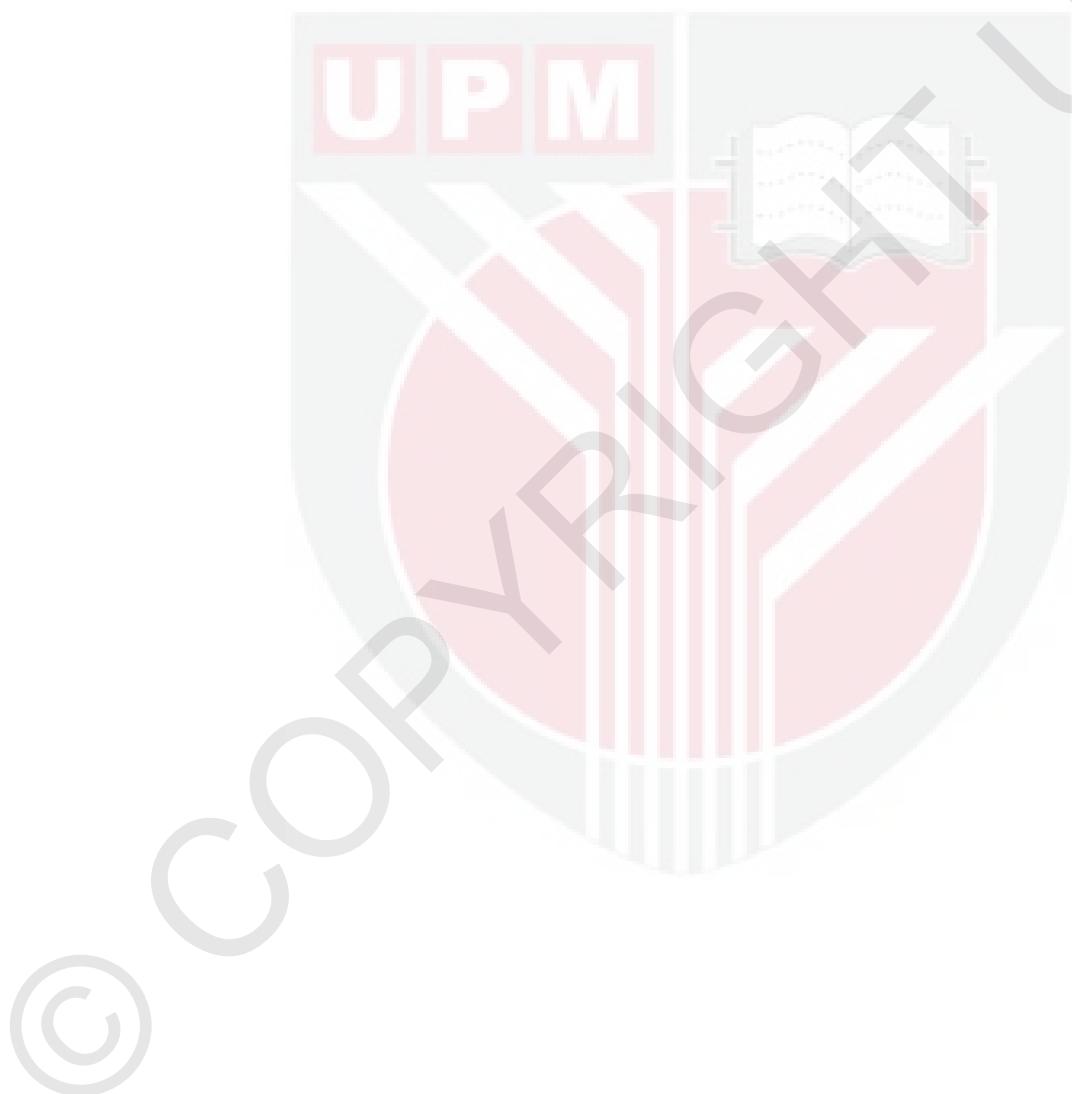
Fakulti : Kejuruteraan

Penggunaan ester sebagai bendaril asas dalam bendaril asas sintetik (SBF) telah menjadi satu trend dalam operasi-operasi penggerudian oleh kerana banyak kelebihan berbanding dengan bendaril penggerudian konvensional. Penghasilan ester untuk SBF daripada minyak-minyak sayuran merupakan penemuan terbaik dari segi mesra alam dan kos-kos penggerudian kerana minyak sayuran mempunyai kemampuan untuk terbiodegradasikan dan menunjukkan prestasi kompetitif sebagai bendaril penggerudian. Sintesis ester sebagai minyak asas terbiodegradasikan telah dijalankan melalui tindak balas transesterifikasi oleh minyak kelapa sawit metil ester (POME) dan 2-ethylheksanol (2EH). Minyak kelapa sawit metil ester tinggi oleik digunakan didalam tindakbalas ini. 2-ethylheksanol digunakan didalam tindak balas ini adalah untuk mendapatkan ester yang berkelikatan rendah dan bercabang. Pemangkin yang telah digunakan dalam tindak balas ini ialah natrium metoksida. Natrium metoksida yang merupakan pemangkin bes terbukti menunjukkan prestasi

yang lebih baik berbanding banyak pemangkin asid dari segi penghasilan produk dan kereaktifan. Tindak balas tersebut telah dilakukan pada suhu-suhu yang berbeza (70°C hingga 140°C) dan tekanan ditetapkan pada 1.5 mbar. Oleh kerana transesterifikasi melibatkan satu tindak balas sahaja, tindak balas ini dipertimbangkan sebagai langkah tindak balas berbalik yang tunggal. Oleh sebab itu, tindak balas tersebut telah lengkap dalam tempoh yang sangat pendek. Sebanyak 98% ester-ester etilheksil berasaskan kelapa sawit telah berjaya disintesikan dalam tempoh kurang daripada 30 minit di bawah tekanan 1.5 mbar, suhu 70°C , dan nisbah molar POME kepada 2EH 1:2. Analisis produk (etilheksil ester) telah dilakukan menggunakan gas kromatografi yang telah beroperasi pada suhu permulaan 80°C , ditangguhkan selama 3 minit, kemudian dinaikkan sebanyak $6^{\circ}\text{C}/\text{min}$ hingga 340°C dan ditangguhkan lagi selama 8 minit. Suhu bagi alat suntikan dan pengesan telah disetkan pada 320°C dan 340°C , secara urutannya. Langkah ini adalah untuk pemisahan lengkap bagi semua produk tindak balas, iaitu di dalam kes ini, minyak kelapa sawit metil ester dan beberapa monoester.

Pemalar kadar tindak balas berbalik bagi tindak balas ini diabaikan, maka pemalar keseimbangan tindak balas adalah pemalar kadar tindak balas. Pemalar kadar tindak balas (k) yang telah didapati daripada kajian kinetik adalah dalam lingkungan 0.44 hingga 0.66. Tenaga pengaktifan tindak balas tersebut adalah sebanyak $15.6 \text{ kJ}\cdot\text{mol}^{-1}$. Penyelidikan awal pada sifat-sifat pelincir pada lumpur gerudi yang telah diformulasikan dengan ester 2EH berasaskan kelapa sawit telah menunjukkan bahawa minyak asas tersebut mempunyai pontensi besar untuk menggantikan minyak asas sintetik ester yang sedia ada untuk bendalir penggerudian. Kelikatan

kinematiknya memberikan pelinciran lebih baik kepada bendalir penggerudian berbanding dengan minyak-minyak asas ester yang lain. Takat-takat tuang dan kilat minyak tersebut adalah lebih tinggi mutunya bagi formulasi bendalir penggerudian. Keputusan nilai-nilai kelikatan plastik, kerugian turasan HPHT dan kestabilan emulsi bagi bendalir adalah dapat diterima. Kekuatan gel dan titik alah boleh diperbaiki lagi melalui proses pengadunan dengan bahan campuran yang sesuai.



ACKNOWLEDGEMENTS

In the name of Allah, The Most Gracious, The Most Merciful. All praise goes to Allah for giving me the courage and patience to finish this study. My deepest appreciation and very sincere thanks to my supervisor, Professor Dr. Robiah Yunus for her supervision, continuous guidance, valuable advice, unlimited patience and understanding throughout the course of this study. I cannot adequately express my gratitude to my co-supervisor, Professor Taufiq Yap Yun Hin and Associate Professor Dr. Zurina Zainal Abidin for providing invaluable guidance and encouragement to help me accomplish the Master programme smoothly.

My high appreciation also goes to all lecturers and staff at the Department of Chemical and Environmental Engineering for their cooperation in providing all necessary facilities throughout this study. I am also grateful to Universiti Putra Malaysia for providing financial support under the Graduate Research Fellowship. Further gratitude goes to all my friends, especially Dr. Azhari, Ferra, Hamidah, Chang, Ummi, Hassan, Melina, Fatimah, Shamimi, Yayat, Atiqah and Dr. Umer for their guidance and motivation during the progress of this research. Not to forget, a big thanks to Dr. Sonny Irawan and Mr. Juhairi from UTP that allow and help me run my properties analysis.

Last but not least, I am most grateful to my beloved family, especially my parents Abdul Habib Alapitchay and Zaharah Dahlan for all the support, love and encouragement given that enable me to finish this thesis timely.

I certify that a Thesis Examination Committee has met on 5th of April 2013 to conduct the final examination of Nor Saiful Hafiz bin Abdul Habib on his thesis entitled “Production of Synthetic based Drilling Fluid from Palm Oil” in accordance with the Universities and University College 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:

Associate Professor Dr. Mohd Halim Shah bin Ismail
Faculty of Engineering
Universiti Putra Malaysia
(Chairman)

Professor Dr. Said Salah Eldin Hamed Elnashaie
Faculty of Engineering
Universiti Putra Malaysia
(Internal Examiner)

Associate Professor Dr. Salmiaton binti Ali
Faculty of Engineering
Universiti Putra Malaysia
(Internal Examiner)

Associate Professor Dr. Zuhairi
Faculty of Engineering
Universiti Sains Malaysia
(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 fulfilment of the requirement for the degree of Master of Science. The members of the Supervisory Committee are as follows:

Robiah Yunus, PhD

Professor

Faculty of Engineering
Universiti Putra Malaysia
(Chairman)

Taufiq Yap Yun Hin, PhD

Professor

Faculty of Science
Universiti Putra Malaysia
(Member)

Zurina Zainal Abidin, PhD

Associate 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 institution.

NOR SAIFUL HAFIZ ABDUL HABIB

Date:



TABLE OF CONTENTS

| | Page |
|--|------|
| ABSTRACT | ii |
| ABSTRAK | iv |
| ACKNOWLEDGEMENTS | vii |
| APPROVAL | viii |
| DECLARATION | x |
| LIST OF TABLES | xiii |
| LIST OF FIGURES | xiv |
| LIST OF ABBREVIATIONS | xvi |
| CHAPTER | |
| 1 INTRODUCTION | |
| 1.0 Background of Study | 1 |
| 1.1 Objectives and Scope of Work | 5 |
| 1.2 Thesis Outline | 6 |
| 2 LITERATURE REVIEW | |
| 2.0 Introduction | 7 |
| 2.1 Types of Drilling Fluid | 9 |
| 2.2 Synthetic-Based Fluid | 12 |
| 2.3 Vegetable Oil-Based Drilling Fluid | 13 |
| 2.4 Prior Research on Ester as Base Fluid | 14 |
| 2.5 Properties of Drilling Fluid | 21 |
| 2.6 Advantages of SBF over WBM and OBF | 24 |
| 2.7 Replacement of Mineral oil by Synthetic ester | 26 |
| 2.8 Transesterification | 29 |
| 2.8.1 Reaction Kinetics | 32 |
| 2.8.2 Kinetic of Synthesis of Palm Oil-Based Ester | 36 |
| 2.8.3 Arrhenius Equation and Activation Energy | 39 |
| 2.9 Conclusion | 39 |
| 3 RESEARCH METHODOLOGY | |
| 3.0 Introduction | 41 |
| 3.1 Raw Material | 42 |
| 3.1.1 Palm Oil Methyl Ester | 42 |
| 3.1.2 2-Ethylhexanol | 43 |
| 3.1.3 Catalyst | 43 |
| 3.2 Experimental Procedures | 44 |
| 3.2.1 Synthesis of EHE | 44 |
| 3.2.2 Product Purification | 46 |
| 3.3 Gas Chromatography : Analysis of Ester Content | 47 |
| 3.4 Sampling Method for Kinetic Study | 48 |
| 3.5 Evaluation of EHE Properties | 49 |
| 3.5.1 Viscosity and Viscosity Index for EHE | 49 |
| 3.5.2 Flash Point | 49 |
| 3.6 Formulation of Drilling Fluid | 50 |
| 3.6.1 Mud Balance Density | 50 |

| | | |
|---------------------------|---|-----|
| 3.6.2 | Rheology of Drilling Fluid | 50 |
| 3.6.3 | High Pressure High Temperature Filter Press | 51 |
| 3.6.4 | Water, Oil and Solid Ratio | 52 |
| 3.6.5 | Electrical Stability | 52 |
| 4 | RESULTS AND DISCUSSIONS | |
| 4.0 | Introduction | 53 |
| 4.1 | Synthesis of Palm oil-based Ester as Base Oil | 53 |
| 4.2 | Preliminary Synthesis of EHE | 54 |
| 4.3 | Optimization of Reaction Condition | 58 |
| 4.3.1 | Effect of Reaction Time | 59 |
| 4.3.2 | Effect of Reaction Temperature | 60 |
| 4.3.3 | Effect of Reactants Molar Ratio | 61 |
| 4.3.4 | Effect of Amount of Catalyst | 63 |
| 4.4 | Synthesis of EHE for Kinetic Study | 65 |
| 4.4.1 | Determination of Kinetic Model | 69 |
| 4.4.2 | Activation Energy | 76 |
| 4.5 | Formulation of Drilling Fluid Using EHE as Base Oil | 77 |
| 4.5.1 | Properties of Ester-based Fluid | 78 |
| 4.5.2 | Ester base Fluid Composition | 79 |
| 4.5.3 | Formulation of Ester base Fluid | 83 |
| 4.6 | Selection of Formulated Mud as Best Drilling Fluid | 87 |
| 5 | CONCLUSION AND RECOMENDATIONS | |
| 5.0 | Conclusion | 91 |
| 5.1 | Recommendation for the Future Works | 93 |
| REFERENCES | | 95 |
| APPENDICES | | 104 |
| BIODATA OF STUDENT | | 115 |