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MASTER OF SCIENCE
UNIVERSITI PUTRA MALAYSIA

2007

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By

NOR HALALIZA BT ALIAS

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

May 2007

DEDICATED TO

MUMMY, DADDY and my family members, with love

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

FORMULATION OF PALM OIL-BASED SYNTHETIC LUBRICANT AS HYDRAULIC FLUID

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May 2007

Chairman : Associate Professor Robiah Yunus, PhD

Faculty : Engineering

Increasing attention to the environmental issues drives the lubricant industry to choose the vegetable-based lubricants which are biodegradable compared to mineral-based fluids. Hydraulic fluids represent one of the most important groups of industrial lubricants, being widely used in industrial hydraulic systems, particularly for machine tools, steering gears, etc. However, the inherent properties of vegetable oil such as poor oxidative stability and high pour point have hindered its use.

In this study, trimethlolpropane (TMP) ester, which is derived from palm based methyl ester (POME), was used as the base fluid. The purpose of this study is to determine the optimum formulation for palm oil-based synthetic lubricant as hydraulic fluid by using suitable additives that can improve the lubricating properties in accordance to the standard regulations.

To determine the range of operating temperature suitable for hydraulic fluid purposes, the test was conducted using bench test. Two types of antioxidant additives, Additive A and Additive B were used at temperature ranged from 80°C, 90°C, 95°C and 100°C. The effects of additives on degradation of oil at these temperatures were determined based on the tests done at 0 hour, 24 hours, 48 hours, 72 hours, 96 hours, 200 hours, 400 hours, 600 hours and 800 hours.

The oxidative stability of oil was evaluated based on the determination of the Total Acid Number (TAN) and viscosity tests. In general, the unadditived based-oil began to degrade after 200 hours. However, the additived oil was quite stable even after 800 hours of operation. The best formulation was obtained at 95°C by using 1.5% of Additive A. Two effects have been studied in this research, i.e. the effect of temperatures and also the effect of additives on TAN and viscosity value. Both TAN and viscosity value increased with heating temperature. Meanwhile, the results have proven that Additive A performs better compared to Additive B, based on the TAN and viscosity results. The final TAN value for additived oil was only 0.54 mg KOH/g as compared to 7.46 mg KOH/g for unadditived oil. Other lubrication properties such as kinematic viscosity and viscosity index were also studied. After 800 hours of operation, the final kinematic viscosity of the oil at 40°C and 100°C were 38.67 mm^2/s and 7.9 mm^2/s, respectively, as compared to 86.02 mm^2/s and 13.5 mm^2/s for unadditived oil. Meanwhile, the viscosity index (VI) was almost unchanged at 185.

The standard working temperature used in the lab scale hydraulic test rig was 60°C. The maximum pressure 910 psi was able to push up the 20 kg load throughout the system, while the minimum pressure was maintained at 240 psi after the load was being pulled down. As the hydraulic cylinder moves up and down, the oil experienced shear stress ranged between 1 rpm to 2500 rpm. At 0 hour, the TAN value, kinematic viscosity of the oil at 40°C and 100°C were 0.22 mg KOH/g, 35.8 mm^2/s and 7.5 mm^2/s, respectively. Meanwhile, after 800 hours operation, the final TAN value was 0.65 mg KOH/g, whereas the final kinematic viscosity of the oil at 40°C and 100°C were 24.7 mm^2/s and 6.3 mm^2/s, respectively. The VI values ranged between 185 up to 221.

Other tests on the formulated hydraulic fluid were also conducted, such as wear and friction, pour point, thermal stability by using TGA-DTA and filterability. These tests are important to ensure that this formulated hydraulic fluid confirm to the standard regulations. The pour point of the formulated oil was recorded at 10°C. Whereas, the wear scar diameter (WSD) in wear and friction tests were 1.0 mm under 15kg load and 3.24 mm under 40kg load. Both readings were recorded at 0 hour. Filterability test shows that some particles were produced in the hydraulic system after 800 hours operation due to the wear and tear. However, the amount of particles before (at 0 hour) and after the operation (at 800 hours) were only 0.32 g and 0.65 g, respectively.

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

PERUMUSAN PELINCIR SINTETIK MINYAK ASAS-SAWIT SEBAGAI BENDALIR HIDRAULIK

Oleh

NOR HALALIZA BT ALIAS

Mei 2007

Pengerusi : Profesor Madya Robiah Yunus, PhD

Fakulti : Kejuruteraan

Peningkatan perhatian terhadap isu alam sekitar menyebabkan industri pelincir memilih pelincir yang berasaskan tumbuhan, yang mana adalah biorosot berbanding dengan bendalir yang berasaskan mineral. Bendalir hidraulik merupakan salah satu daripada kumpulan penting dalam industri pelincir, yang digunakan secara meluas dalam industri sistem hidraulik; khususnya untuk alat mesin, kemudi gear dan lain-lain. Walaubagaimanapun, sifat-sifat terwujud minyak tumbuhan seperti kekurangan kestabilan beroksida dan takat tuang yang tinggi telah menyekat penggunaannya.

Dalam kajian ini, Trimethlolpropane (TMP) Ester yang telah diterbitkan daripada bahan asas minyak sawit Metil Ester (ME) telah digunakan sebagai bendalir asas. Tujuan

kajian ini adalah untuk menentukan perumusan optimum untuk pelincir sintetik minyak asas sawit sebagai bendalir hidraulik dengan menggunakan bahan tambah yang sesuai yang boleh membaiki sifat-sifat pelincir bertepatan dengan peraturan piawai.

Untuk memperoleh julat suhu kendalian yang sesuai untuk kegunaan bendalir hidraulik, ujian telah dijalankan dengan menggunakan ujian meja. Dua jenis bahan tambah antibahan pengoksidaan, bahan tambah A dan bahan tambah B digunakan pada julat suhu 80°C, 90°C, 95°C dan 100°C. Kesan bahan tambah ke atas penurunan minyak pada suhu tersebut telah ditentukan berdasarkan ujian-ujian yang telah dijalankan pada 0 jam, 24 jam, 48 jam, 72 jam, 96 jam, 200 jam, 400 jam, 600 jam dan 800 jam.

Kestabilan beroksida minyak telah dinilaikan berdasarkan penentuan jumlah nombor keasidan (TAN) dan ujian kelikatan. Secara umumnya, minyak tanpa bahan tambah mula menurun selepas 200 jam. Walaubagaimanapun, minyak dengan bahan tambah adalah agak stabil walaupun selepas 800 jam pengendalian. Perumusan terbaik telah diperoleh pada suhu 95°C dengan menggunakan 1.5% bahan tambah A. Dua kesan telah dikaji dalam penyelidikan ini; iaitu kesan suhu dan juga kesan bahan tambah ke atas nilai jumlah nombor keasidan (TAN) dan kelikatan. Kedua-dua nilai jumlah nombor keasidan (TAN) dan kelikatan meningkat dengan suhu pemanasan. Sementara itu, keputusan telah membuktikan bahawa prestasi bahan tambah A adalah lebih baik berbanding bahan tambah B, berdasarkan keputusan jumlah nombor keasidan (TAN) dan kelikatan. Nilai akhir jumlah nombor keasidan (TAN) bagi minyak dengan bahan tambah adalah hanya 0.54 mg KOH/g berbanding dengan 7.46 mg KOH/g bagi minyak tanpa bahan tambah. Sifat-sifat pelinciran yang lain seperti kelikatan kinematik dan kelikatan indek juga telah dikaji. Selepas 800 jam pengendalian, nilai akhir kelikatan kinematik pada 40°C dan 100°C masing-masing adalah 38.67 mm^2/s dan 7.9 mm^2/s, berbanding dengan 86.02 mm^2/s dan 13.5 mm^2/s bagi minyak tanpa bahan tambah. Sementara itu, kelikatan indek adalah hampir kekal tidak berubah pada 185.

Suhu bekerja piawai yang telah digunakan dalam rig ujian hidraulik berskala makmal adalah 60°C. Tekanan maksimum 910 psi adalah mampu untuk menolak beban 20kg ke atas sepanjang sistem, sementara tekanan minimum adalah kekal pada 240 psi selepas beban tersebut ditarik ke bawah. Semasa sistem hidraulik bergerak ke atas dan ke bawah, minyak tersebut telah mengalami tegasan ricih berjulat diantara 1 rpm hingga 2500 rpm. Pada 0 jam, jumlah nombor keasidan (TAN), kelikatan kinematik minyak pada 40°C dan 100°C masing-masing adalah 0.22 mg KOH/g, 35.8 mm^2/s and 7.5 mm^2/s. Sementara itu, selepas 800 jam pengendalian, nilai akhir jumlah nombor keasidan (TAN) adalah 0.65 mg KOH/g, manakala nilai akhir kelikatan kinematik pada 40°C dan 100°C masing-masing adalah 24.7 mm^2/s dan 6.3 mm^2/s. Julat kelikatan indek adalah di antara 185 hingga 221.

Ujian-ujian lain ke atas bendalir hidraulik yang telah dirumuskan juga telah dijalankan, seperti kehausan dan geseran, takat tuang, kestabilan haba dengan menggunakan TGA-DTA dan penurasan. Ujian-ujian ini adalah penting untuk memastikan bendalir hidraulik yang telah dirumuskan sah mengikut peraturan. Takat tuang minyak yang telah dirumuskan telah dicatatkan pada 10°C. Manakala, kehausan parut garis pusat (WSD) dalam ujian kehausan dan geseran adalah 1.0 mm dibawah beban 15kg dan 3.24 mm dibawah beban 40kg. Kedua-dua nilai dicatatkan pada 0 jam. Ujian penurasan menunjukkan bahawa sejumlah zarah telah dihasilkan didalam sistem hidraulik selepas 800 jam pengendalian yang disebabkan oleh kehausan dan kekoyakan. Walaubagaimanapun, jumlah zarah sebelum (pada 0 jam) dan selepas pengendalian (pada 800 jam) masing-masing adalah hanya 0.32 g dan 0.65 g.

ACKNOWLEDGEMENTS

Alhamdulillah. First of all, I would like to express heartiness gratitude, appreciation and deep thanks to my supervisor, Assoc. Prof. Dr. Robiah Yunus, for her guidance, helpful advise, generous encouragement, never-ending patience, kind attention and willingness to assists me throughout this research. Without her valuable advice and support, it would not be possible for me to complete my research. I have learnt a lot of useful knowledge from her throughout this research.

I am also grateful to numerous individual who gave technical support and sincere assistance especially during hydraulic lab work. My sincere appreciation also goes to all Chemical Engineering master students year 2003-2006, who have helped and guided me throughout my studies.

Last but not least, my greatest appreciation will always go to my parents and my family members for their overwhelming encouragement, support and care given to me throughout this research.

I certified that an examination committee has met on 15th May 2007 to conduct the final examination of Nor Halaliza Binti Alias on her Master of Science thesis entitled "Formulation of Palm Oil-Based Synthetic Lubricant as Hydraulic Fluid" in accordance with Universiti Pertanian Malaysia (Higher Degree) Act 1980 and Universiti Pertanian Malaysia (Higher Degree) Regulation 1981. The committee recommended that the candidate be awarded the relevant degree. The Committee Members for the candidate are as follows:

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This thesis 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:

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Date: 13 September 2007

DECLARATION

I hereby declare that the thesis is based 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.

NOR HALALIZA BINTI ALIAS

Date: 6 August 2007

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LIST OF ABBREVIATIONS

ASTM	American Society for Testing and Materials
DE	Diester
HM	Hydraulic Mineral
KOH	Potassium Hydroxide
ME	Monoester
OEM	Original Equipment Manufacturers
PME	Palm-oil Methyl Ester
PP	Pour Point
RBD	Refined Bleached Deodorized
SAE	Society of Automotive Engineers
T_{on}	Onset Temperature
TAN	Total Acid Number
TE	Triester
TGA-DTA	Thermogravimetry with Differential Thermal Analysis
TMP	Trimethylolpropane
TMPE	Palm-based Trimethylolpropane Ester
VI	Viscosity Index

CHAPTER 1

INTRODUCTION

1.1 Background

All mechanical equipments must be lubricated. The purpose of this lubrication is to reduce friction and wear. If not controlled, these can lead to inefficiencies, damage and ultimately to equipment seizure. Pictorial records depicting the use of lubricants date as far back as 1650 B.C. Analysis of the residue from chariot axle hubs suggests that the use of animal fat as a lubricant dated as early as 1400 B.C (Rizvi, 1992). This practice continued until 1859 when petroleum-based lubricants became available. Most modern lubricants are petroleum-based although vegetable oils and animal fats are also used.

However in the past few decades, petroleum price has become unstable due to war and depleting reserves. In times of crude oil shortage such as when a Middle Eastern conflict occurs, there is always a rush to develop alternative bases. Nevertheless, once the conflict is resolved and crude oil price stabilize, this impetus is removed. Furthermore people are now more conscious with the impact of hazardous and toxic materials on the environment which lead to an increase demand for environmental friendly lubricants. It is reported that 1.2 to 2.3 million gallons of hydrocarbon oil are dropped, leaked and spilled annually (Wilson, 1998). Therefore using plant oils as mixture of base stock and/ or additives is a good alternative where environmental impact is of concern.

Increasing attention to the environmental issues drives the lubricant industry to increase the ecological friendliness of its products. For the last decade, the industry has been trying to formulate biodegradable lubricants with technical characteristics superior to those based on mineral oil (petroleum). Volumes of lubricants, especially engine oils and hydraulic fluids, are relatively large and most of them are based on mineral oils. Lubricants based on vegetables oils still comprise a narrow segment; however, they are finding their way into such applications as chainsaw bar lubricants, drilling muds and oils, straight metalworking fluids, food industry lubricants, open gear oils biodegradable grease, hydraulic fluids and marine oils. (Asadauskas and Erhan, 2000).

At least 13 independent North American companies and cooperatives are commercially producing plant-based lubricants. These groups face the daunting task of introducing a new product into a market dominated by huge petroleum companies. Globally, the top three lubricant producers—Shell, Exxon/Mobil and BP Amoco (who recently purchased Burmah Castrol)—hold at least 50 percent of the lubricant market. Interestingly, these companies do produce lubricants containing different percentages of vegetable oil, although their focus remains petroleum-based products. In order to ensure success, plant-based lubricant manufacturers must address a number of existing concerns, including negative industry preconceptions about plant-based lubricants (based on earlier and less successful technologies), unresolved questions about performance standards and testing protocols and the direction of governmental policy (Nelson, 2000).

According to Padavich and Honary (1995), hydraulic fluids, which are consumed at approximately 5 million metric tons per year rate in the US, have the highest need for