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IMPROVEMENT IN LUBRICATING PROPERTIES OF ENGINE OIL BY BLENDING WITH PALM OIL, TRIMETHYLOLPROPANE ESTER AND NANOPARTICLES

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By

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Engine oil plays an important role in reducing the friction and wear between the metal parts of an engine and acts as a coolant to lower the engine temperature. Mineral oil has been dominated the market as the base oil for engine oil due to its availability, and good performance. However, due to the decomposition issues and emission of toxic gases during its use, it becomes a source of pollution to the environment. The present study aimed at evaluating the alternative base oil for engine oil which is more environmentally friendly and effective in reducing wear and friction. This is achieved by blending the mineral oil with biodegradable base oils namely palm oil and trimethylolpropane (TMP) ester. The effect of adding nanomaterials on the friction properties were evaluated. The study also investigated the thermal degradation characteristics of the formulated engine oil in air and inert nitrogen.

The study was divided into three steps. In the first step, TMP ester was added to the palm oil at the percentages 2, 4, 5, 6, and 7 wt% to obtain the best wear and friction results. TMP ester can be used as an additive to improve the thermal and oxidative stability of palm oil. In the second step, the best formulation of the first step was added to the mineral oil at 10, 30 and 50% weight percentages. In the third step, the nanoparticles were added at 0.25, 0.5, and 0.75 wt% to the best formulation of the second step to improve the oil performance and reduce the coefficient of friction (COF) and wear scar diameter (WSD). The results showed that the blend of palm oil with 2 wt% TMP ester was the best formulation with the value of coefficient of friction at 0.0751 and 485 μm for WSD. Mixing this blend with 70 wt% mineral oil improved the results of COF from 0.1 to 0.0609 and WSD 565.2 μm to 318.9 μm. To further improve the performance of the newly formulated engine oil, five types of nanomaterials, copper oxide (CuO), aluminum oxide (AL_2O_3), Titanium silicon oxide ($TiSiO_4$), graphene and borosilicate nanoglass powder were added to the blend. The addition of 0.5 wt% of nanoglass powder reduced

COF and WSD further to 0.0565 and 295.5 µm respectively. Adding other nanomaterials yielded detrimental effects on the formulation, both COF and WSD increased significantly. This may be due to the additional friction attributed by the nanomaterials.

It was revealed that blending mineral oil with palm oil, TMP ester and nanomaterials improved all physicochemical properties, such as density, viscosity, viscosity index, TAN and pour point. It has also been proven that adding palm oil and TMP ester into the mineral oil improved the thermal stability which exceeded the thermal stability of the commercial lubricants. This is evidenced from the degradation profiles of newly formulated engine in air and nitrogen environment. The onset temperature improved from 334.7 ^{o}C to 409.6^{o}C. It is also worth-noting that the blend of palm oil-TMP ester with mineral oil without nanoglass showed better performance than the oil blended with nanoglass powder.

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

MENINGKATKAN PRESTASI MINYAK ENJIN DENGAN PENCAMPURAN MINYAK ASAS KELAPA SAWIT,TMP ESTER DAN PARTIKEL NANO

Oleh

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Minyak mesin memainkan peranan penting dalam mengurangkan geseran dan haus antara bahagian logam enjin dan berfungsi sebagai penyejuk untuk menurunkan suhu enjin. Minyak mineral telah menguasai pasaran sebagai minyak asas untuk minyak enjin kerana ketersediaan, kos rendah dan prestasi yang baik. Bagaimanapun, disebabkan oleh isu penguraian dan gas toksik yang dikeluarkan semasa penggunaan, ia telah menjadi sumber pencemaran terhadap alam sekitar. Kajian ini bertujuan untuk menilai minyak asas alternatif untuk minyak enjin yang lebih mesra alam dan berkesan dalam mengurangkan haus dan geseran. Hal ini dicapai dengan mencampurkan minyak mineral dengan minyak asas terbiodegradasi iaitu minyak kelapa sawit dan trimetilolpropana (TMP). Kesan menambah bahan nano pada sifat geseran juga dinilai. Kajian ini juga menyiasat ciri-ciri kemerosotan termal formulasi minyak enjin baik di udara maupun dalam nitrogen lengai.

Kajian ini dibahagikan ke dalam tiga langkah. Dalam langkah pertama, ester TMP dicampurkan ke dalam minyak kelapa sawit pada 2, 4, 5, 6, dan 7 peratus berat untuk mendapatkan hasil haus dan geseran terbaik. Tujuannya adalah untuk menggunakan ester TMP sebagai tambahan untuk meningkatkan kestabilan haba dan oksidatif minyak kelapa sawit. Dalam langkah kedua, rumusan terbaik langkah pertama ditambahkan pada minyak mineral pada 10, 30 dan 50 berat peratusan. Dalam langkah ketiga, partikel nano ditambahkan pada 0.25, 0.5, dan 0.75 berat peratusan pada formulasi terbaik langkah kedua untuk meningkatkan prestasi minyak dan mengurangkan pekali geseran (COF) dan diameter kehausan (WSD). Untuk menyiasat sifat minyak enjin yang dirumuskan, beberapa analisis telah dijalankan, di antaranya adalah analisis tribology empat bola, SEM, FTIR, FSEM, TAN, kelikatan, ketumpatan dan VI. Keputusan menunjukkan bahawa penambahan 2 wt% ester TMP pada minyak kelapa sawit adalah rumusan terbaik dengan nilai koefisien geseran pada 0.0751 dan 485 µm untuk WSD. Mencampurkan formulasi ini dengan minyak mineral 70% wt meningkatkan hasil COF pada 0.0609 dan WSD pada 318.9 µm. Untuk mempertingkatkan lagi prestasi minyak

enjin yang telah dirumuskan, lima jenis nanomaterial, CuO, AL_2O_3, $TiSiO_4$, graphene dan serbuk nanokaca borosilikat ditambahkan pada campuran. Penambahan 0.5 peratusan berat serbuk kaca nano mengurangkan COF dan WSD pada 0.0565 dan 295.5 µm. Akan tetapi, menambahkan bahan nano lain menghasilkan kesan buruk terhadap minyak formulasi, iaitu kedua-dua COF dan WSD meningkat dengan ketara. Ini mungkin disebabkan oleh peningkatan geseran yang disebabkan oleh bahan nano.

Kajian ini juga mendedahkan bahawa minyak mineral campuran dengan minyak kelapa sawit, ester TMP dan bahan nano memperbaiki semua sifat fizikokimia, seperti ketumpatan, kelikatan, indeks kelikatan, TAN dan takat tuang. Telah terbukti bahawa menambah minyak kelapa sawit dan ester TMP ke dalam minyak mineral telah meningkatkan kestabilan haba yang melampaui kestabilan terma pelincir komarsial. Ini dibuktikan dari profil kemerosotan formulasi enjin yang baru dalam udara dan nitrogen yang menunjukkan peningkatan yang ketara. Suhu permulaan bertambah baik dari 334.7 hingga 409.6^{o}C. Ia juga bernilai menyatakan bahawa campuran ester TMP minyak kelapa sawit dengan minyak mineral tanpa kaca nano menunjukkan prestasi yang lebih baik daripada minyak yang dicampur dengan kaca nano.

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TABLE OF CONTENTS

LIST OF TABLES

LIST OF FIGURES

LIST OF ABBREVIATIONS

Al_2O_3	Aluminum Oxide
AW	Anti-Wear
CFC	Chlorofluorocarbons Refrigerants
COF	Coefficient Of Friction
CuO	Copper Oxide
DSC	Differential Scanning Calorimeter Analysis
EP	Extreme Pressure
FESEM	Field-Emission Scanning Electron Microscope
FM	Friction Modifier
FT	Friction Torque
FTIR	Fourier Transform Infrared Spectroscopy
HFC	Hydrofluorocarbon Refrigerants.
MO	Mineral Oil
NG	Nano Glass
PO	Palm Oil
SEM	Scanning Electron Microscopy
TAN	Total Acid Number
TGA	Thermal Degradation Analysis
$TiSiO_4$	Titanium Silicon Oxide
VI	Viscosity Index
WSD	Wear Scar Diameter

CHAPTER 1

INTRODUCTION

1.1 Background

When any two hard objects move on each other, and there is a direct contact between their surfaces, this results in adhesion or friction that generates heat (Gunsel, et al., 1999; Nurul & Syahrullail, 2017). In internal combustion engines of motor vehicles, oils are used to lubricate the metal parts, to overcome the friction or reduce its effect. The oil forms a thin layer separating the metal parts and provides a distance between them to avoid the direct contact and thus reduces the resistance of the movement. This could prolong the life of engine parts and reduces the amount of fuel consumed, and increase the efficiency of the engine (J. Bart et al., 2013; Holmberg 2014). Engine oils are generally divided into three categories, mineral oil, synthetic oil and semi-synthetic oil (Shahabuddin et al., 2013).

Mineral oils are oils extracted from petroleum after refining processes (Osama et al., 2017). Synthetic oils are oils which chemical composition are modified in order to have different properties from the mineral oils. Since they are synthetic oils, they come with various names namely fully synthetic, super synthetic, 100% synthetic, long life semi-synthetic or blended oils which are a combination of the former. However, the industrial blending ratio typically does not exceed 30% and comes with the names of synthetic, semi-synthetic and synthetic blends. Several studies have been published to investigate the possibility of utilization vegetable oils as engine oils, for example jatropha, sunflower, flaxseed, corn and olive oils (Karmakar 2017; Nuraliza & Syahrullail, 2015; Pirro & Wessol, 2010; Shashidhara & Jayaram, 2010). While being environmentally friendly, non-toxic, and fully biodegradable, vegetable oils have also shown good performance in terms of its ability to reduce friction (Biresaw et al., 2002; Choi & Chun, 1998; Karmakar et al., 2017; Syahrullail & Shakirin, 2013). For example, Masjuki et al. (1999) found that palm oil-based lubricating oil showed better wear performance compared to that of mineral oil.

Blending of mineral oil with vegetable oil is generally performed to improve the overall lubrication performance of the base oil (Bahari et al., 2018b; Hassan & Syahrullail, 2016; Jabal et al., 2014; Nagendramma & Kaul, 2012; Syahrullail & Shakirin, 2014). Hassan et al. (2016) found that a blend of 40% of RBD palm olein with mineral oil improved the lubricant performance in terms of COF after comparing with pure mineral oil (Hassan et al., 2016). In a different study, Masjuki et al. (1997) emphasized that 5 wt% of palm oil methyl ester (POME) added to mineral oil enhanced the performance of the oil in higher temperature and reduce the COF because POME acts as anti-wear additive (Masjuki & Maleque, 1997). However, the blending of mineral oil with vegetable oil only is not sufficient to obtain oil with better specifications (Maheswari & Ganai, 2014). Several studies have shown many ways to add materials to the vegetable-mineral oil blended, such as polymers, esters and various nanomaterials (Shenoy et al., 2012; Binu et al, 2014). Zulkifli et al. (2014) reported, the trimethylolpropane (TMP) ester added to paraffin provided better surface protection for surface compared to paraffin oil (Zulkifli et al., 2014). Gryglewicz, et al. (2006) noted that adding about 10% of the respective esters to oils based on polyalphaolefins enhanced their properties, pour point, viscosity at low temperature and viscosity index (Gryglewicz & Surawska, 2006).

The addition of nanomaterials would increase the oil performance in term of friction and wear, because of its small size and possibility to enter the dent and crack on the metal surfaces thus increase the smoothness of the surfaces. Nano-spherical-shaped materials form sliding surfaces above each other, thus reduces the friction and resistance. This will increase the operating life of the oil and the operational life of the engine. Many studies used nanomaterials as additive to improve engine oils performance (Gao & Xue, 2002; Hosseini & Mohammadi, 2013; Zhmud & Pasalskiy, 2013;Peña-Parás et al., 2018). Improvement in COF and reduction in wear rate were found with the addition of CuO (Pena-Paras et al., 2014; Gulzar et al., 2015; Gulzar et al., 2017) and hexagonal boron nitride (hBN) nanoparticles (Elik et al., 2013; Talib et al., 2018).

The understanding of thermal effect is very important in order to assess to the performance of engine oil at high temperatures. Generally, the increase in temperature will affect the physicochemical properties of the oil, which will further affect the rheology and tribology properties of the oil (Devlin et al ., 2018; Hu & Cen, 2016; Peng 2015; Santos et al., 2005). High temperature also accelerates wear, destroys hydrodynamic lubrication regimes, increases the oxidation rate, fosters additive depletion, and affects other critical aspects of the machine (Devlin et al., 2018; Peng et al., 2015; Santos et al., 2005). Several studies have indicated that vegetable oils and esters have poor degradation stability than mineral oil, due to its chemical composition for instance. However, the presence of polyunsaturated bonds in vegetable oils has led to oxidation or hydrolysis of the oils (Arumugam, Sriram, & Subadhra, 2012; Raof et al., 2016; Zulkifli et al., 2014).

Mineral oil is composed of thousands of hydrocarbons and isomers. In all cases there is a breakage of the saturated or unsaturated bonds, with the presence of oxygen. Santos (2007) stated the least molecular weight compounds evaporate first, which are often thermally unstable. It is because the low the molecular weight of a compound, the less heat or energy it requires to evaporate. In addition, the mineral oil is thermally unstable since the chemical properties change with heating (Gnanasekaran & Chavidi, 2018; Salimon et al., 2010).

1.2 Problem statement

The friction between the metal parts in the engine increases the temperature of the engine hence will damage and corrode the engine parts. This will affect the efficiency of the engine and increase the fuel consumption.

1. The mineral oil has poor lubricating properties in term of low flash point, non-biodegradable properties, etc., compared to plant oil. On the contrary, vegetable oil possesses high flash point and excellent lubricity and is expected to improve the tribology of the oil.
2. Polyol ester, such as TMP ester has excellent lubricating properties and thermal stability but expressive which are 3 to 4 time more than palm oil.
3. Nanomaterials play a main role in improving oil lubricating properties especially the wear and friction. However, there are still lacks of studies on the effect of certain nanoparticles on the blending of mineral oil, palm oil and TMP ester.
4. The information on the thermal degradation of the formulated oil and its effect on the properties of the oil is still insufficient.

1.3 Objective of the study

The overall objective of the present study is to evaluate the effect of blending palm oil and trimethylolpropane (TMP) ester and adding nanoparticles on the lubricating properties of mineral oil.

1. To determine the friction coefficient and wear scar diameter of palm oil blended with TMP ester when added into mineral oil.
2. To investigate the effect of adding nanomaterials on the lubricating properties of the newly formulated engine oil.
3. To evaluate the thermal degradation process of the formulated engine oil.

1.4 Scope of work

The scope of study of this research is to produce high performance lubricant for engine oil by using palm cooking oil (93, 94, 95, 96 and 98) wt% , TMP ester (2,4, 5, 6 and 7) wt% blending together to get Palm base TMP ester. Then blending with commercial mineral oil 10W-40 (30, 50 and 70) wt% together to find the best formulation under standard condition for four ball tribotester with temperature 75 ºC, load 40 kg, with rotation speed 1200 rpm and run the test for one hour. The usage of cooking palm oil and TMP ester are preferred due to the availability of palm oil in Malaysia and also its friction and wear performance which are expected to improve engine oil performance. In addition, the effects of 5 different types of nanomaterials, namely CuO, Al_2O_3, $TiSiO_4$, Graphene plates and nanoglass powder on wear and friction of formulated engine oil have been determined.

1.5 Thesis outline

This thesis consists of five chapters. The first chapter contains an introduction to the engine oils and their importance and impact on the protection of metal parts inside the engine. The gap and the objectives of the study is also mentioned in Chapter 1. The second chapter covers the literature reviews, with a list of previous studies on oils of various types of oils (mineral, vegetable, mixed, chemically manufactured). Comparison of the additives and nanoparticles used to improve the performance of oils in terms friction, wear and reducing the engine temperature is also discussed. The third chapter includes materials used and the methods of conducting the scientific tests (four ball tribotester, scanning electron microscopy (SEM), Field-Emission Scanning Electron Microscope (FESEM), Fourier Transform Infrared Spectroscopy (FTIR), Density, viscosity, Viscosity index (VI), Thermal degradation analysis (TGA/DSC), The fourth chapter presents the results of all analysis and followed by discussions on the findings. The fifth chapter is a summary of the research results and important recommendations for those interested in the future work.

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