UNIVERSITI PUTRA MALAYSIA

OPTIMIZATION OF HIGH OLEIC PALM OIL-BASED TRIMETHYLOLPROPANE ESTERS SYNTHESIS IN PULSED LOOP REACTOR

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By

HAMIDAH ABD HAMID

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

May 2016

DEDICATION

Dedicated to the ones who grew me up and always supporting me.

I owe them each moment of my life and praise them in every breath.

Dedicated to my beloved parents, Esah binti Dinin and Abd Hamid bin Lateh.

Abstract of thesis presented to the Senate of Universiti Putra Malaysia
in fulfilment of the requirement for the degree of Doctor of Philosophy

OPTIMIZATION OF HIGH OLEIC PALM OIL-BASED TRIMETHYLOLPROPANE ESTERS SYNTHESIS IN PULSED LOOP REACTOR

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HAMIDAH ABD HAMID

May 2016

Chairman: Professor Robiah Yunus, PhD

Faculty: Institute of Advanced Technology

Some concerns about undesired saponification have been raised during the synthesis of high oleic palm oil-based trimethylolpropane (TMP) esters through transesterification of high oleic palm oil-based methyl esters (PME) with TMP in the presence of sodium methoxide catalyst by using a batch reflux reactor. A pulsed loop reactor was proposed to intensify the transesterification process. The main goal of this research work was to minimize fatty soap formation while maintaining the quality of high oleic TMP esters as biolubricant base oil by the use of a pulsed loop reactor. Response surface methodology (RSM) was applied to optimize the process conditions in the conventional reactor, based on maximum yield of high oleic TMP triesters (TMPTE) and minimum fatty soap amount. An Aspen Plus simulation study was conducted to facilitate the design of the proposed reactor. The performance of the pulsed loop reactor was evaluated under different process conditions to find the optimum yield of high oleic TMPTE and fatty soap. Subsequently, the quality of physicochemical properties of fractionated high oleic TMP esters were evaluated against other high oleic TMP esters base oils produced in the previous studies.

The optimization study for the synthesis of high oleic TMP esters in the batch reflux reactor by using RSM has shown that the optimum yield of high oleic TMPTE and fatty soap were at 80 wt% and 85 mg/g respectively. The optimum process conditions were obtained at 130 °C, 0.3 wt% sodium methoxide catalyst and 53 min of reaction time, at fixed molar ratio of high oleic PME: TMP at 3.9:1 and vacuum level of 20 mbar. The total liquid and vapor product throughputs from Aspen Plus simulation were estimated at 2106 g/h and 192 g/h respectively under the optimum process conditions. The predicted heat duty values for the reactor and vacuum condenser was 0.25 kW and -0.01 kW respectively. After several commissioning runs, the newly designed pulsed loop reactor was successfully commissioned to produce more than 90 wt% of TMPTE in 1 hour.

For the optimization of high oleic TMP esters synthesis in the pulsed loop reactor, the optimum process conditions were at 20 mbar, 1.0 wt% catalyst solution (30 % pure sodium methoxide in methanol), molar ratio of high oleic PME:TMP at 3.9: 1, 120 °C and 180 rpm. The optimum yield of high oleic TMPTE and fatty soap were 97 wt% and 167 mg/g, respectively. The predicted fatty soap amount from Aspen Plus simulation was 17 mg/g. The experimental fatty soap contains not only fatty soap but other substances such as TMP esters, PME, TMP and also the catalyst. The fractionated high oleic TMP esters satisfied ISO VG 46 lubricant standard and showed comparable properties to the other high oleic TMP esters base oils synthesized previously via conventional method.

Abstrak tesis yang dikemukakan untuk Senat Universiti Putra Malaysia sebagai memenuhi keperluan untuk ijazah Doktor Falsafah

PENGOPTIMUMAN SINTESIS TRIMETILOLPROPANA ESTER BERASASKAN MINYAK SAWIT BEROLEIK TINGGI DI DALAM REAKTOR GELUNG DENYUT

Oleh

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Kebimbangan terhadap saponifikasi yang tidak diingini telah dibangkitkan semasa sintesis trimetilolpropana (TMP) ester berasaskan minyak sawit beroleik tinggi melalui transesterifikasi metil ester berasaskan minyak sawit (PME) beroleik tinggi dan TMP telah dijalankan dengan kehadiran pemangkin natrium metoksida menggunakan reaktor refluks kelompok. Reaktor gelung denyut telah dicadangkan untuk memperhebatkan lagi proses transesterifikasi. Matlamat utama penyelidikan ini adalah untuk meminimumkan pembentukan sabun berlemak dengan mengekalkan kualiti TMP ester beroleik tinggi sebagai minyak asas biopelincir dengan penggunaan reaktor gelung denyut. Kaedah permukaan gerak balas (RSM) telah digunakan untuk mengoptimumkan keadaan proses tersebut di dalam reaktor konvensional, berdasarkan hasil maksimum TMP triester (TMPTE) dan amaun minimum sabun berlemak. Kajian simulasi Aspen Plus telah dijalankan untuk memudahkan proses rekaan bentuk reaktor. Prestasi reaktor gelung denyut telah dinilai pada keadaan proses yang berbeza untuk mendapatkan hasil TMPTE beroleik tinggi dan sabun berlemak yang optimum. Kemudian, kualiti sifat-sifat fizikokimia TMP ester beroleik tinggi yang telah melalui proses pemeringkatan telah dinilai dan dibandingkan dengan beberapa TMP ester beroleik tinggi lain yang telah dihasilkan oleh kajian sebelum ini.

Kajian pengoptimuman untuk sintesis TMP ester beroleik tinggi di dalam reaktor refluks kelompok menggunakan RSM telah menunjukkan bahawa hasil TMPTE beroleik tinggi dan amaun sabun berlemak yang optimum masing-masing adalah pada 80 wt% dan 85 mg/g. Keadaan proses optimum telah diperolehi pada 130 °C, 0.3 wt% pemangkin natrium metoksida dan 53 min masa tindak balas, oleh ketetapan nisbah molar PME beroleik tinggi: TMP pada 3.9:1 dan tekanan vakum pada 20 mbar. Jumlah keseluruhan pemprosesan produk-produk cecair dan wap daripada simulasi Aspen Plus masing-masing telah dianggarkan pada 2106 g/j dan 192 g/j di bawah keadaan proses optimum. Nilai duti haba untuk reaktor dan pemeluwap vakum diramalkan adalah masing-masing 0.25 kW dan -0.01 kW. Selepas beberapa proses pentauliahan, reaktor

gelung denyut yang direka telah berjaya ditauliahkan untuk menghasilkan TMPTE lebih daripada 90 wt% dalam masa 1 jam.

Bagi kajian pengoptimuman sintesis TMP ester beroleik tinggi di dalam reaktor gelung denyut, keadaan proses optimum telah didapati pada 20 mbar, 1.0 wt% larutan pemangkin (30 % natrium metoksida tulen di dalam metanol), nisbah molar PME beroleik tinggi: TMP pada 3.9: 1, 120 °C, dan 180 rpm. Larutan natrium metoksida optimum (30 % dalam metanol) adalah pada 1.0 wt%. Hasil optimum bagi TMP ester beroleik tinggi dan sabun berlemak adalah pada 97 wt% dan 167 mg/g. Anggaran kandungan sabun berlemak berdasarkan simulasi Aspen Plus adalah 17 m/g. Kandungan sabun berlemak daripada eksperimen bukan hanya mengandungi sabun berlemak, malah juga mengandungi bahan-bahan lain seperti TMP ester, PME, TMP dan juga pemangkin. TMP ester beroleik tinggi yang telah melalui proses pemeringkatan memenuhi keperluan piawai pelincir ISO VG 46 dan telah menunjukkan ciri-ciri yang setanding dengan minyak pelincir asas TMP ester beroleik tinggi lain yang telah disintesis sebelum ini menggunakan kaedah konvensional.

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I certify that a Thesis Examination Committee has met on 18 May 2016 to conduct the final examination of Hamidah Abd Hamid on her thesis entitled "Optimization of High Oleic Palm Oil-Based Trimethylolpropane Esters Synthesis in Pulsed Loop Reactor" 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 Doctor of Philosophy.

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

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LIST OF ABBREVIATIONS

ANOVA	Analysis of variance
API	American Petroleum Institute
ASTM	American Standard Testing Method
CCRD	Central composite rotatable design
CH_3OH	Methanol
CV	Coefficient of variation
D	Column diameter
df	Degree of freedom
d_o	Orifice diameter
f	Oscillation frequency
F value	A test comparison between model variance and residual variance
FID	Flame ionization detector
GC	Gas chromatography
IV	Iodine value
JOME	Jatropha *curcas* oil methyl esters
k	General reaction rate constant
L	Baffle spacing
$NaOCH_3$	Sodium methoxide
p-value	Probability of observed effect on process studied
PME	Palm oil methyl esters
PSRK	Predictive Redlich-Kwong-Soave method
R^2	Coefficient of determination
R^2_{adj}	Adjusted coefficient of determination
Re_o	Oscillatory Reynolds number
Re_n	Net flow Reynolds number
RSM	Response Surface Methodology
S	Baffle orifice open area
St_r	Strouhal number
TAN	Total acid number
TMP	Trimethylolpropane
TMPME	Trimethylolpropane monoesters
TMPDE	Trimethylolpropane diesters

TMPTE	Trimethylolpropane triesters
ψ	Velocity ratio
ρ	Fluid density
μ	Fluid dynamic viscosity
ω	Angular frequency of oscillation
x_o	Center-to-peak oscillation amplitude
V	Volume of reaction chamber
VI	Viscosity index
Y	Dependent variable response
z	Total tube of length

CHAPTER 1

INTRODUCTION

1.1 Background

Lubricants are normally used as a layer of fluids to reduce friction, wear and heat effects between moving surfaces. The property of decreasing friction is called lubricity, which is the main requirement of a fluid to be identified as a lubricant. High performance and environmentally friendly lubricants after formulating with one or more base oils and performance-enhancing additives must be environmentally non-toxic, easily biodegradable that do not cause adverse effects to humans, animals, flora and aquatic life, perform sufficiently well, economically feasible and practical and capable for the large-scale production (Schneider, 2006; Rensselar, 2011).

Lubricants usually consist of 70-90 % of base oils derived from mineral, synthetic or biobased oils and 10-30% additives. According to American Petroleum Institute (API), the base oils are categorized into five different groups based on their viscosity variations, levels of saturated components and sulfur content (Institute, 2015). Biolubricants are categorized in Group V since they are not found in any of other API base oil groups. They are usually used in small quantities as secondary base oils today and could also become the raw material for additives.

Over the past thirty years, a global awareness on environmental issues related to lubricants has increased the interest in biolubricants research and development. Besides, the increasing petroleum market price, demand and strict legislations imposed by several countries also escalate the interests in biolubricants. Generally, a biolubricant must be biodegradable, nontoxic and mainly formulated from renewable base stocks, i.e. vegetable oils and derivatives. It conforms to sustainable carbon dioxide balance and has specific performance requirements when it becomes the end product (Nehls & Moore, 2007).

World lubricants demand is forecasted to grow to about 45.4 million metric tons in year 2019, due to the expanding vehicle ownership and continuing industrialization (The Freedonia Group, 2015). Asia represents the fastest growing demand, followed by Africa-Middle East region, Eastern Europe, Central and South America, North America and Western Europe. In terms of products, industrial oils are expected to be the fastest growing product segment, whereas hydraulic fluids and process oils will overtake engine oils to be the largest products demand. The important opportunities will exist for the renewable and environmentally adaptable lubricating oils based on the rising strict regulations in developed countries.

For the forecasts of global lubricating oils and greases market up to year 2020, it is expected to achieve 12.4 billion gallons as reported recently by Global Industry Analysts (Global Industry Analysts, 2015). The growth is due to the dependency of our modern life on fuel driven machines and technology systems, which leads to the increasing number of motor vehicle ownership globally. Biolubricants is also expected to expand their markets globally at compound annual growth rate (CAGR) of 6.3% between 2015 and 2019 especially in automotive, transportation and industrial applications. This is due to their advantages such as able to reduce half of the total greenhouse gases emission and the two per three of energy consumption as compared to mineral oil-based lubricants (Technavio, 2015).

Most biobased lubricants are derived from vegetable oils that contain a combination of saturated and unsaturated fatty acids. Some oils also contain additional functional groups. Vegetable oils that mostly contain high unsaturated fatty acids have some inherent limitations as lubricants such as poor oxidative stability, sensitivity to hydrolysis and low-temperature fluidity. However, it could be resolved by genetic modification or chemical modification of the vegetable oils to convert the oils into synthetic esters-based lubricants by using chemical catalysts or biocatalysts. Several researchers have reported the enhancement of thermo-oxidative stability of the vegetable oil-based synthetic lubricants (Schlosberg *et al.*, 2001; Yunus, 2003; Joseph *et al.*, 2007; Erhan *et al.*, 2008; Åkerman *et al.*, 2011; Salimon *et al.*, 2012).

In Malaysia, the synthesis of vegetable-oil synthetic esters is from palm oil methyl esters (PME) as the main raw materials since PME is readily available in this country (Yunus *et al.*, 2003). Malaysia is one of the world's major oils and fats producers and also the second largest palm oil producer after Indonesia since 2006 (Ahmad *et al.*, 2008). Palm oil product such as PME has been used as green biodiesel with positive results and comparable with petroleum diesel (Choo *et al.*, 2005). The availability of PME from the biodiesel industry would provide extra push to the biolubricant industry in Malaysia. The usage of PME as the feedstock alternative to fatty acids has also been well accepted by the oleochemical industry.

In the prior studies (Yunus *et al.*, 2005; Chang *et al.*, 2012; Masood *et al.*, 2012), high oleic PME has been used as the feedstock in the synthesis of polyol esters as lubricant, with trimethylolpropane (TMP) as the branched polyol. The PME was selected due to its easiness to separate from the final products (Yunus, 2003). The reactions were carried out in the presence of alkaline catalysts. The chemical structure modification involves the substitution of the unstable hydrogen element of polyol with a stable molecule, which is the ester group from PME. Hence, the polyol esters produced would have improved lubrication properties and thermo-oxidative stability compared to the original palm oil.

Biolubricants are commonly produced in the conventional batch reactors. The drawbacks of these conventional batch reactors are the difficulty to maintain the product quality, longer time of reaction, high capital and operational costs and less effective for their start-up and shut-down routines. An alternative technology to solve these problems is by using an oscillatory flow reactor. This type of reactor offers an

enhanced mixing intensity, which leads to better mass and heat transfer and yield a better product. The in-depth knowledge of the reaction mechanism and kinetics and also a proper method for designing an oscillatory flow reactor are required to obtain the feasible and practical process with the desired products.

To date, no research has been published for the biolubricant synthesis using a pulsed loop reactor although the technology has been used for the biodiesel production (Azhari, 2011). In a similar study, Syam *et al.* (2012) used the oscillatory flow mixing for the synthesis of Jatropha *curcas* oil-based biodiesel in the U-tube pulsed reactor. The study was carried out at atmospheric conditions. The results showed that at the reaction temperature of 60 °C, only 10 minutes of reaction time was needed to achieve the conversion of 99.7% (Syam *et al.*, 2012). The conventional process usually took about 1 hour to complete the reaction (Azhari, 2006). Hence, the design principle of their research work was used as the main reference in designing the pulsed loop reactor for the synthesis of palm oil-based TMP esters. Similar information on the design of pulsed loop reactor for biolubricant synthesis is not available.

To reduce the formation of fatty soap materials, one of the approaches would be to reduce the catalyst amount. However, the use of less catalyst would normally slow down the reaction in a conventional reactor. Due to its enhanced mixing intensity, which leads to better mass and heat transfer, a pulsed flow reactor is proposed for this study. The design of the pulsed flow reactor for the synthesis of palm oil-based TMP esters is more complicated since the process involves a vacuum condition. In addition, the process has the tendency to form fatty soap materials due to the use of alkaline catalyst.

1.2 Objectives

This study has several following objectives:

1. To optimize the synthesis of high oleic palm oil-based TMP esters in a batch reactor based on maximum yield and minimum fatty soap
2. To perform a simulation study by using Aspen Plus and examine the process performance prior to reactor design
3. To design a pulsed loop reactor for the synthesis of high oleic palm oil-based TMP esters and to determine the optimum process conditions for the reactor
4. To evaluate the quality of physicochemical properties of high oleic palm oil-based TMP esters base stock

1.3 Scope of Work

The scope of work of this research is primarily on the design of the pulsed loop reactor that is suitable for a process under vacuum condition. The conceptual design covers the geometrical configuration of the integral baffles, which are installed in the reactor tube for palm oil-based TMP esters synthesis. Prior to the design, the simulations of material and energy balances were done by using Aspen Plus. The influences of related parameters on the transesterification reaction conversion were also examined. The high oleic PME was selected prior to the synthesis to obtain a lower pour point product. The alkaline catalyst selected was sodium methoxide because of its higher mass transfer performance as compared to calcium methoxide. The reaction was investigated in the conventional batch reactor first, to study the optimum fatty soap and yield of high oleic TMP esters, before the reaction was conducted in the pulsed loop reactor. The characterization of the biolubricant was also carried out. The properties investigated include pour point, flash point, density, kinematic viscosity, viscosity index, soap content, total acid number, total oxidative stability, iodine value and ASTM color.

1.4 Thesis Outline

This report comprises of five chapters. Chapter 1, the introductory chapter, provides the background of study, the problem statement, objectives and scopes of work. Chapter 2 gives detailed analysis of the literature review which includes reviews on plant oil-based synthetic esters as biolubricants, previous researches related to the synthesis of plant oil synthetic lubricants, the literature view on the oscillatory flow reactor and parameters that influences the reaction in the oscillatory reactor.

All materials and methods are discussed in chapter 3. Chapter 4 comprises the results and discussions of the optimization of transesterification between PME and TMP in the batch reflux reactor, Aspen Plus simulation, the pulsed loop reactor design and commissioning, the optimization of high oleic TMP esters synthesis in the pulsed loop reactor and the physicochemical properties of the fractionated high oleic TMP esters base stock. Finally, chapter 5 concludes the present research work and provides the recommendations for the future works.

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