UNIVERSITI PUTRA MALAYSIA

SYNTHESIS OF HYDROXYL-ALKOXYLATE FATTY ESTERS FROM METHYL ESTERS OF PALM FATTY ACID DISTILLATE AND THEIR POTENTIAL APPLICATION AS LUBRICANT-BASED STOCK

LEE PEI LUAN
FS 2010 10

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By

LEE PEI LUAN

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

February 2010

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

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Chairman **:Prof. Dato' Dr. Wan Md Zin Wan Yunus**

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Methyl ester of palm fatty acid distillate (PFAD-ME) was used to synthesize hydroxyl-alkoxylate fatty esters *via* epoxidation of its double bonds in the fatty acid chains and alcohol oxirane group cleavage reaction. The effects of reaction temperature, molar ratio of the hydrogen peroxide to PFAD-ME unsaturation and molar ratio of the formic acid to the unsaturation on the epoxidation of PFAD-ME were studied. The study showed that more than 98% conversion of the unsaturation to epoxide ring moiety could be achieved within 3 hours of reaction period without any catalyst with the molar ratio of unsaturation: formic acid: hydrogen peroxide of 1:1:4 respectively and reaction temperature of 50 °C. For the epoxy ring opening process, optimization was also studied by changing various reaction parameters. The amount of boron trifluoride ethyl etherate, the catalyst used for the ring opening reaction of the epoxidized PFAD-ME with 2-ethyhexanol, was 0.75 % (wt/wt). The ring-opening reaction was carried out at 70 °C for 3 hours.

Other primary alcohols such as methanol, ethanol, n-propanol, n-butanol, iso-butanol, n-pentanol, and n-hexanol were also used for the ring opening reaction of the epoxidized PFAD-ME. The viscosity of the products improved by around 85% as compared to those of the epoxidized PFAD-ME and original PFAD-ME. Although their pour points were similar to that of the PFAD-ME of which was 18 ℃, the cloud point of the hydroxyl-alkoxylates decreased as the carbon chain of the alcohol used was increased. The RBOT time increased from 27.9 min for the PFAD-ME to 120.4 min for epoxidized PFAD-ME indicating that introduction of the epoxy group in the PFAD-ME increased its polarity. However, oxidation stability of the hydroxyl-alkoxylate fatty ester after reacting with 2-ethylhexoxyl groups was slightly better than that of the PFAD-ME. When the thermal stability of the products was examined using TGA/DTG, it was observed that the decomposition temperature increased from 290 ℃ for PFAD-ME and epoxidized PFAD-ME to around 390 ℃ for the hydroxyl-alkoxylate samples.

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

SINTESIS HIDROKSIL-ALKOKSILAT ESTER LEMAK DARIPADA SULINGAN METIL ESTER ASID LEMAK SAWIT DAN POTENSI APLIKASINYA SEBAGAI BAHAN ASAS MINYAK PELINCIR

Oleh

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Metil ester yang hasilkan daripada sulingan asid lemak minyak sawit (PFAD-ME) digunakan untuk menghasilkan hidroksil-alkoksilat melalui pengepoksidaan ikatan ganda dua dalam rantai asid lemak and pembukaan gelang epoksi dengan alkohol. Kesan suhu tindakbalas, nisbah mol hidrogen peroksida terhadap ketaktepuan, dan nisbah mol asid formik terhadap ketaktepuan ke atas pengepoksidaan telah dikaji. Didapati bahawa pengepoksidaan PFAD-ME boleh mencapai 98% penukaran ketaktepuan kepada gelang epoksi dalam masa 3 jam dengan menggunakan nisbah mol ketaktepuan: asid formik: hidrogen peroksida pada 1: 1: 4. Kesan pelbagai parameter tindakbalas ke atas pembukaan gelang epoksi telah dikaji. Sebanyak 0.75% (wt/wt) boron trifluorat etil eterat digunakan untuk tindakbalas pembukaan gelang epoksi dengan 2-etilheksanol. Tindakbalas pembukaan gelang epoksi telah dijalankan sebanyak 3 jam dengan 70 ℃.

Alkohol prima yang lain telah juga digunakan untuk tindakbalas seperti metanol, etanol, n-propanol, n-butanol, iso-butanol, n-pentanol, n-heksanol. Kelikatan produk-

produk hidroksil-alkoksilat telah meningkat sebanyak 85% berbanding dengan kelikatan PFAD-ME yang telah diepoksi and PFAD-ME asal. Walaupun titik curah untuk produk yang dihasilkan adalah sama dengan titik curah PFAD-ME asal and epoksida PFAD-ME iaitu 18 ℃, akan tetapi takat keruh menurun apabila rantai karbon untuk alkohol yang digunaan bertambah panjang. Masa RBOT untuk PFAD-ME telah meningkat dari 27.9 min kepada 120.4 min untuk PFAD-ME yang telah diepoksi, disebabkan oleh struktur yang lebih polar dalam epoksida PFAD-ME. Hidroksil-alkoksilat ester lemak yang menpunyai kumpulan 2-etilhekosilat telah menunjukkan kestabilan pengoksidaan yang sedikit lebih baik daripada PFAD-ME. Kestabilan termal untuk produk-produk telah diuji dengan menggunakan TGA/DTG dimana suhu penguraian telah meningkat dari 290 ℃ untuk PFAD-ME and PFAD-ME yang telah diepoksi kepada sekitar 390 ℃ untuk hidroksil-alkoksilat sampel.

ACKNOWLEDGEMENT

Firstly, I would like to express my heartfelt gratitude and deepest appreciation to my supervision committee chairman, Prof. Dato Dr. Wan Md Zin Wan Yunus, and member Prof. Dr. Zulkefly Kuang Abdullah for their guidance throughout the course of this study. I am equally indebted to Dr. Yeong Shoot Kian and Dr. Lim, consultants of this project, for their kind supervision and guidance during the course of my research work at Advanced Oleochemical Technology Division (AOTD), Malaysian Palm Oil Board (MPOB).

I would like to acknowledge with thanks for the financial support from Malaysian Palm Oil Board Graduate Students' Assistantship Scheme (MPOB-GSAS), in form of graduate assistantship. I would like to express my sincere thanks to staffs of Chemistry Department, Universiti Putra Malaysia and AOTD for their assistances.

Last but not least, I am also greatly indebted to my parent, siblings and friends, for their relentless encouragement and support that have contributed towards the accomplishment of this project.

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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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

Lee Pei Luan (GS 16954)

Date: 15th April 2010

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LIST OF ABBREVIATIONS

AV	Acid value
DTG	Derivative thermogravimetric
ESO	Epoxidized soybean oil
FTIR	Fourier transform infra-red
HOSO	High oleic soybean oil
IV	Iodine value
NMR	Nuclear magnetic resonance
OHV	Hydroxyl value
OOC	Oxirane oxygen content
PFAD	Palm fatty acid distillate
PFAD-ME	Methyl ester of palm fatty acid distillate
POD	Palm oil diesel
RBOT	Rotary bomb oxidation test
SO	Soybean oil
TGA	Thermogravimetric analyses
VI	Viscosity index
2-EH	2-ethylhexanol

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CHAPTER 1

INTRODUCTION

Vegetable oils have been used for a very long time. The role of vegetable oils has been increasing from time to time mostly due to its sustainable and renewable properties. The depletion of petroleum supply, awareness of environmental issues and the damages caused to the environment from the excessive use of petroleum-based products have accelerated the world demand for more sustainable and environmental friendly products. Vegetable oil based products are well known to be environment friendly, biodegradable, and readily available. The use of vegetable oil is not only limited to edible oils and personal care products, but has expanded to other downstream products like detergent, surfactant, lubricant, biodiesel, *etc*. The escalating price of petrochemicals has also motivated researchers to search for vegetable oils as alternative substitutes in petroleum-based products.

The production of oils and fats has increased by 2.7% to 154 million tonnes in 2007 compared to the previous years. Malaysia and Indonesia are the major two palm oil producers in South East Asia (SEA) as well as in the world, together accounting to around 85% of the world's palm oil production in year 2007 (Wahid, 2008). The major palm oil importers are China (19.1%), European Union (16.0%), India (12.3%) and Pakistan (5.5%). China alone has imported 5 million tonnes of palm oil in 2007. China has steadily increased its consumption of palm oil (Wahid *et al*., 2007).

There are 17 major types of vegetable and animal oils commercially traded in the world and palm oil is the highest traded followed by soybean oil. For non-vegetable oil, tallow is the highest traded compare to lard, butter and fish oil. In year 2007, Malaysia has contributed 41.4% out of the total palm oil traded in the world. With such abundant supply of palm oil, Malaysia Palm Oil Board (MPOB) has aimed at maximizing productivity, improving production efficiency and quality, and increasing value chain.

1.1 Palm oil

Oil palm was first introduced in Malaysia as an ornamental plant in 1871, and then commercially exploited as an oil crop only from 1911. Palm fruit yields two very different oils, palm oil and palm kernel oil, with very different compositions, properties and applications. About 80% of palm oil and palm kernel oil are used for food applications and the rest goes to non-food application. Among the food applications include frying oils, shortening, margarine and others, while non-food applications include the direct use of the oils into the production of diesel fuel substitute, epoxidized palm oil products, polyols, polyurethane, painting ink, *etc* (Teoh, 2002).

Palm oil contains almost equal proportions of saturated acid (palmitic ~44% and stearic ~4%) and unsaturated acids (oleic ~39% and linoleic ~10%). Palm oil is mainly used for food applications but finds some non-food uses as it is a source of valuable by-products such as carotene (500 - 700 ppm), and tocopherols and tocotrienols (710 - 1140 ppm). Table 1 has listed the characteristic of the crude palm oil. Palm oil can be crystallized to give palm stearin which melt between 48 ℃ and 50 ℃, and liquid fraction palm olein which melt between 18 ℃ and 20 ℃ (Gunstone, 2004). Generally, palm oil is exported in three major forms. They are palm oil, palm olein and palm stearin.

Table 1. Characteristic of crude palm oil

Fatty acid composition	% wt
14:0 (Myristic)	11.0
16:0 (Palmitic)	44.0
18:0 (Stearic)	4.5
18:1 (Oleic)	39.2
18:2 (Linoleic)	10.1
Iodine value	53.3
Tocopherols (ppm) Tocotrienols	880
Carotene (ppm)	600

(Source: O'Holohan, 1997)

In order to obtain palm oils that are suitable for its end uses, the crude palm oils need to be refined. This involves removing of undesirable components and odour, and minimizes the colour of the oils. The undesirable components that removed include phospholipids, free fatty acids, mono- and di-acylglycerols, colour, trace metals, oxidation products, and environmental contaminants (Gunstone, 2004). Refining process can be grouped as chemical refining and physical refining.

Physical refining requires only degumming, bleaching and steam distillation. Physical refining is suitable for oils that have high levels of free acid and low levels of phospholipids such as palm oil (Gunstone, 2004). During the physical processing of palm oil products to produce fully refined, bleached and deodorized palm oil products, palm fatty acid distillate (PFAD) is generated as a by-product (Ab Gapor *et al*., 2002b) (Figure 1.2).

However, chemical refining involves chemical neutralization, bleaching and deodorization. It is mostly preferred for oils with high phospholipid levels that can only be conveniently removed by alkali treatment. With chemical refining of palm oil, palm acid oil is generated as by-product. Figure 1.1 has illustrated the connection between the triacylglycerides and their derivatives.

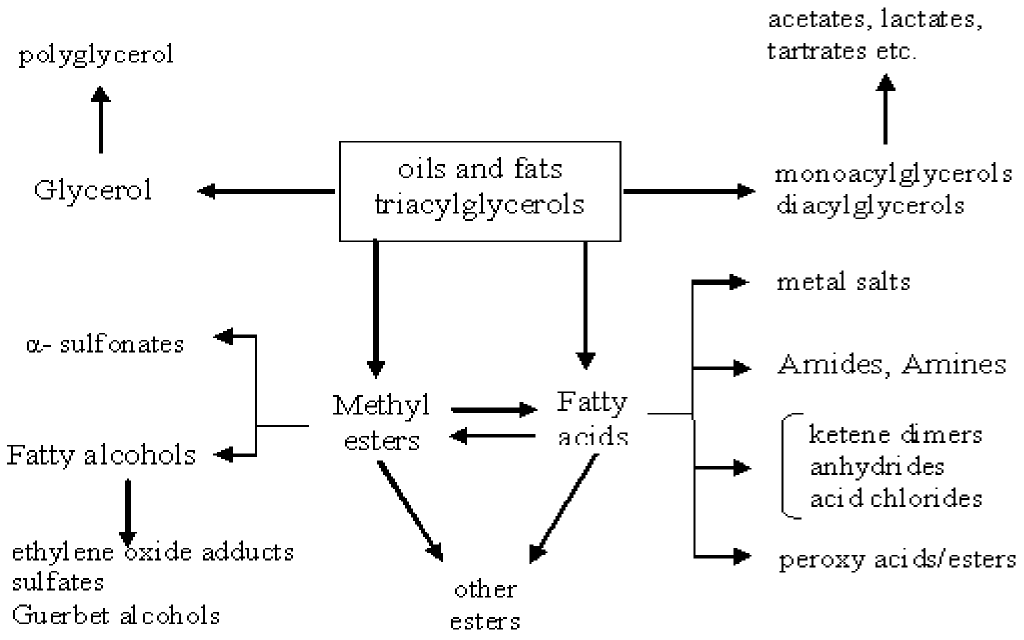
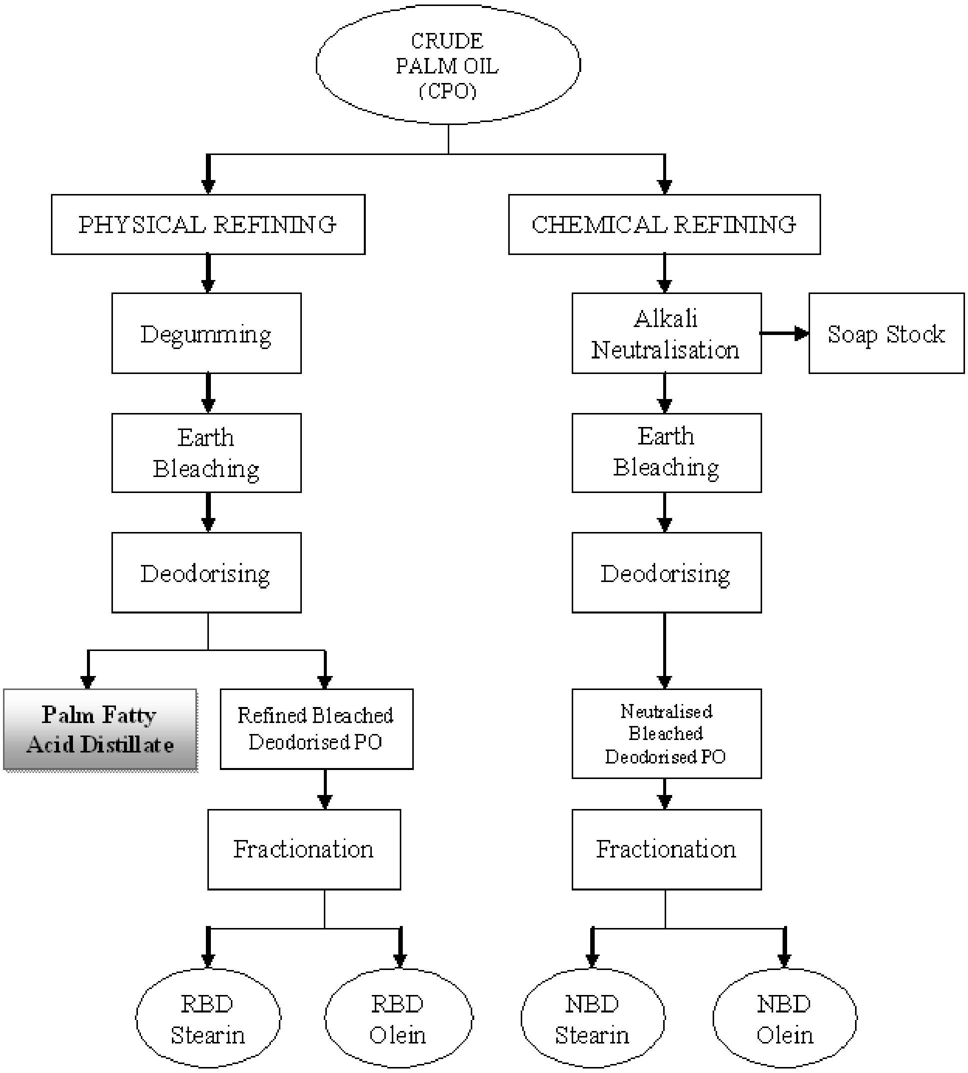


Figure 1.1. Inter-relationship between triacylglycerols and their derivatives
(Source: Gunstone and Hamilton, 2001)



PO = Palm oil
R = Refined
B = Bleached
D = Deodorised
N = Neutralised

Figure 1.2. Flow chart showing the palm oil refining process
(Source : Teoh, 2002)

1.2 Basic oleochemicals

Oleochemicals are chemical products derived from animal or vegetable triglycerides. The first oleochemical plant was set up in Malaysia in year 1982. Oleochemicals basically divided into two groups which are basic oleochemicals and derivatives. The five basic oleochemicals are fatty acids, fatty alcohols, methyl esters, nitrogen compound and glycerols (Hill, 2000). Besides that there is also the palm fatty acid distillate (PFAD) with its own field of applications in oleochemicals and animal feeds. These basic oleochemicals can be used to produce various types of derivatives through various chemical modifications such as alcohol ethoxylates, alcohol sulfates, alcohol ether sulfates, *etc*. Besides, oleochemicals can be also applied in many different industries, majority in the soap and detergents, plastic, personal care products, cosmetic, and lubricant industries.

Fatty acids or esters can be then used as the starting materials for producing of fatty alcohol and fatty nitrogens (Ahmad, 1994). Moreover, these products can be further modified to produce various types of derivative (Table 2).

Fatty acids are the most important of the five basic oleochemicals due to its versatility (Augustine *et al*., 1990). Fatty acids can be obtained from oils by hydrolysis with water dissolved in the oil phase, also known as splitting. Oleic acid is the widely occurring of the natural fatty acids. It is a monoethenoid normal aliphatic acid with 18 carbon atoms with the unsaturated bond between the ninth and tenth carbon atoms in the chain with formula $CH_3(CH_2)_7CH{=}CH(CH_2)_7COOH$ (Figure 1.3). Oleic acid is consisted more than 50% of the total fatty acids in many fats. Besides this, linoleic acid with two double bonds per molecule C18:2 and palmitoleic acid C16:1 also widely occur (Aviachem Marketing Report).

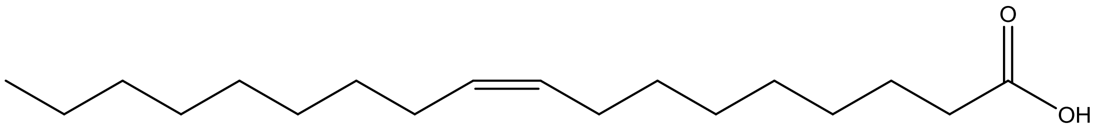


Figure 1.3. Oleic acid

Table 2. The oleochemical industry: materials used and processes employed

Raw materials

tall oil, tallow, coconut oil, palm oil, palm kernel oil, soybean oil, sunflower oil, canola oil

Unit operations to produce basic oleochemicals

Splitting, distillation, fractionation, separation, hydrogenation, methylation, hydrophilisation

Basic oleochemicals

Fatty acids, methyl esters, fatty alcohols, fatty amines, glycerol

Operations to produce derivatives of basic oleochemicals

Amidation, chlorination, dimerisation, epoxidation, ethoxylation, quaternisation, sulfation, sulfonation, transesterification, saponification.

Oleochemicals derivatives

Amides, dimer and trimer acids, epoxidized oils and esters, ethoxylates, sulfonates, esters, soaps and salts.

End-use markets

Building auxiliaries, candles, cleaning agents, cosmetics, detergents, fire extinguishing agents, flotation agents, food emulsifiers, insecticides, leather, lubricants, paints, paper, pesticides, pharmaceuticals, plastics, rubber, soaps, textiles.

(Source: Gunstone and Hamilton, 2001)

However, for the saturated fatty acid palmitic acid (C16:0) is the most common of all the saturated fatty acids that present in vegetable oils (Figure 1.4). Palmitic acids have high melting point compared to the methyl esters as a consequence of hydrogen bonding (Gunstone, 2004).

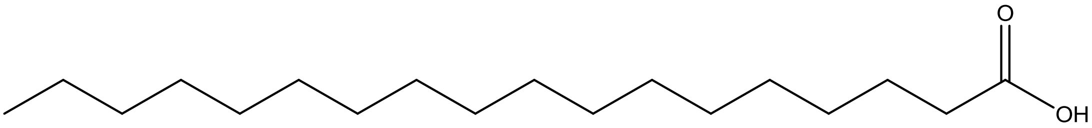


Figure 1.4. Palmitic acid

1.3 Objectives

Palm fatty acid distillate (PFAD) that generated during the physical refining is only used primarily as animal feed and low quality soap (Leong, 1988; Palmquist 2004; Thiagarajan, 1990). However, during the refining, those valuable minor components such as tocopherols, tocotrienols, squalene and phytosterol have been distillated off together with the PFAD. Thus, PFAD has been developed as a rich source of those phytonutrients (Ab Gapor *et a*l., 2002b). During the accumulation of phytonutrients from the PFAD, a by-product, the methyl esters of PFAD (PFAD-ME) is generated. With the purpose of compromising value-addition strategy, sustainable raw material and producing environmental friendly products, this methyl ester of palm fatty acid distillate (PFAD-ME) will be utilized into new applications.

The objective of this study is to synthesize a hydroxyl-alkoxylate *via* epoxidation of PFAD-ME followed by epoxy ring opening with alcohols. Hence, the optimum reaction conditions for the production of epoxidized PFAD-ME and the epoxy ring opening with 2-ethylhexanol will be studied in detail. Besides that, the opening of the epoxy rings with various primary alcohols to yield hydroxyl-alkoxylate PFAD-ME will also be compared and discussed. The properties of these hydroxyl-alkoxylate PFAD-MEs will be evaluated its potential as a lubricant base fluid.

CHAPTER 2

LITERATURE REVIEW

Palm oil consists of triglycerides and also small and variable non-glyceride components. In order to render the oil to an edible form, the oil needs to undergo various stages of refining to remove some of those non-glycerides to an acceptable level. There are two routes to process crude palm oil into refined oil, which are chemical refining and physical refining. The difference between these two routes are basically in the way of the free fatty acids are being removed from the oil. Chemical refining utilizes an alkali to neutralize the free fatty acid which will be removed as soap. While physical refining which involves subjecting the oil to steam distillation under high temperature and vacuum for the removal of the free fatty acid.

Most of the oil produced is processed locally and physically refined. Physical refining of crude palm oil is the common process in Malaysia because of it higher efficiency, less losses, lower operating costs, lower capital input and less effluent to handle (Mohd Suria Affandi, 1994). Palm fatty acid distillate (PFAD) are condensed and collected as the by-product of the physical refining.

2.1 Palm fatty acid distillate (PFAD)

PFAD is obtained as a condensate of the volatile matters carried over from the deodorizer by the action of the stripping steam and mainly characterized by high free